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S. M. Barnes
D. E. Larson

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

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MATERIALS AND DESIGN EXPERIENCE IN A SLURRY-FED ELECTRIC GLASS MELTER

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Pacific Northwest Laboratory
Richland, Washington 99352

DOE-Richland, WA

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ABSTRACT

The design of a slurry-fed electric gas melter and an examination of the performance and condition of the construction materials were completed. The joule-heated, ceramic-lined melter was constructed to test the applicability of materials and processes for high-level waste vitrification. The developmental Liquid-Fed Ceramic Melter (LFCM) was operated for three years with simulated high-level waste and was subjected to conditions more severe than those expected for a nuclear waste vitrification plant.

The melter examination and analyses resulted in the following conclusions:

- Inconel 690® is an excellent material for the electrodes (corrosion rate ≤0.11 cm/yr).
- The Monofrax K-3® glass containment refractory developed moderate cracking, but this cracking did not produce a marked increase in refractory corrosion. The refractory cracking could be largely eliminated with improved design techniques. Monofrax K-3 wall corrosion was ≤0.85 cm/yr.
- The Alfrax 66® insulating refractory exhibited cracking caused by an excessive initial heat-up rate and by thermal expansion stresses of the melter refractory differential. No significant refractory corrosion was noted.
- The Zirmul® backup refractory was in excellent condition.
- The melter lid and cover plate were warped during the operating period as a result of materials differential thermal expansion.

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® Monofrax is a registered trademark of the Carborundum Co., Falconer, New York.
® Alfrax is a registered trademark of the Carborundum Co., Falconer, New York.
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This deformation created some air inleakage problems. Increased thermal expansion allowance will reduce this concern. Several metal components, attached to the lid and exposed to process offgas (for which the metals were not selected), exhibited significant corrosion.

Thus, the melter materials were in generally good condition after the three-year operating period. Design modifications to this experimental melter can significantly extend the service life of a vitrification system to longer operating periods. All of the major glass containment refractories displayed very good resistance to glass corrosion and are recommended for future designs.

SUMMARY

An electric glass-melting furnace designed to vitrify simulated high-level waste was operated for three years at the Pacific Northwest Laboratory. This report documents the analysis of the melter undertaken to study the effects of long-term operation on the construction materials.

The Pacific Northwest Laboratory has been developing joule-heated, ceramic-lined glass melters since 1973 under the sponsorship of the U.S. Department of Energy. An experimental, full-scale electric melter began operation in February 1977. This melter was designed to vitrify simulated high-level waste at a rate compatible with that of waste slurry feed produced by a 5 MTU/d commercial nuclear-fuel reprocessing plant. This melter operated from February 1977 to February 1980 and processed both slurry and dry feeds (representative of a coupled spray-calciner/ceramic-melter system). A pool of molten glass at operating temperature was maintained in the melter for the entire operating period. The details of the individual glass-production tests are presented in Appendix B. The Liquid-Fed Ceramic Melter (LFCM) produced about 80 tonnes of glass from 54 tonnes of calcine plus glass formers, and 19,000 L of slurry feed. The feed was nonradioactive synthetic wastes representative of reprocessed defense and commercial nuclear fuels.

The melter was shut down and examined in detail to determine the construction materials' performance and to evaluate the LFCM design. The melter was methodically dismantled, physical dimensions of the construction materials were measured, and selected materials were analyzed in the laboratory. Also, a stress analysis, based on the LFCM refractory design, was performed to predict refractory stress levels in the idling (melter at temperature, but not processing glass) and liquid-feeding modes.

The Monofrax K-3 glass contact refractory was uniformly cracked throughout the melter. The Monofrax K-3 refractory along the two-electrode and north melting cavity walls was in good condition (especially considering the severity of service the LFCM was exposed to relative to a production glass melter), but pieces in the south (drain) wall Monofrax K-3 refractory had

detached from the wall and fallen to the floor. The refractory cracking causing detachment was caused by the arrangement of thermal profiles in the melting cavity wall. The temperature profiles in all but the south wall produced thermal cracking perpendicular to the glass contact face; thus, the cracked refractories remained in position. The south wall thermal pattern, however, induced cracks that were inclined with respect to the glass contact face. These nonperpendicular cracks allowed the damaged refractory sections to fall from the wall. The general Monofrax K-3 corrosion rate was calculated to be ∿0.85 cm/yr.

The Alfrax 66 and Zirmul backup refractories were very resistant to glass corrosion, but numerous glass-filled cracks were found in the Alfrax 66 castable refractory. These cracks were the result of the rapid temperature changes in the LFCM startup, thermal expansion differences between the refractory materials, and tensile thermal stresses in the castable refractory regions.

The melter containment box and lid were largely unaffected by the corrosive conditions present during the three-year operating period, but the lid and one of the coolant channels were warped during service. Stress analysis showed that lid warpage can be reduced by designing increased thermal expansion freedom. The coolant channel distortion is thought to be caused by the high pressure developed by steam formation in the channel.

The Inconel 690 electrodes were in excellent condition; in fact, marks from the original machining operations were visible on the LFCM secondary electrode faces following the three-year exposure to the glass. The maximum corrosion rate of the electrodes was 0.11 cm/yr.

Evidence of sulfidation attack (corrosion by sulfur compounds and halides) was detected on an auxiliary heater and offgas bellows attached to the melter lid. Recent work with a modified slurry-fed melter indicates that this corrosion mechanism can affect components that demonstrated good corrosion resistance in the LFCM (e.g., lid, offgas ducting). This evidence suggests that use of alloys in contact with melter offgas need be considered for corrosion resistance for specific feeds.

The melter operating performance and the corrosion resistance of the Monofrax K-3 are not affected by the cracked refractory if the refractory remains in position. The materials selected for the LFCM generally performed as expected for the three-year period and are recommended for future use in melters operated under similar corrosive conditions. The majority of the refractory cracking and containment box warpage can be prevented by allowing additional design accommodation for the thermal expansion and contraction properties of the construction materials.

CONTENTS

FIGURES

TABLES

GLOSSARY

A	amp
® Alfrax	a registered trademark of the Carborundum Co., Falconer, New York.
CFCM	Calcine-Fed Ceramic Melter
cm	Centimeter
^oC	Degree centigrade
d	Day
DOE	Department of Energy
^oF	Degree Fahrenheit
h	Hour
HEPA	High Efficiency Particulate Air
HLW	High-Level Waste
in.	Inch
INEL	Idaho National Engineering Laboratory
® Inconel	a registered trademark of the International Nickel Co., Huntington, West Virginia.
K	Kelvin
kg	Kilogram
KVA	Thousand Volt Amps
L	Liter
LFCM	Liquid-Fed Ceramic Melter
mm	Millimeter
mo	Month
® Monofrax	a registered trademark of the Carborundum Co., Falconer, New York.
MPa	Pascal x 10^6

MTU	Metric Tonne Uranium
NC	Normally Closed
NO	Normally Open
PNL	Pacific Northwest Laboratory
s	second
SCR	Silicon-Controlled Rectifier
SEM	Scanning Electron Microscope
psi	Pounds per Square Inch
® Zirmul	a registered trademark of the Charles Taylor Sons Co., Cincinnati, Ohio.

1.0 INTRODUCTION

Pacific Northwest Laboratory (PNL) has been developing technology for the immobilization of high-level radioactive waste (HLW) under the sponsorship of the Department of Energy (DOE) and its predecessors for over 20 years. For the last 15 years, emphasis has been placed on the vitrification of HLW. One of the processes being developed at PNL for HLW vitrification is the slurry-fed electric melter process (Brouns et al. 1980; Dierks 1980; Buelt et al. 1979). The electric melter process has been under development at PNL since 1973. As of March 1981, nine electric melters (excluding laboratory melters) have been constructed and tested for process development. These have produced 130 tonnes of glass.

The largest of the PNL melters, the Liquid Fed Ceramic Melter (LFCM), pictured in Figure 1, was placed in service in February 1977. The LFCM was scaled to process simulated HLW at a rate compatible with that generated by a 5 MTU/d spent-nuclear-fuel processing facility. During the operating life of the melter, slurry and calcine feeds (which represent both commercial and defense wastes) have been processed. Most recently, the LFCM has been used for full-scale development studies in support of the Defense Waste Processing Facility at the Savannah River Plant. The melter has demonstrated processing slurry feeds of 100 L/h with only joule heating and up to 150 L/h with additional process energy input (e.g., ionic boosting, lid heaters). Calcined or dry feeds have been vitrified at rates of 160 kg/h.

During the three-year service, the LFCM was used to develop ceramic-lined melter technology, establish melter operating parameters, and study the processing characteristics of the glass formulations (presented in Appendix B). Modifications to the melting system, as well as processing experiments required to perform these studies frequently subjected the LFCM to conditions more extreme than those expected in a plant HLW vitrification system. In February 1980, the melter was shut down. Following the final draining, a comprehensive investigation was undertaken to determine the condition and performance of the construction materials and to assess the adequacy of the melter design.

FIGURE 1. Liquid-Fed Ceramic Melter

This report documents the materials and design experience of the LFCM so the knowledge developed may be used to construct reliable, long-life electric melters for radioactive waste immobilization in a plant. This report provides the context for interpreting the LFCM experiences by describing the LFCM vitrification system (Sections 1.0 through 4.0), documenting the effects of the operating period on the construction materials (Section 5.0), and interpreting the findings of the detailed examination of the melter and components (Sections 6.0 and 7.0).

In addition, comparisons are made with observations concerning the calcine-fed ceramic melter materials experience (Dierks et al. 1980), which was investigated in 1980. Results and conclusions are presented, and recommendations are made to provide guidance for future melter design.

2.0 CONCLUSIONS AND RECOMMENDATIONS

Conclusions and recommendations drawn from the LFCM examination and analyses are found in this section

2.1 GENERAL

The design of the LFCM and the performance of the structural materials were generally good. The corrosion performance of the glass containment refractories (Monofrax K-3, Zirmul, and Alfrax 66) was excellent. The melter operation was not affected by cracked primary or secondary refractories, provided the cracked refractory remained in place. Corrosion of the glass contact refractory, Monofrax K-3, was not enhanced by the refractory cracking. Future melter designs should consider the melter temperature and stress patterns in the ceramics to minimize the cracking and thus reduce the likelihood of failed refractory sections falling from the walls.

Only minor corrosion of the melter metallic materials was observed, with the exception of ionic booster system components and a section of flexible offgas ducting. In both of these cases, the corrosion was due to service under conditions for which they were not originally intended. The corrosion of these components can be alleviated by specifying alloys designed to resist the mixture of volatile sulfur and halogen compounds produced by recently tested waste-glass formulations.

The analyses performed on the LFCM indicate that the service life of ceramic-lined, waste-glass melters can be increased beyond the three years demonstrated by this melter. The most important factor for extended melter service is adequate accommodation of the thermal expansion properties of the construction materials.

2.2 MELTER CONTAINMENT BOX AND LID

Type 304L stainless steel is adequate for service in the melter containment box where contact with glass vapors and molten glass is not normally expected. This stainless steel, normally resistant to attack by sulfur

compounds, will corrode rapidly at elevated temperatures in sulfur atmospheres also containing halides and water vapor. Typical 18-8 stainless steels should not be used in sulfur-halide atmospheres above 200°C. Selection of the proper alloy for exposure to these conditions requires further characterization of the melter offgases to determine the concentration of the corroding species (NaCl, HCl, Na_2SO_4, SO_2, SO_3, etc.). The Inconel 601 LFCM lid was protected from sulfidation (high temperature halide and sulfur corrosion) by the external insulation. This insulation maintained the lid in the 850°C to 950°C temperature range during idling and (roughly) in the 100°C to 400°C range during operation. These temperatures are too hot and cold, respectively, for sulfidation to occur.

The thermal cycling inherent to externally insulated lids, however, produced the warpage and tearing detected in the lid following the melter shutdown. This damage was primarily due to the restriction of free thermal expansion of the heated lid by the water cooled containment box. Improved design to reduce the induced thermal stresses at the containment box-lid junction will produce an effective, inherently corrosion-resistant melter containment method.

The current trend to increase slurry feed processing rates by heating the "cold cap" from above will increase the operating temperature of an externally insulated lid into the sulfidation temperature range. In this case, an alternative insulation method or an alloy other than Inconel 601 may be needed to produce extended service life.

The warpage of the containment-box cooling channel was probably caused by a pressurization in the channel as residual water was converted to steam following the elimination of water cooling to this region. This damage can be avoided in the future by insuring that the coolant channels are either actively used or are dry and isolated from the coolant drain.

2.3 MELTING CAVITY REFRACTORY

Monofrax K-3, Zirmul, and Alfrax 66 demonstrated excellent corrosion resistance during the three-year operating period. Very little refractory

corrosion (<0.85 cm/yr) was observed in this melter examination. The Monofrax K-3 cracking did not increase the corrosion rate of this material.

The cracking of the Alfrax 66 observed during the LFCM disassembly was due to the rapid (∿22 h) initial melter startup period, differences in thermal expansion between the Alfrax 66 and the Monofrax K-3, and thermal stresses developed in the Alfrax 66 region. The rapid melter startup would not have permitted sufficient time for the residual water from refractory mixing (both excess water and water of hydration) to diffuse through the refractory and escape. This water would generate steam within the refractory and create damaging tensile stresses. This startup schedule heats the refractory much faster than the manufacturer recommends (presented in Section 5.3.2). Second, the Alfrax 66 has significantly different thermal-expansion characteristics than the Monofrax K-3. The Alfrax 66 tends to maintain roughly the same dimensions during the curing process whereas the Monofrax K-3 would be thermally expanding. As the Alfrax is cast surrounding the Monofrax K-3, this also would result in tensile stresses in the Alfrax 66 layer. Finally, the thermal-stress analyses of the LFCM refractory design presume tensile stresses as high as 83 MPa in the Alfrax 66 layer under the assumed liquid-feeding boundary conditions. This exceeds the Alfrax 66 tensile rupture strength by ∿30 times. Therefore, to reduce the magnitude of the Alfrax 66 cracking observed, the initial curing temperature schedule should be followed and the refractory installation should be designed to allow slippage along Alfrax 66-Monofrax K-3 joints, as well as to reduce the general thermal stress patterns. Following these suggestions, the Alfrax 66 cracking, and thus the number of glass penetrations through the Alfrax 66 layer, will be reduced.

Most of the Monofrax K-3 cracking can probably be prevented in future melters by allowing sufficient space for refractory thermal expansion, reducing the thermal gradients in the refractory by increasing refractory wall thickness, and reducing the severity of thermal transients such as startup and liquid-feeding initiation. The damage to the Monofrax K-3 refractory of the south wall (where cracked refractory pieces were missing) can be eliminated by modifying the temperature profile in the drain region.

2.4 ELECTRODES

The Inconel 690 electrodes demonstrated excellent corrosion resistance and no structural deformation. Similar good performance was noted in all of the other applications of this alloy. The electrode corrosion rate was calculated to be ≤0.11 cm/yr.

2.5 GLASS DISCHRAGE SECTION

The monolithic Monofrax K-3 riser block exhibited enlargement of the melter cavity inlet nozzle, about 0.32 cm (0.1 cm/y) horizontally and 5.7 cm (1.9 cm/y) vertically). Corrosion of the riser inlet was expected to be larger than the general refractory-face corrosion rate because: the velocity of the glass flow through the riser is higher than the velocity produced by convection currents in the melting cavity (slag buildup on the melter floor produces a smaller glass-flow area at the riser inlet, leading to even higher velocity); the riser leaves the melter at $10^{\circ}24'$ angle from the horizontal, resulting in a thin refractory section at the top of the riser inlet which is surrounded by hot glass; and melter refractory temperatures are greatest in this region because of the glass in the riser and the overflow drain heaters.

3.0 PROCESS AND EQUIPMENT DESCRIPTION

The LFCM processes and melter equipment are described in this section. Following a general description of the melter, process flowsheets representative of the LFCM operating conditions are presented. Completing this section is a detailed description of the melter subassemblies.

The LFCM may be characterized as a ceramic-lined melting cavity containing two sets of electrodes. Melter heat is primarily supplied by the joule-heating effect created by passing an alternating current through the glass between the electrodes. Feed and glass-forming materials enter the melter from the top and drop onto the molten glass pool. Unreacted feed materials accumulated on the glass surface are known as a "cold cap." As the feed and glass-forming materials are converted into vitrified oxides, moisture and other decomposition products are converted into a process offgas. These effluents are routed to an offgas treatment system for removal of water, particulates, volatile radionuclides, and toxic volatile chemicals. The vitrified molten product is emptied from the melter into a canister where it solidifies. The overall melter assembly is illustrated in Figure 2.

The LFCM is composed of the subassemblies indicated below and shown in the schematic in Figure 3.

Assembly	Function
Refractory	Glass containment and thermal insulation
Containment Box	Surrounds melter refractory providing cooling and controlled atmosphere
Lid	Covers melter containment box to house nozzles for melter-cavity access and to contain decomposition effluents and volatile species
Power Electrodes and Control	Main power source for cavity heat supply
Overflow Section	Melter section designed to transfer the molten glass product to the storage canister

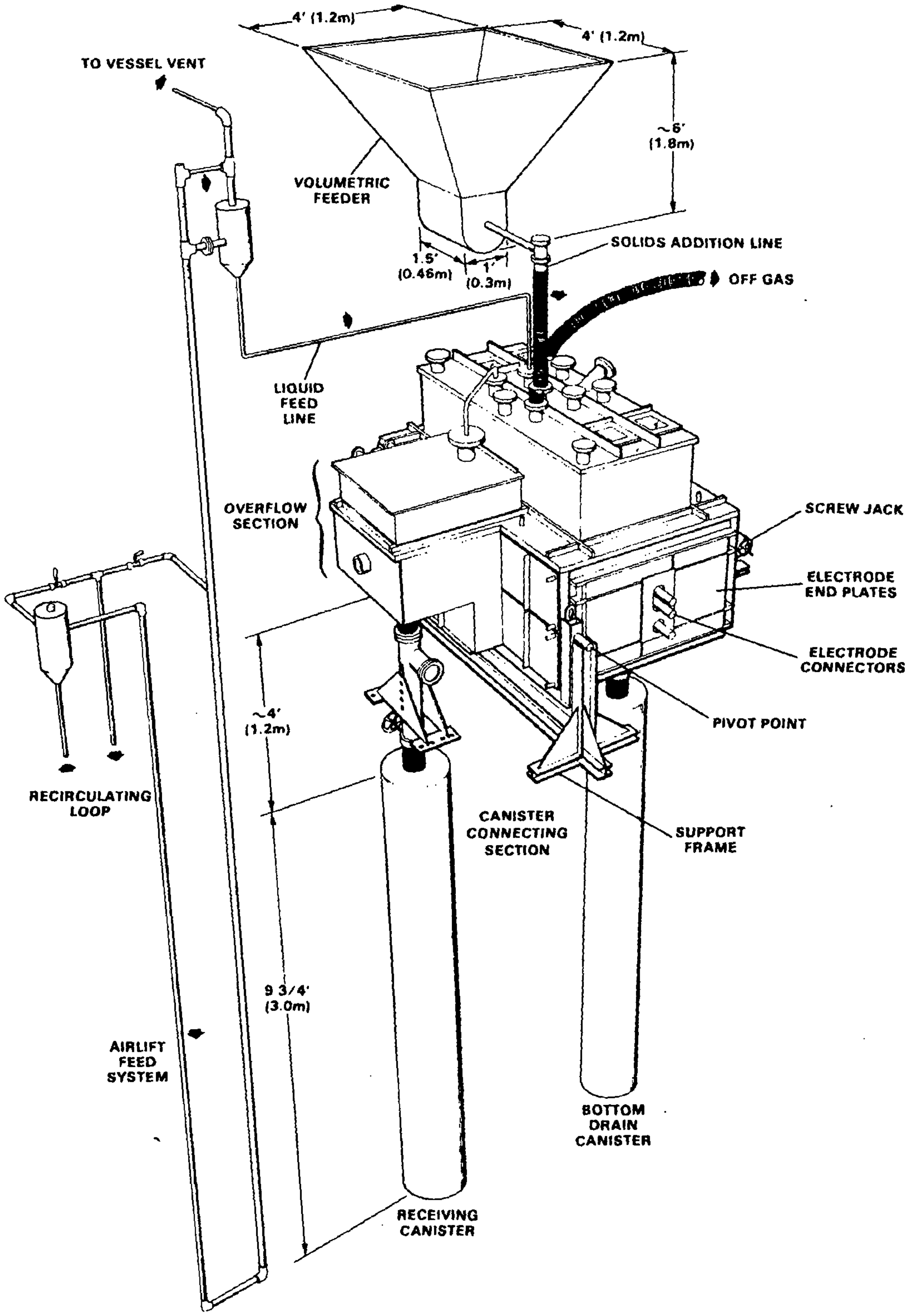


FIGURE 2. Liquid-Fed Ceramic Melter System Assembly

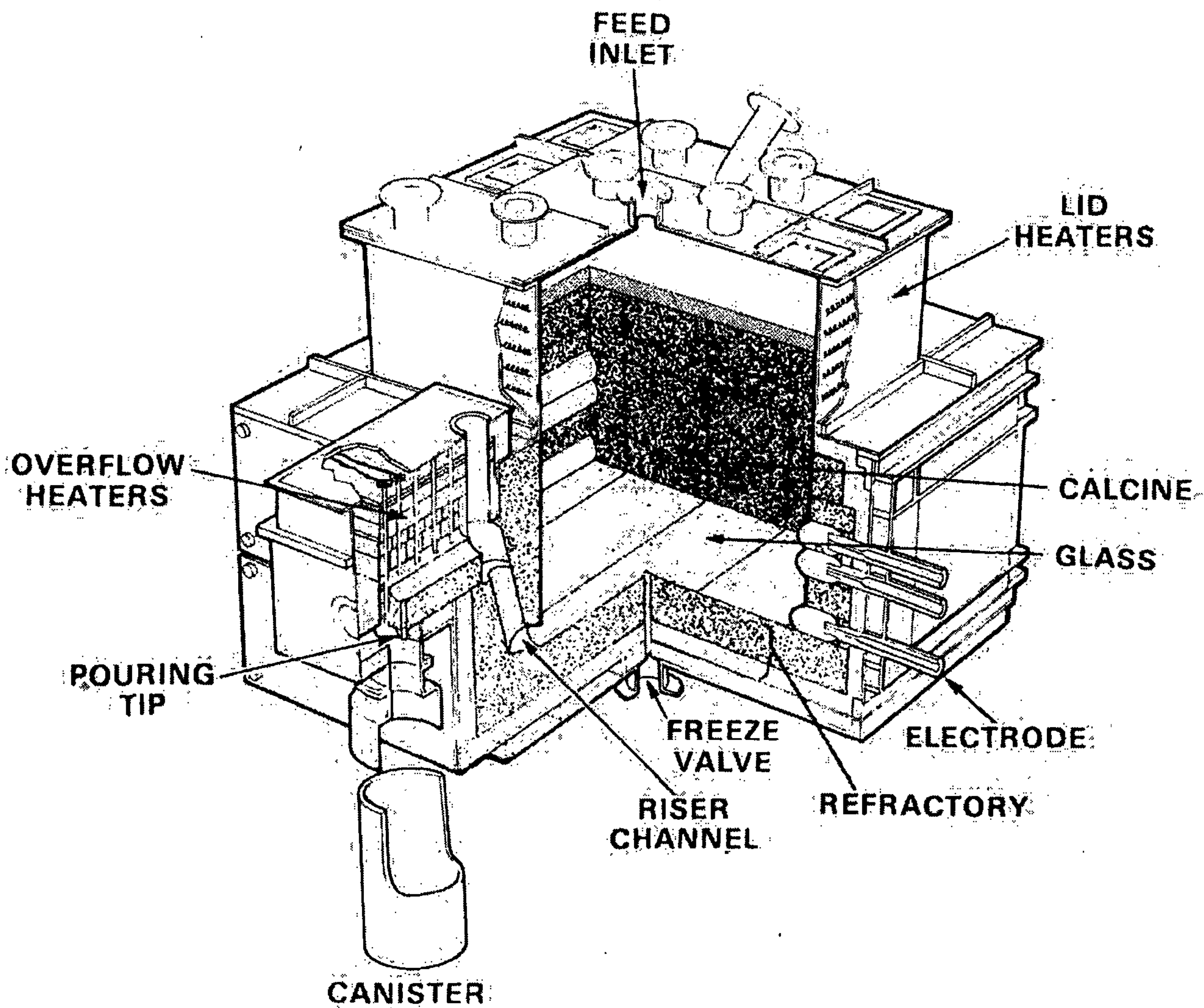


FIGURE 3. Liquid-Fed Ceramic Melter Cutaway

Assembly	Function
Auxiliary Power Systems	Three additional power systems are used in the LFCM: 1) glass-overflow section heaters, 2) lid heaters, 3) bottom drain heaters
Lid Heaters	Provide heat source for melter startup and boosting
Ionic Booster Electrodes and Control	Provide power for increased liquid feed evaporation capacity by directly heating aqueous "cold cap"

Assembly	Function
Bottom Drain	Molten glass drain for melter cavity draining
Instrumentation	Process monitoring and control
Feed Systems	Provide aqueous and calcined feed together with glass-forming materials to melter
Offgas System	Melter offgas handling and treatment

3.1 PROCESS FLOWSHEET DESCRIPTION

The LFCM has processed a variety of nonradioactive waste feeds. The principal melter feeds have been synthetic, commercial acid-waste slurry, defense-waste slurry, and defense-waste calcine (dry feed materials representative of a coupled spray-calciner/ceramic-melter system). Other minor feed materials generally consisted of a variety of frits. The general method of melter operation, representative flowsheets, and typical melter thermal conditions are discussed in this section.

The LFCM was operated in two predominant modes--slurry feeding and calcine feeding. In the liquid-fed process, the glass formers are premixed with simulated liquid waste before feeding the waste slurry to the melter. Normally, a crust of solid, partially reacted feed materials forms in the melting cavity separating the slurry and the molten glass. This solid crust and the boiling slurry above are known as the "cold cap." Heat from the molten glass is transferred through the crust to evaporate the slurry. This process continually generates new crust materials to replace the feed reacting to form glass at the molten interface. Supplementary heat may be supplied to the feed materials in the cold cap by radiation from lid heaters or electrodes placed in the slurry pool (ionic boosting). As the crust melts into the glass, additional gases are generated by the final decomposition stages. The steam and gases are collected in the plenum above the melting surface. This region is maintained at a slightly negative pressure relative to ambient conditions (-250 mm of water). From here, the effluents pass through the offgas treatment system. Particulates and volatiles collected in this system are recycled to the melter as required. The waste glass product is drained continuously into a receiving canister.

The slurry-feeding process is characterized by two operating modes based on the volume of liquid feed maintained in the cold cap. These operating styles are termed "fully flooded" and "nonflooded." In the fully flooded mode, the feed slurry covers the entire melting cavity surface. Maximum vitrification rates are achieved with fully flooded cold caps. The gases generated by the final decomposition stages of the feed forming the crust must, however, vent through the crust. Release of these decomposition gases results in local crust fractures and momentary contact of feed slurry and molten glass. The contact with the molten glass results in rapid heat transfer to the slurry and increased offgas flow (roughly three times the average steady-state effluents flowrate--Brouns et al. 1980). This phenomenon occurs regularly every few minutes and is depicted in Figure 4.

The nonflooded cold cap is shown in Figure 5. In this mode, the feedrate is deliberately maintained below the maximum processing capability of the melter. This results in more stable melter operation with fewer offgas flow peaks as the decomposition gases are released around the cold cap periphery. The

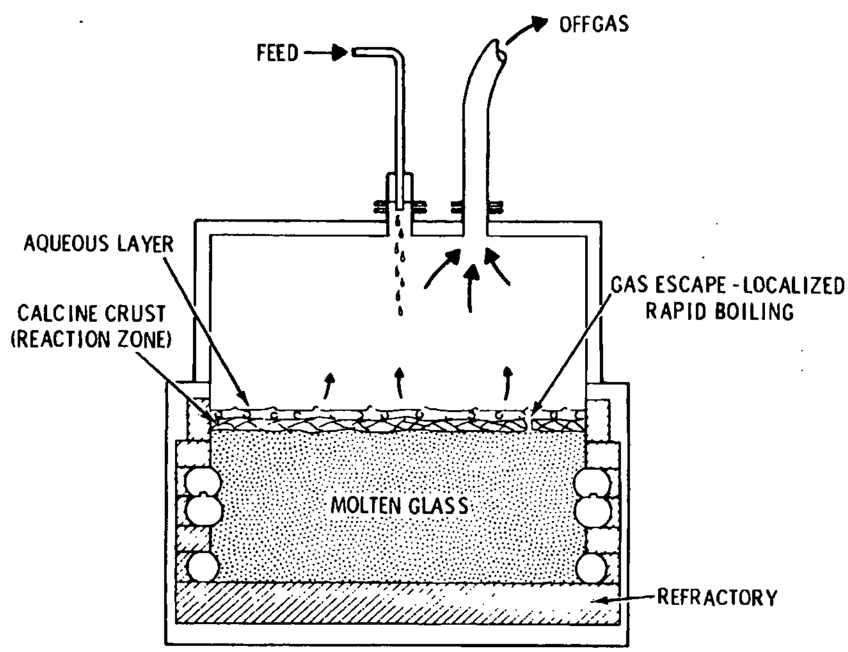


FIGURE 4. Fully Flooded Liquid-Fed Cold Cap

offgas surges in this mode take place as the result of the feed slurry pouring over the edge of the crust onto the glass surface.

The slurry processing capabilities of the melter can be enhanced (or boosted) by using auxiliary heating systems. The primary boosting technique demonstrated to date is heating the cold cap from above with the lid heaters (see Section 3.2.9) and the ionic booster system. The ionic boosters, shown in Figure 6, are metal electrodes that are immersed in the aqueous slurry pool in the cold cap. An alternating electric current, in the same phasing as the power electrodes in the glass, is passed between the ionic booster electrodes creating joule-heat generation in the slurry. The ionic boosting system has increased the capacity of a melter by 50% (Buelt and Chapman 1979), and higher rates are anticipated. The boosters must be water-cooled to prevent film boiling at the slurry-electrode interface. Water-cooling also extends booster life by reducing the electrode temperature, and thereby the corrosion rate, during melter idling periods. The water-cooled ionic booster electrodes can

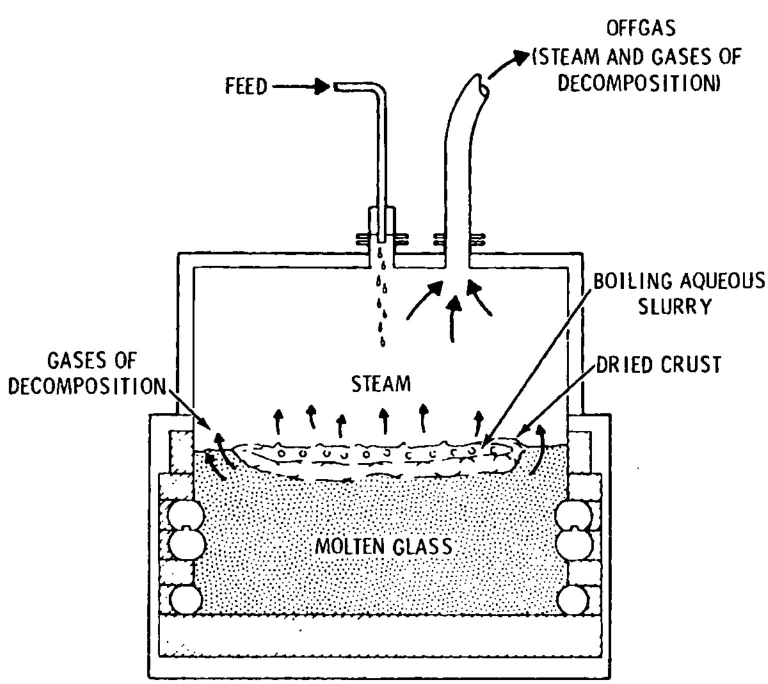


FIGURE 5. Nonflooded Liquid-Fed Cold Cap

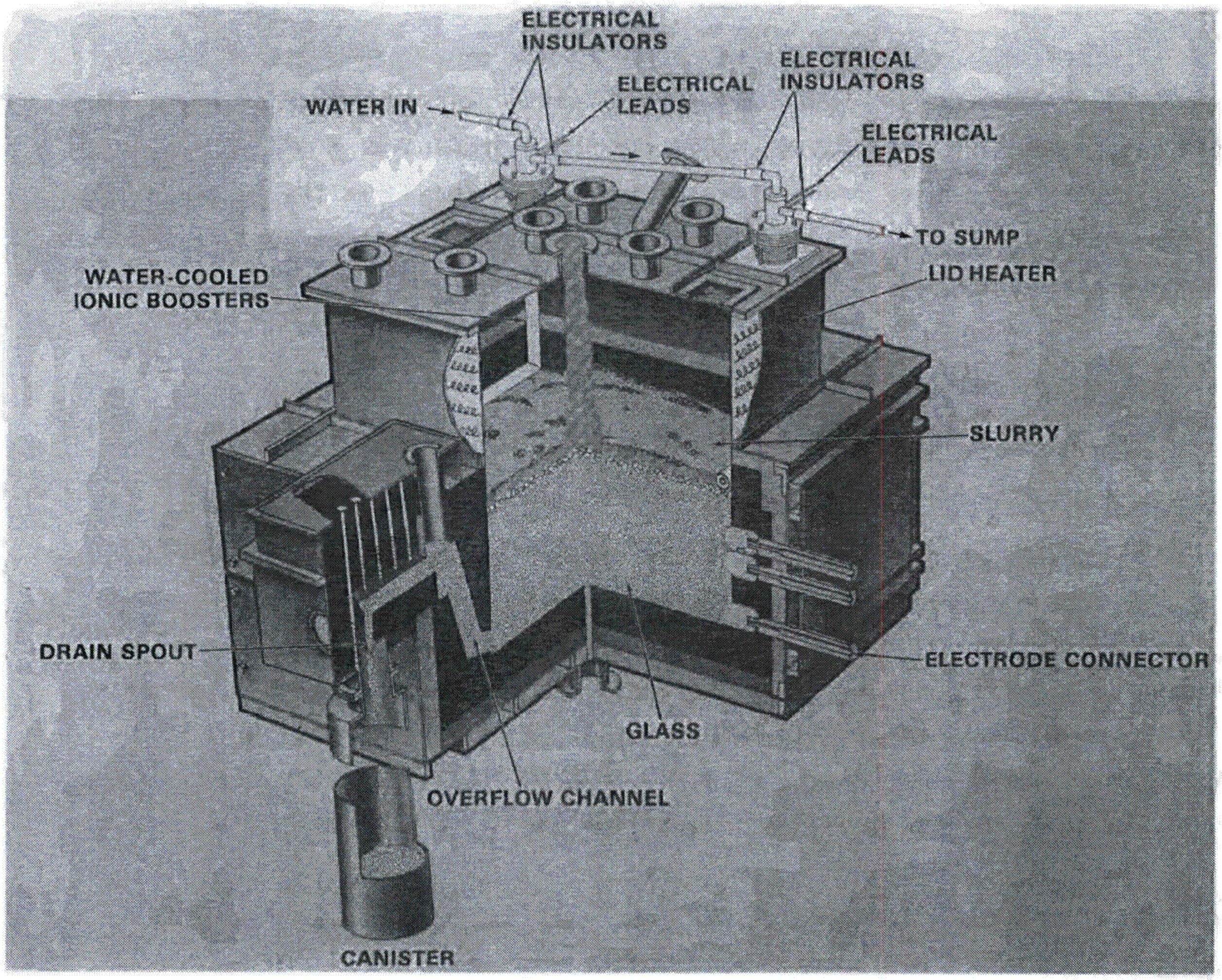


FIGURE 6. LFCM Ionic Booster Electrodes and Lid Heaters

also reduce the melter operating capacity up to 25% during slurry feeding if they are not used. This capacity reduction is the result of the electrodes acting as reflux condensers in the melting cavity.

In the LFCM calcine-feeding mode, the calcine and feed materials are premixed and added to the melter as a single stream. This mixture enters the top of the melter and drops onto the cold cap of solids floating on the molten glass in the melter cavity. Melter operation with calcine feeding is similar to that with slurry feeding, except that there is much less gas generation and a more uniform offgas generation rate without the pressure surges.

Typical flowsheets for the major processing modes are shown in Figures 7 and 8 for defense-waste slurry and defense-waste calcine. Heat balances for typical slurry and calcine feed modes are shown in Figures 9 and 10, respectively. The calculated temperature distributions (based on thermocouple measurements) within the melter during liquid feeding and the idling modes of operation are provided in Section 6.2.

3.2 MELTER DESCRIPTION

The melter components are described below (Buelt and Chapman 1978).

3.2.1 Containment Box

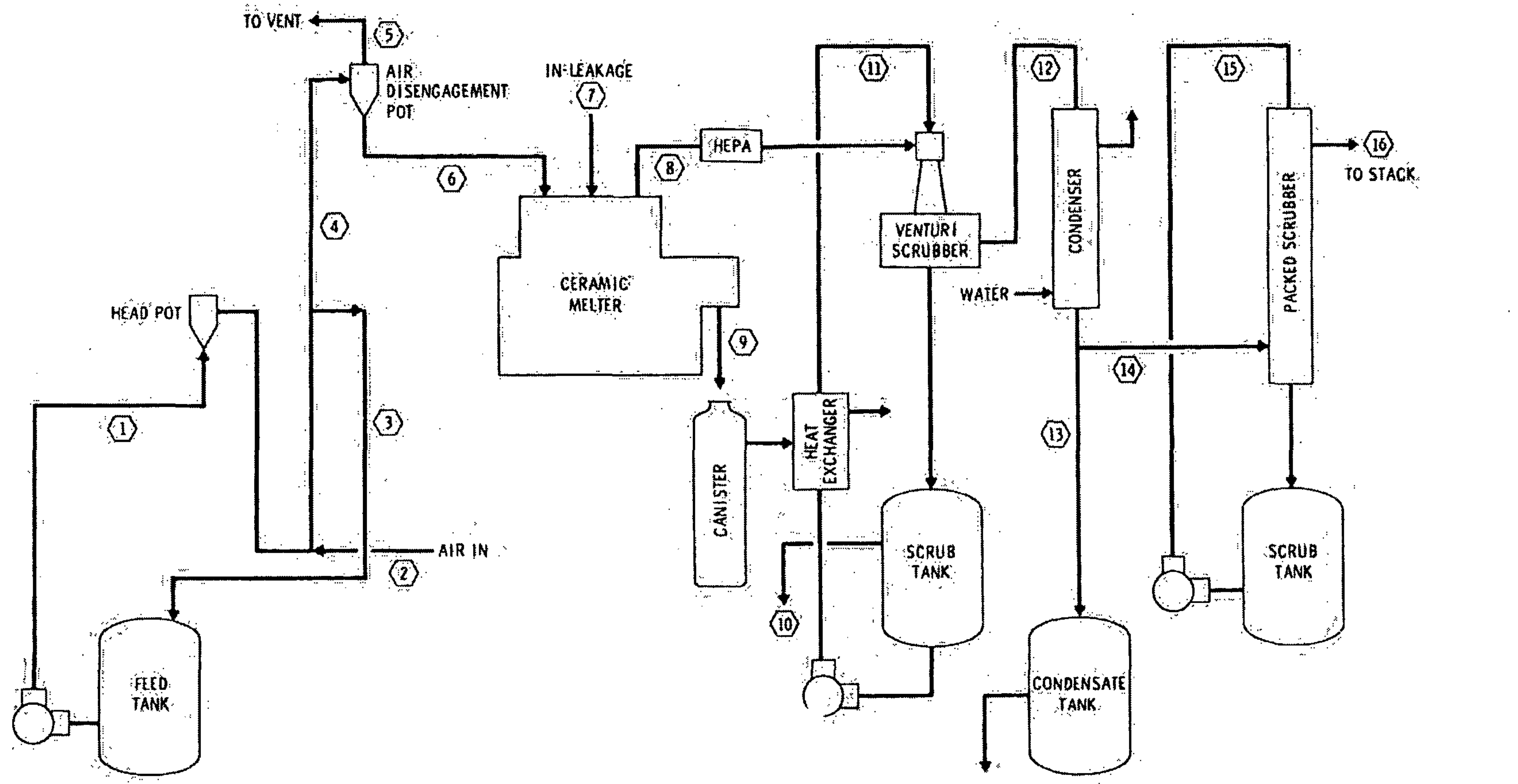
To contain gaseous effluents generated during the vitrification process, an air-tight box encompasses the entire ceramic melter. This is illustrated in Figures 11 and 12. The construction material for the LFCM container is 1/4-in. 304L stainless steel for the container box and 1/4-in. Inconel 601 for the lid. The lower part of the shell, which contains the refractory components, is lined with cooling baffles. Each of these zones can be controlled independently, with either water, steam, or air as the cooling fluid. Aside from keeping the refractories cool, the cooling is intended to solidify the glass as it approaches the outer wall and acts as a second barrier for preventing glass leakage through cracks in the refractory.

3.2.2 Lid

The upper section of the melter container, termed the lid (Figures 11 and 12), provides for de-entrainment of dust or aerosol vapors from the melter effluents. The lid is surrounded by plate heaters, which can be used for melting built-up material that accumulates during liquid feeding. The lid heaters are controlled manually, automatically by temperature control, or are ramped for heatup and cooldown. The cover plate of the lid has various access ports for feeding, viewing, and acquiring data.

3.2.3 Refractory

The molten glass in the LFCM is contained by high-temperature refractories constructed of two basic layers. First, the glass-contact refractory maintains

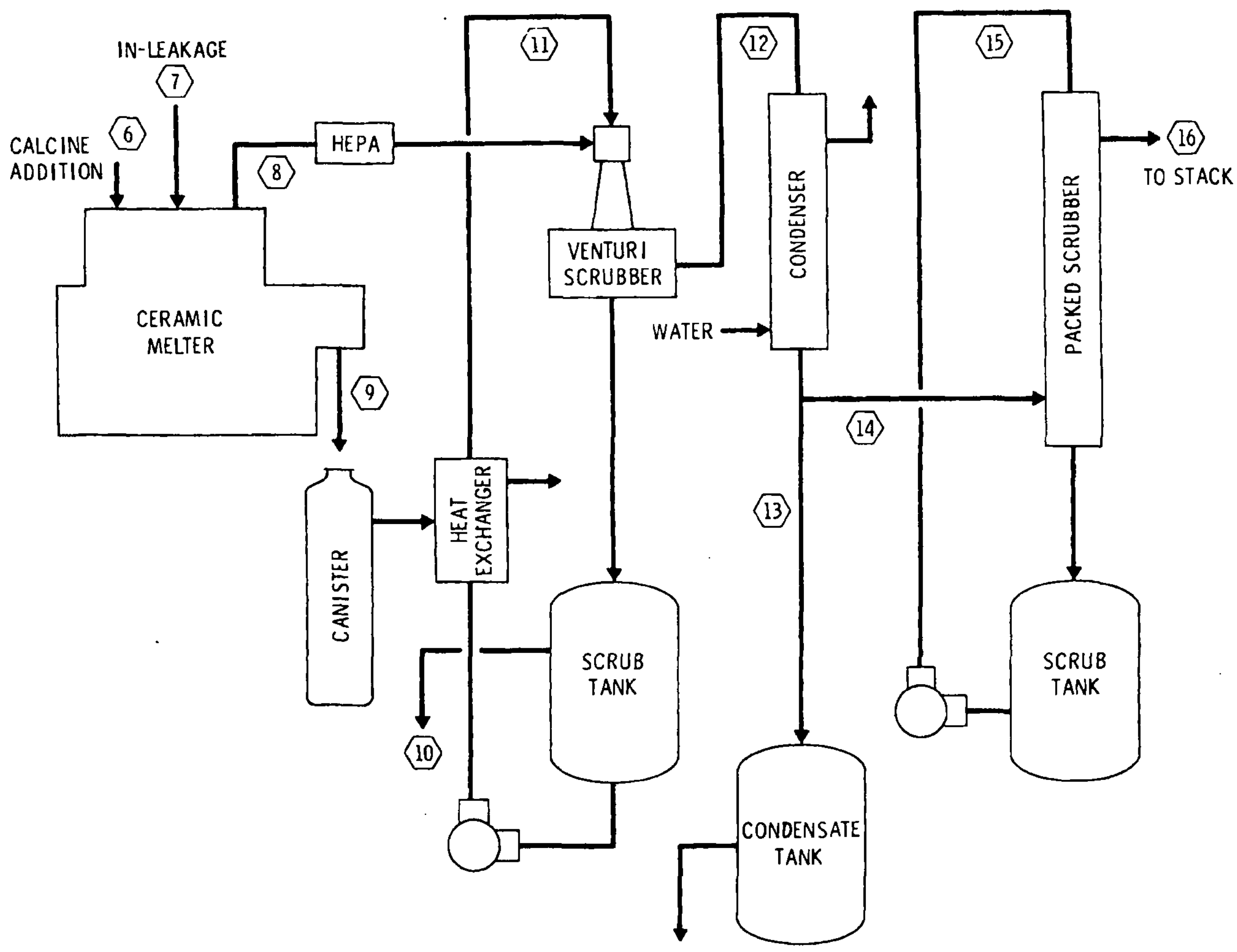


STREAM NUMBER	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
	FEED TO HEAD POT (3)	AIR INLET	RECYCLE	AIRLIFT	AIR OUTLET	LIQUID FEED	IN-LEAKAGE (3)	GAS FLOW	GLASS	SCRUB TANK OVERFLOW	VENTURI SCRUB SOLUTION	VENTURI OUTLET	CONDENSATE	PACKED SCRUB INLET	PACKED SCRUB SOLUTION	OFFGAS TO STACK (5)
LIQUID FLOW (L/h)	300		200	100		100			36.4	35.6	9 E5		0.4		2 E3	
MASS FLOW H_2O (kg/h)	249	NEG.	166	83	NEG.	83	0.42	86.0		85.3	9 E5	0.4	0.4	0.03	2 E3	0.02
MASS FLOW O_2 (kg/h)		0.036		0.036	0.036		6.4	2.22		NEG.	NEG.	2.22	NEG.	2.22	NEG.	2.22
MASS FLOW N_2 (kg/h)		0.12		0.12	0.12		32.4	0.540		NEG.	NEG.	0.540	NEG.	0.540	NEG.	0.540
MASS FLOW CO_X (kg/h) (1)								0.712		NEG.	NEG.	42.7	NEG.	42.7	NEG.	42.7
MASS FLOW NO_X (kg/h) (2)								8.76		2.64	2 E6	6.12	NEG.	6.12	VAR.	6.12

1. ASSUMED 50 mole % CO, 50 mole % CO_2
2. ASSUMED 50 mole % NO, 50 mole % NO_2
3. ASSUMED 20 SCFM
4. ASSUMED DF[a] = 2 FOR NO_2, DF = 1 FOR NO
5. ASSUMES DF = 10 FOR NO_2, DF = 1 FOR NO

(a) DECONTAMINATION FACTOR

FIGURE 7. Defense-Waste Slurry Flowsheet



STREAM NUMBER	1	2	3	4	5	6	7	8	9	10	11
	CALCINE FEED (1)	IN-LEAKAGE (2)	GAS FLOW	GLASS	SCRUB TANK OVERFLOW	VENTURI SCRUB SOLUTION	VENTURI OUTLET (3)	CONDENSATE	PACKED SCRUB INLET	PACKED SCRUB SOLUTION	OFFGAS TO STACK (4)
CALCINE ADDITION (kg/h)	109.6										
LIQUID FLOW (L/h)		0.82	8.53	38.6	7.74	9E5	0.79	NEG.	8.79	2E3	0.79
MASS FLOW H_2O (kg/h)		0.82	8.53			9E3				2E3	
MASS FLOW O_2 (kg/h)		2.07	2.77		NEG.	NEG.	2.77	NEG.	2.77	NEG.	7.77
MASS FLOW N_2 (kg/h)		6.77	6.77		NEG.	NEG.	6.77	NEG.	6.77	NEG.	6.77
MASS FLOW CO_2 (kg/h)		NEG.	0.79		NEG.	NEG.	0.79	NEG.	0.79	NEG.	0.79
MASS FLOW NO_2 (kg/h)		NEG.	0.88		0.44	5E4	0.44	NEG.	0.44		0.04

1. SEE TABLE A2 - SRL-13 FOR FEED COMPOSITION
2. ASSUMED 4 SCFM
3. ASSUMED DF[a] = 2 FOR NO_2
4. ASSUMES DF = 10 FOR NO_2

(a) DECONTAMINATION FACTOR
(b) 100 kg/h

FIGURE 8. Defense-Waste Calcine Flowsheet

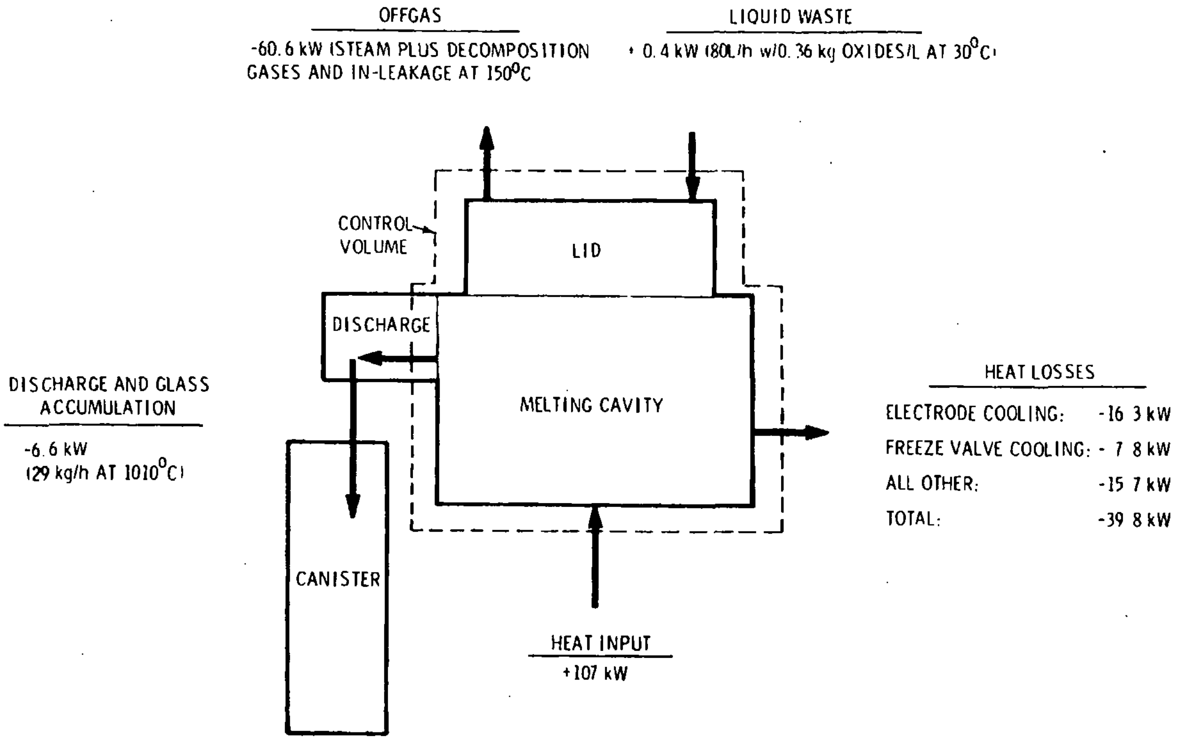


FIGURE 9. LFCM Liquid-Fed Heat Balance

the highest resistance to corrosive attack and contains the molten glass directly. A backup layer of insulating refractory provides higher thermally insulative properties, yet still maintains good corrosive resistance.

The glass contact refractory, shown in Figures 13 and 14, must be:

- resistant to corrosion over long periods of time at operating temperatures (1200°C)
- more electrically resistive than the molten glass to avoid electrical shorting
- thermally insulative
- resistant to thermal shock.

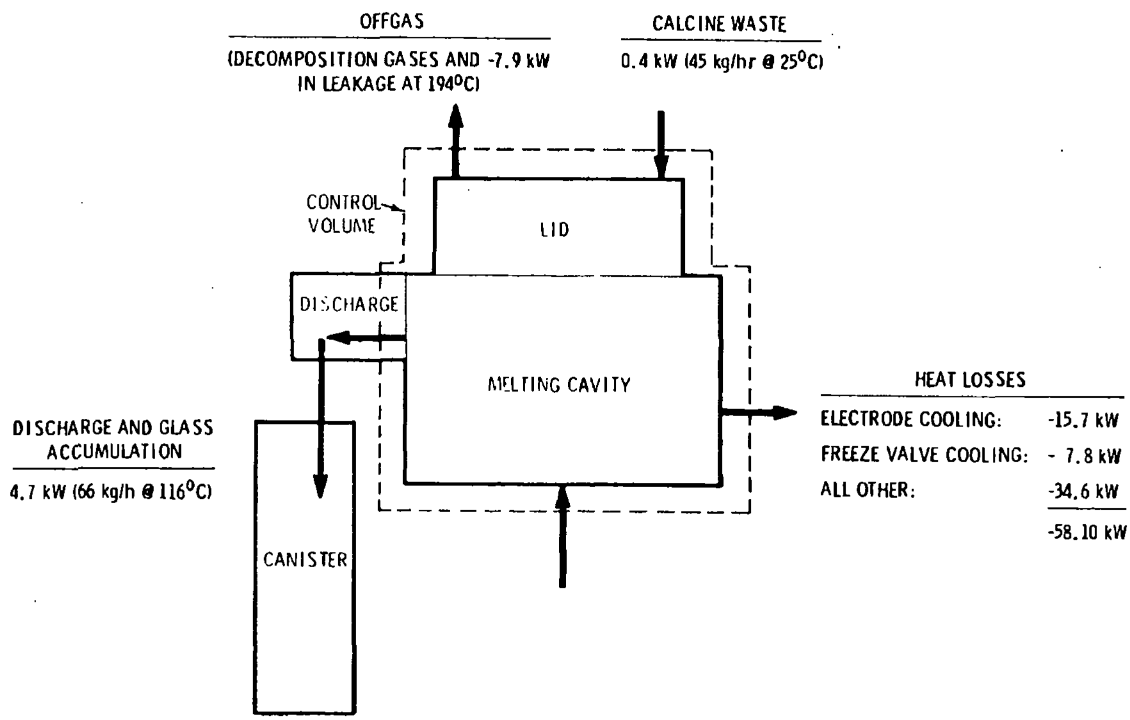


FIGURE 10. LFCM Calcine-Fed Heat Balance

High chromia refractories have excellent resistance to corrosion; however, chromia refractories also have high electrical conductivity properties. For the refractory lining in the LFCM, Monofrax K-3, which contains about 30% chromia and 60% alumina was chosen. The chemical composition and physical properties of the Monofrax K-3 bricks are shown in Table A.1. Typical brick dimensions are 15.2 cm by 30.5 cm by 45.7 cm, although many custom sizes were installed.

Because the Monofrax K-3 is fused-cast, it is very dense and has a fairly high thermal conductivity. Therefore, the walls are backed with 7.62 cm of Alfrax 66 castable refractory (see Figure 15). The chemical composition and properties of the Alfrax 66 are provided in Tables A.1 and A.2. The floor is insulated with a dense Zirmul brick, which has better insulative properties than Monofrax K-3 but still maintains good resistance to molten glass attack. Zirmul brick is also used as the backup refractory behind the electrodes. The chemical composition and physical properties of the Zirmul bricks are provided

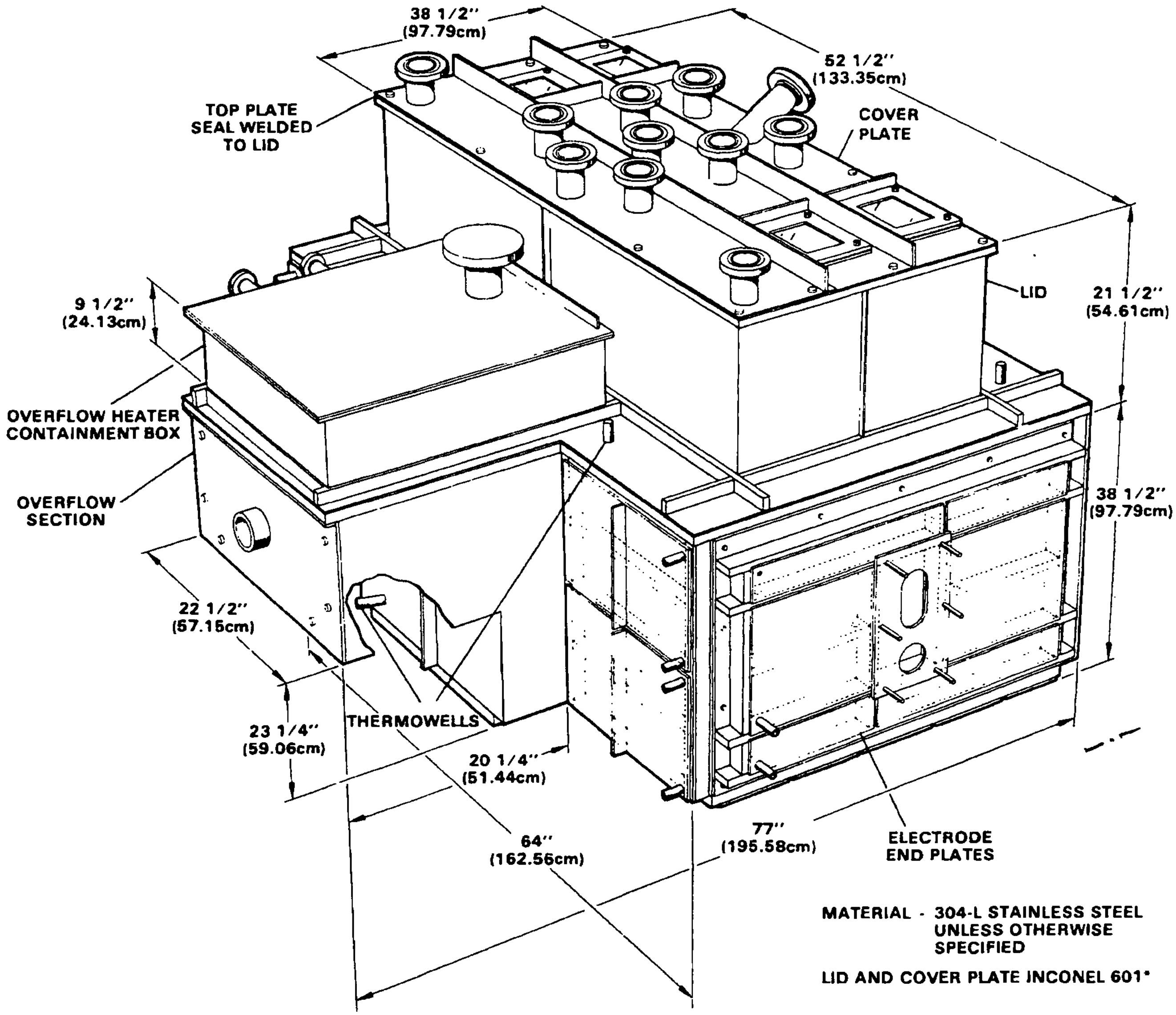


FIGURE 11. Containment Box and Lid, Southeast View

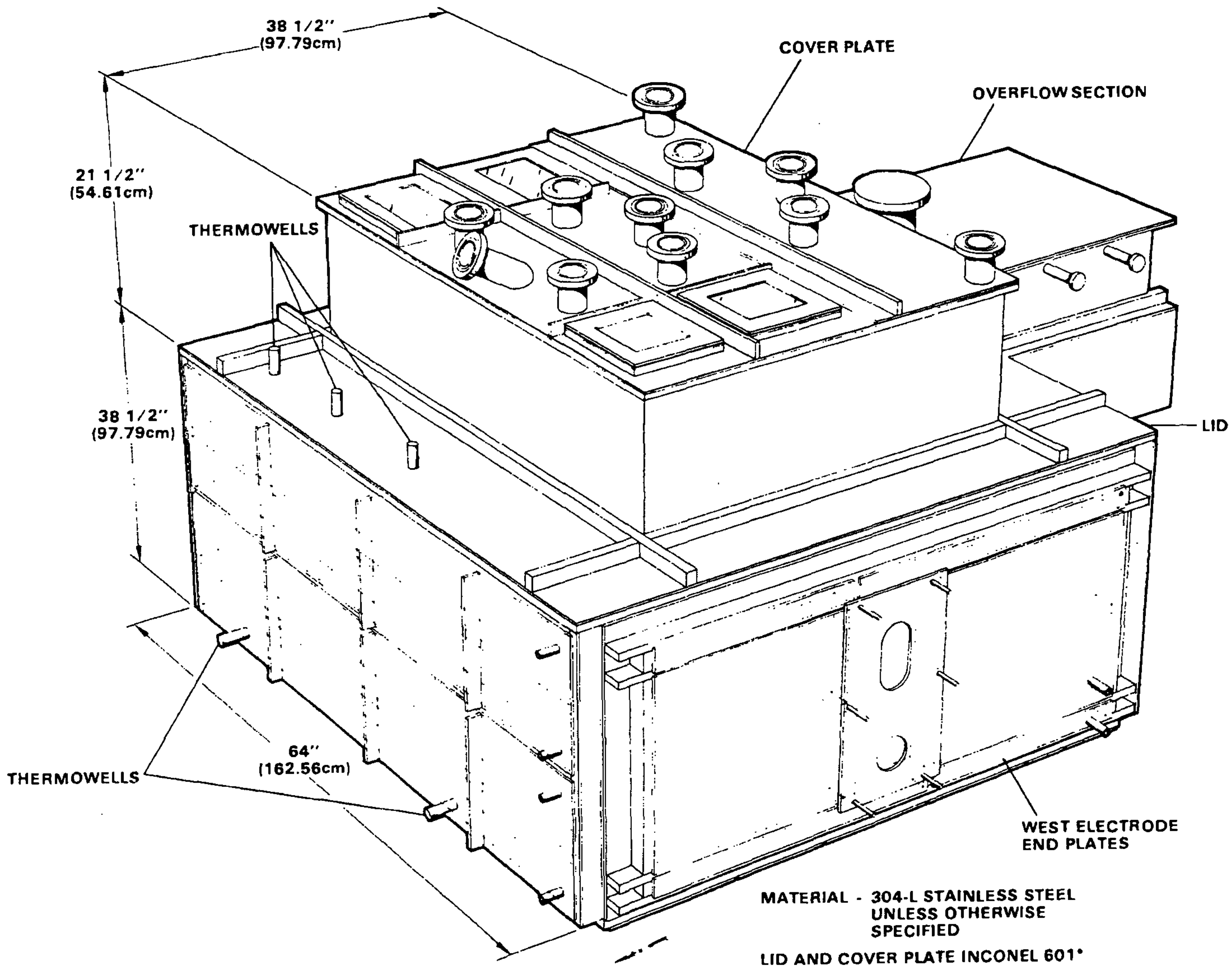


FIGURE 12. Containment Box and Lid, Northwest View

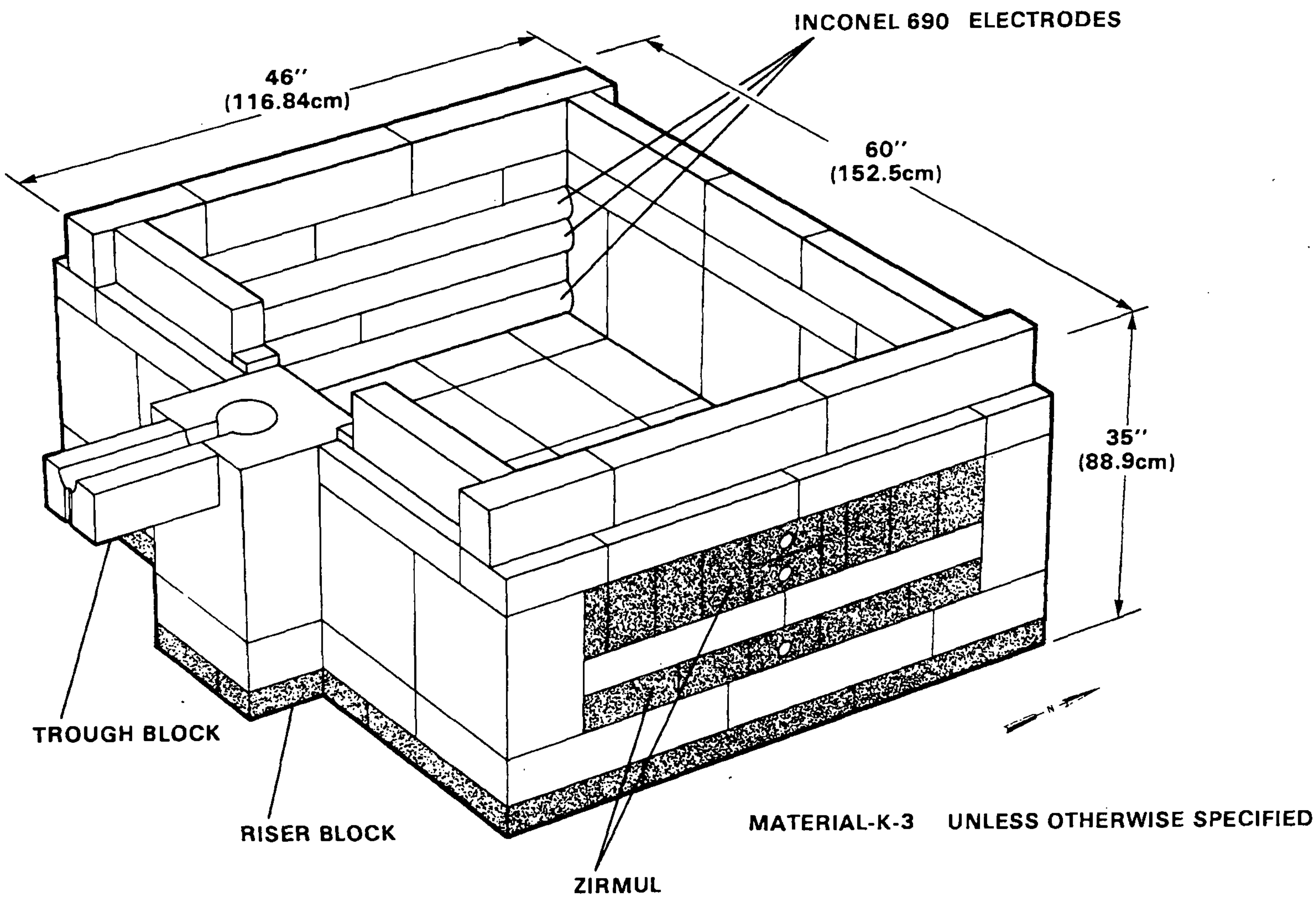


FIGURE 13. Glass Contact Refractory, Southeast View

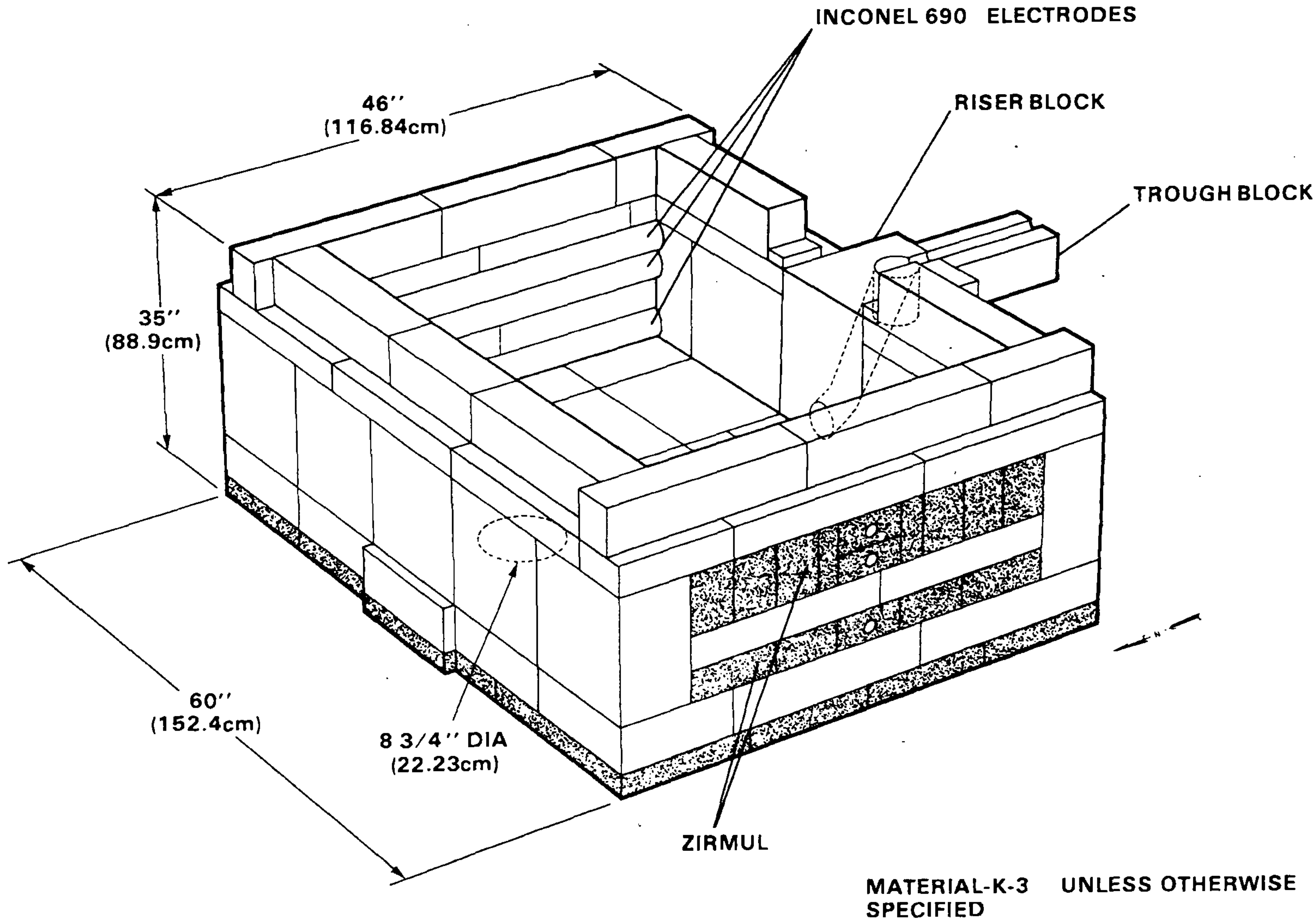


FIGURE 14. Glass Contact Refractory, Northwest View

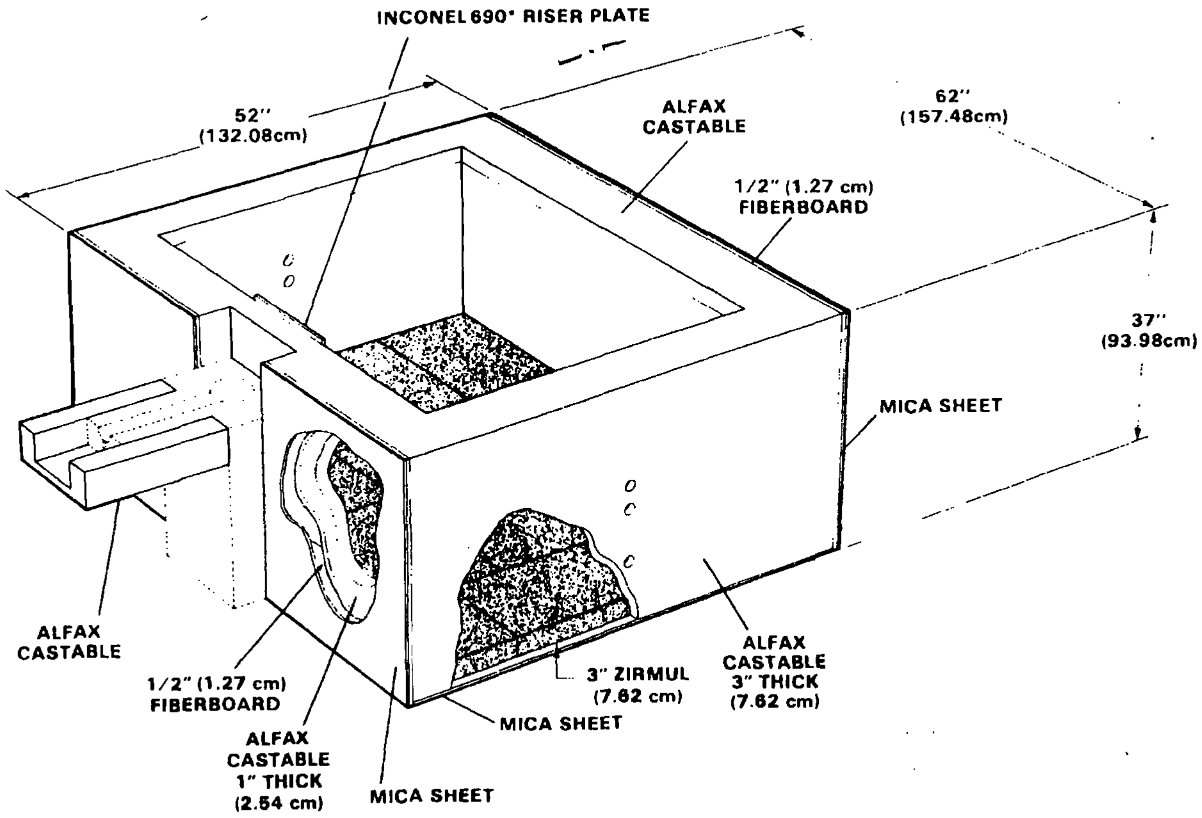


FIGURE 15. Backup and Insulating Refractory Detail

in Tables A.1 and A.2. Typical brick dimensions used were 30.5 cm by 7.6 cm by 45.7 cm. The floor and walls are surrounded by sheets of mica for added electrical protection. Crushable fiberboard, 1.3 cm thick, was placed between the refractory walls and containment box to accommodate the thermal expansion of the refractory during heatup.

3.2.4 Overflow Drain

During operation, the glass flows from near the melting cavity floor, through a riser block, over a trough, and into a receiving canister. The glass drainage is controlled by a tilting mechanism (screw jack), shown in Figure 2. This tilting mechanism operates much the same way as a tea kettle. When the screw jack is raised or lowered, the melter tilts about the pivot point, allowing the glass to drain. Molten glass falls from a pouring spout

made of Inconel 690 through an airtight spool piece into the canister below. More than 100 kg of the glass reservior maintained in the melting cavity may be drained with an $\sim 2^{o}$ tilt angle with this melter.

The overflow section must be heated externally so the glass will remain fluid enough for pouring. The LFCM uses three separately controlled zones of silicon carbide heaters. Each of these zones is controlled independently, either manually or by automatic temperature control. The riser block also has two zones of plate-type heaters located outside the containment box.

3.2.5 Power Electrodes and Control

Inconel 690 was chosen for the LFCM electrodes based on prior good experience with this alloy in other melters. The electrode positions are shown in Figure 16. The electrodes, which were machined out of 5-in.-dia Inconel ingot, are imbedded in the opposing electrode walls. For additional control of glass tank temperatures, the electrodes are arranged in a dual electrode system consisting of an upper and lower set of electrodes, as shown in Figure 16.

The upper electrodes receive their power from a multitapped, 250-KVA, single-phase transformer. The power can be controlled by a manual constant-current or constant-power signal feedback loop. Since electrical resistivity of glass decreases with temperature, constant-current control offers a desirable self-regulating feature because the voltage, and thus power, decreases as the temperature rises. The electrodes can also be controlled by constant temperature, which is read by an internal thermocouple in the electrodes or by an infrared optical pyrometer. The lower electrodes can either be controlled manually or by a ratio of the current feedback signal from the upper electrodes. The actual power to the electrodes is regulated by silicon-controlled rectifiers (SCRs). Figure 17 shows the overall power system component schematic.

3.2.6 Ionic Booster System

The ionic booster system is designed to increase slurry feed throughput by heating the slurry pool directly with the joule effect. The system

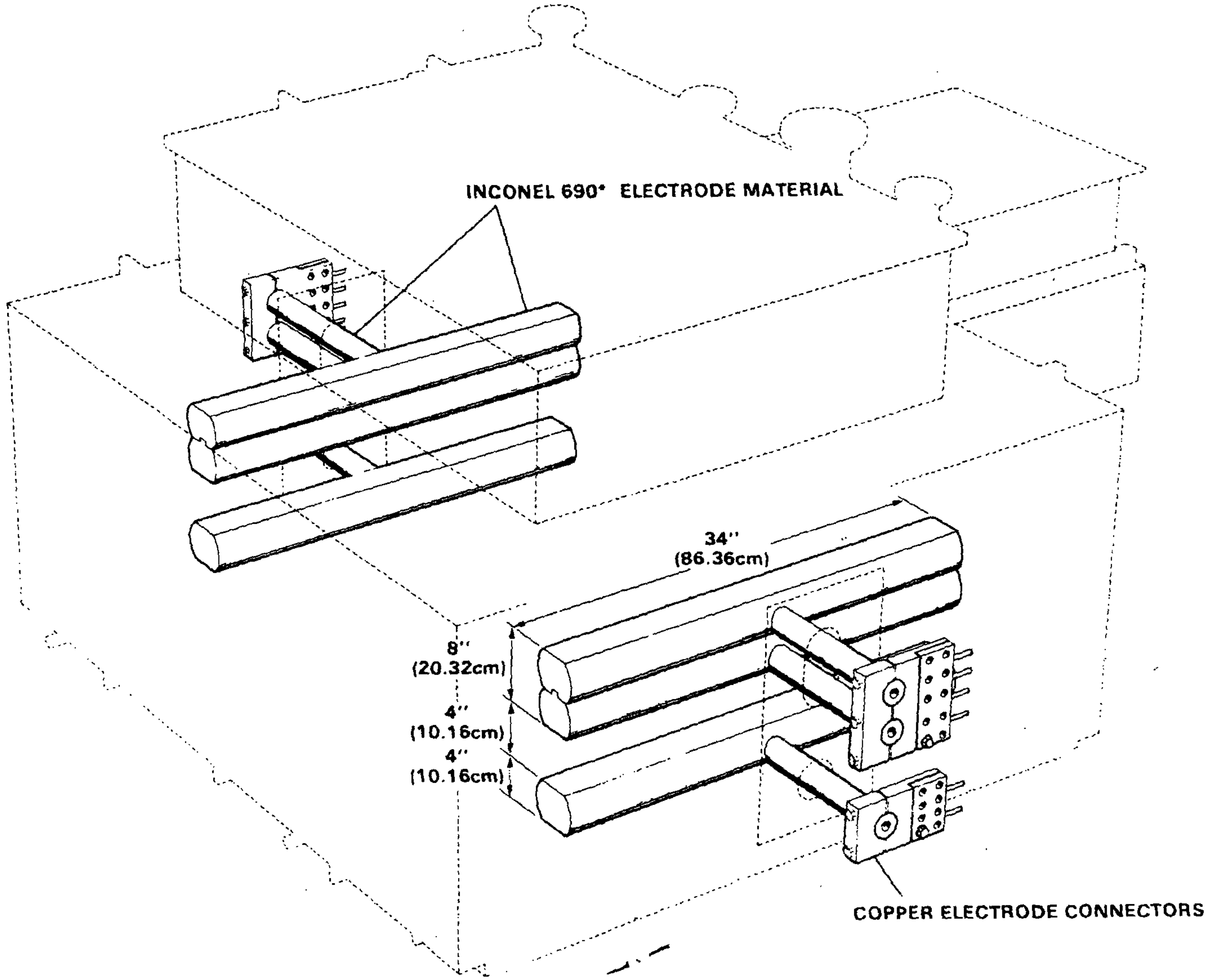


FIGURE 16. Electrode Arrangement

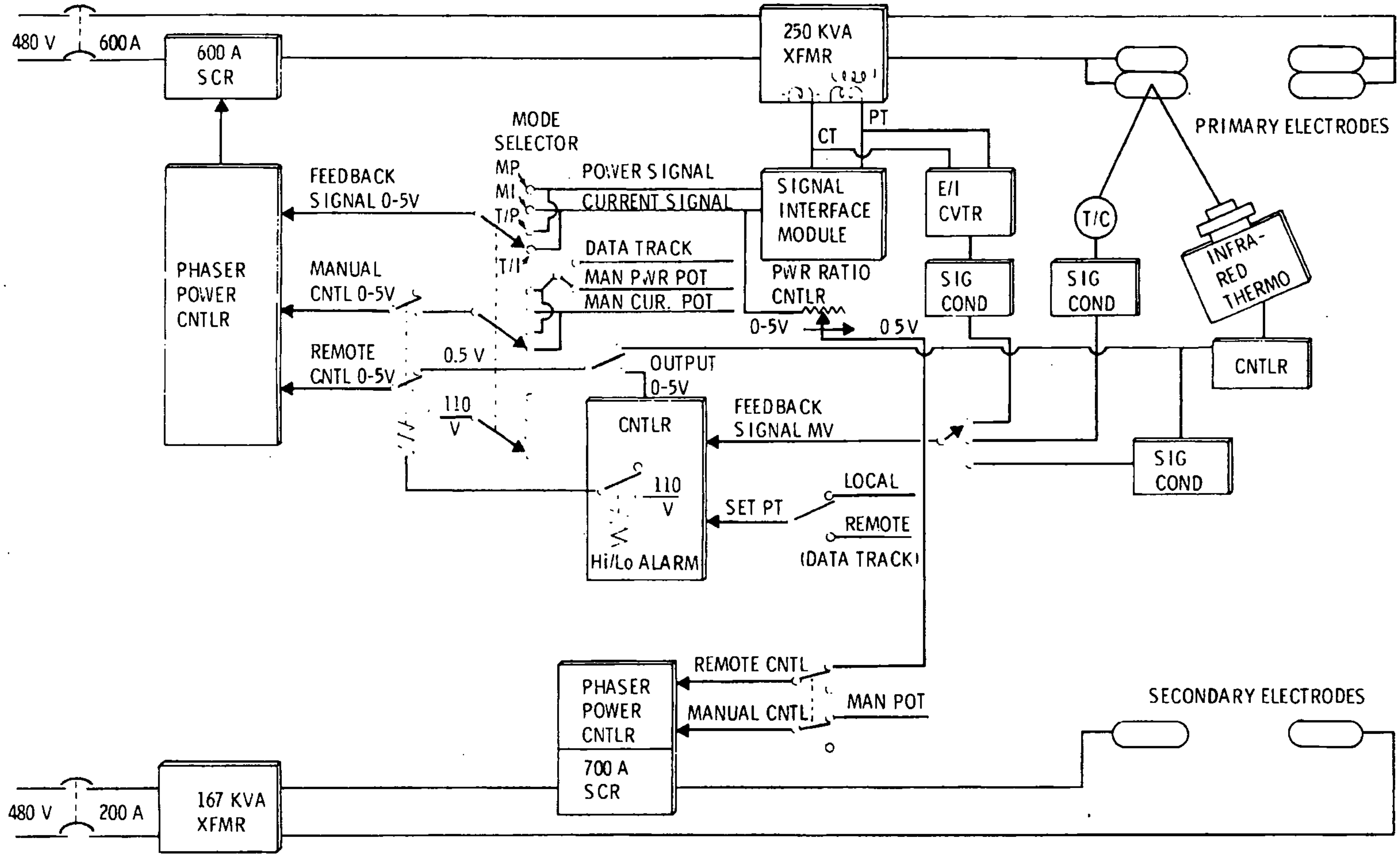


FIGURE 17. Liquid-Fed Melter Electrode Power Control Systems

comprises two water-cooled, stainless steel 304L electrodes with an associated power supply and control system. An electrode is installed at the east and west ends of the melter above the glass, as shown in Figure 6. The ionic booster electrodes are placed in contact with the slurry, and an alternating electric current, in the same phasing as the electrodes in the glass, is passed between them. This generates joule heating directly in the slurry feed.

The electrodes are constructed of 5-cm, schedule-40 pipe. The water coolant passes through the electrodes in series with electrical isolators between the electrodes. Provisions were made for stopping the cooling water flow in the event of a melter pressurization (a possible result of a leak in the ionic booster cooling design, among other causes). The electrodes were designed to be vertically mobile with flexible metal bellows sealing the system to the melter containment.

3.2.7 Offgas System

The gases generated from the vitrification process first pass through a venturi scrubber and then through a packed-column scrubber for removal of nitrates and other effluents. The noncondensibles are then exhausted through a blower to the atmosphere. Figure 18 shows the offgas system used for the LFCM. Vacuum in the melter is controlled by a valve upstream from the offgas blower.

3.2.8 Freeze Valve

A bottom drain was incorporated into the design of the LFCM as an auxiliary drain system and as a method of completely removing the glass from the melter for shutdown purposes. For hot-cell use, the drain must operate remotely. This type of technology is not available in the commercial glass industry. A schematic of the freeze valve is shown in Figure 19.

The freeze valve is an annulus 0.5-cm wide. The annulus is provided with cooling jackets on both the internal and external sides and is also supplied with a heat source. When not in operation, there is a solidified glass plug in the annulus, preventing any glass from flowing. When glass drainage is

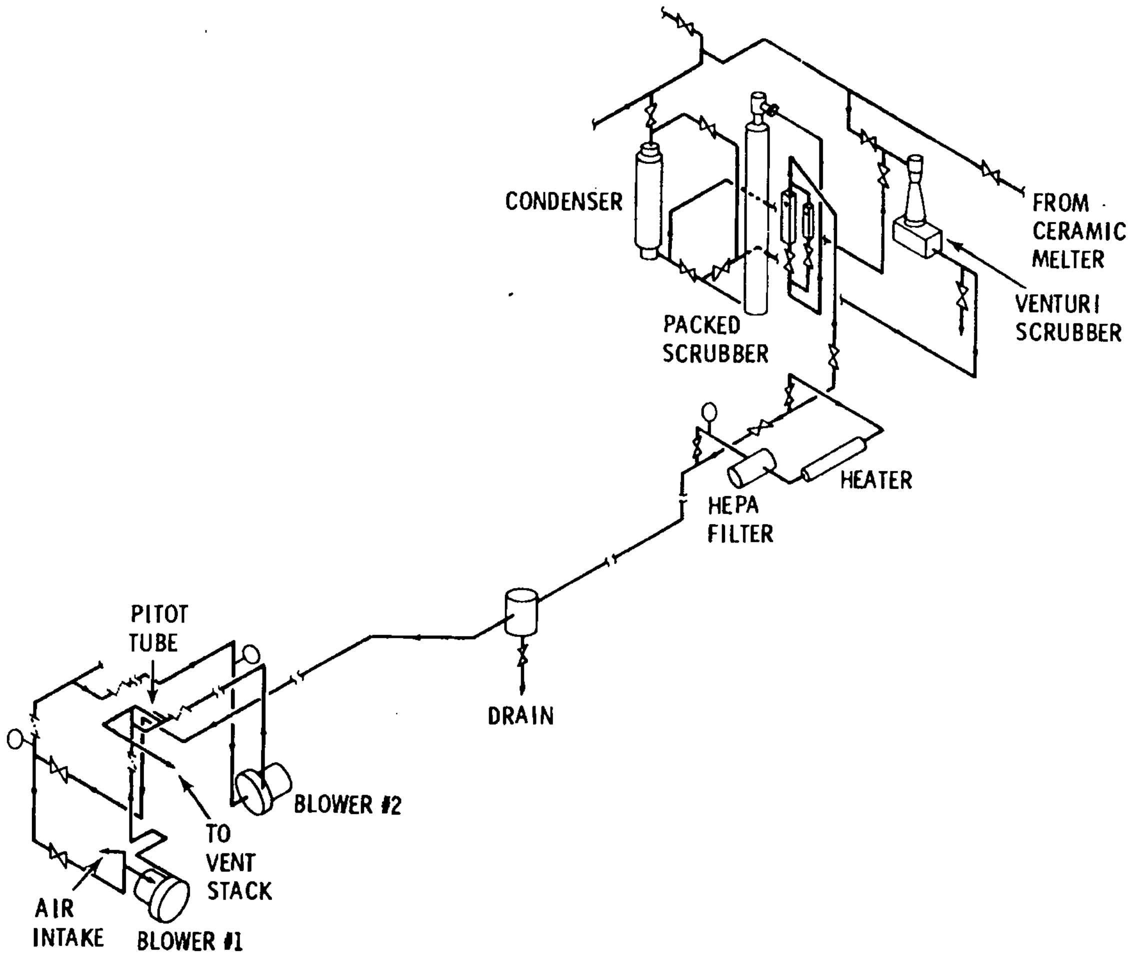


FIGURE 18. Offgas Treatment System

desired, the cooling is turned off and heat is applied. This thaws the solid glass plug and allows the glass to drain. Glass drainage can be controlled and stopped if a cooling fluid is introduced in the cooling jacket. This fluid can be water, steam, or air.

3.2.9 Auxiliary Power Systems

As discussed earlier, certain sections of the LFCM require heat generation in addition to the energy supplied by the power electrodes. The heaters

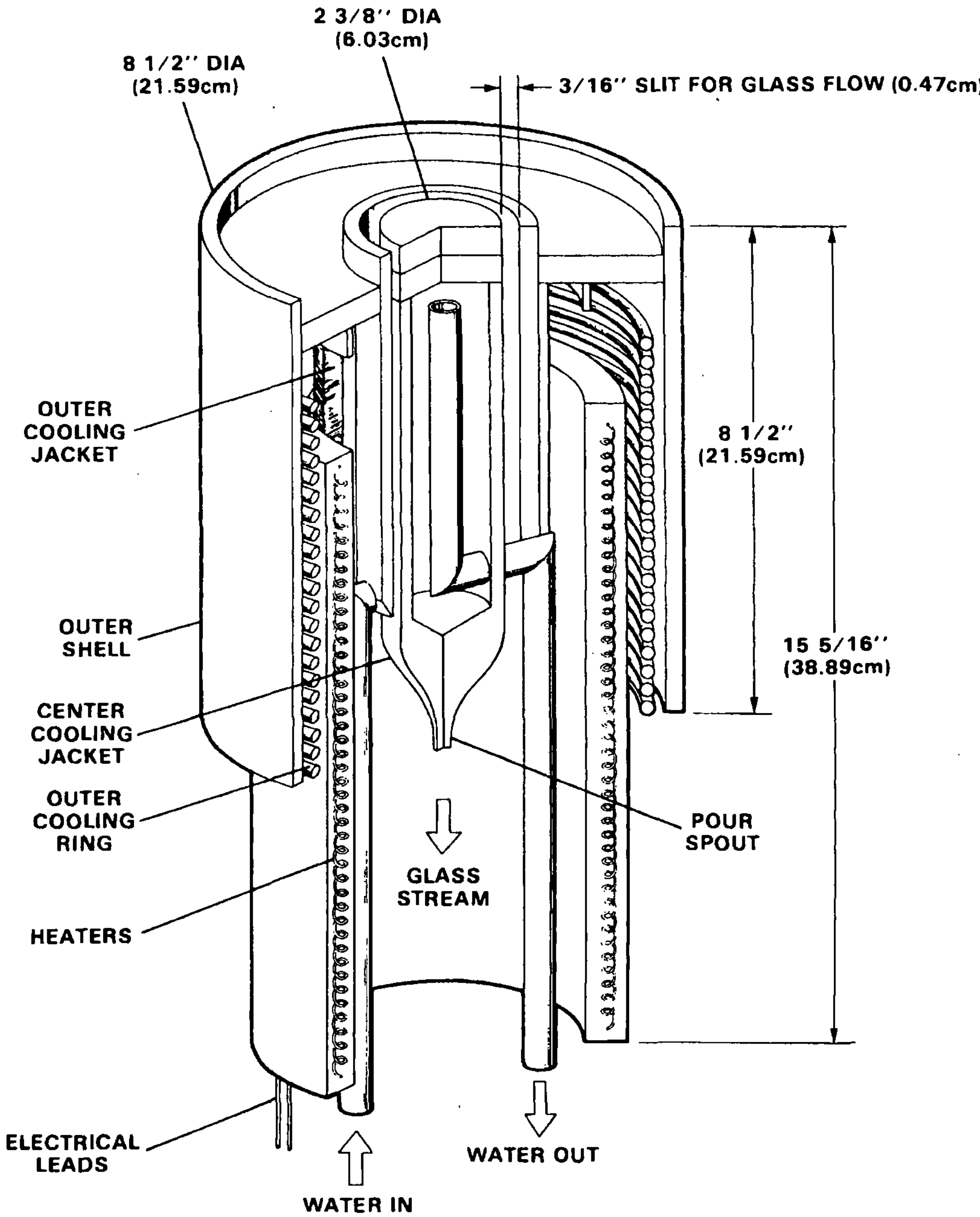


FIGURE 19. LFCM Freeze Valve Detail

are either ceramic plate heaters with resistance heating wire as the heating element, or silicon carbide resistance heaters. Their locations and type are shown in Figure 20 and are identified below:

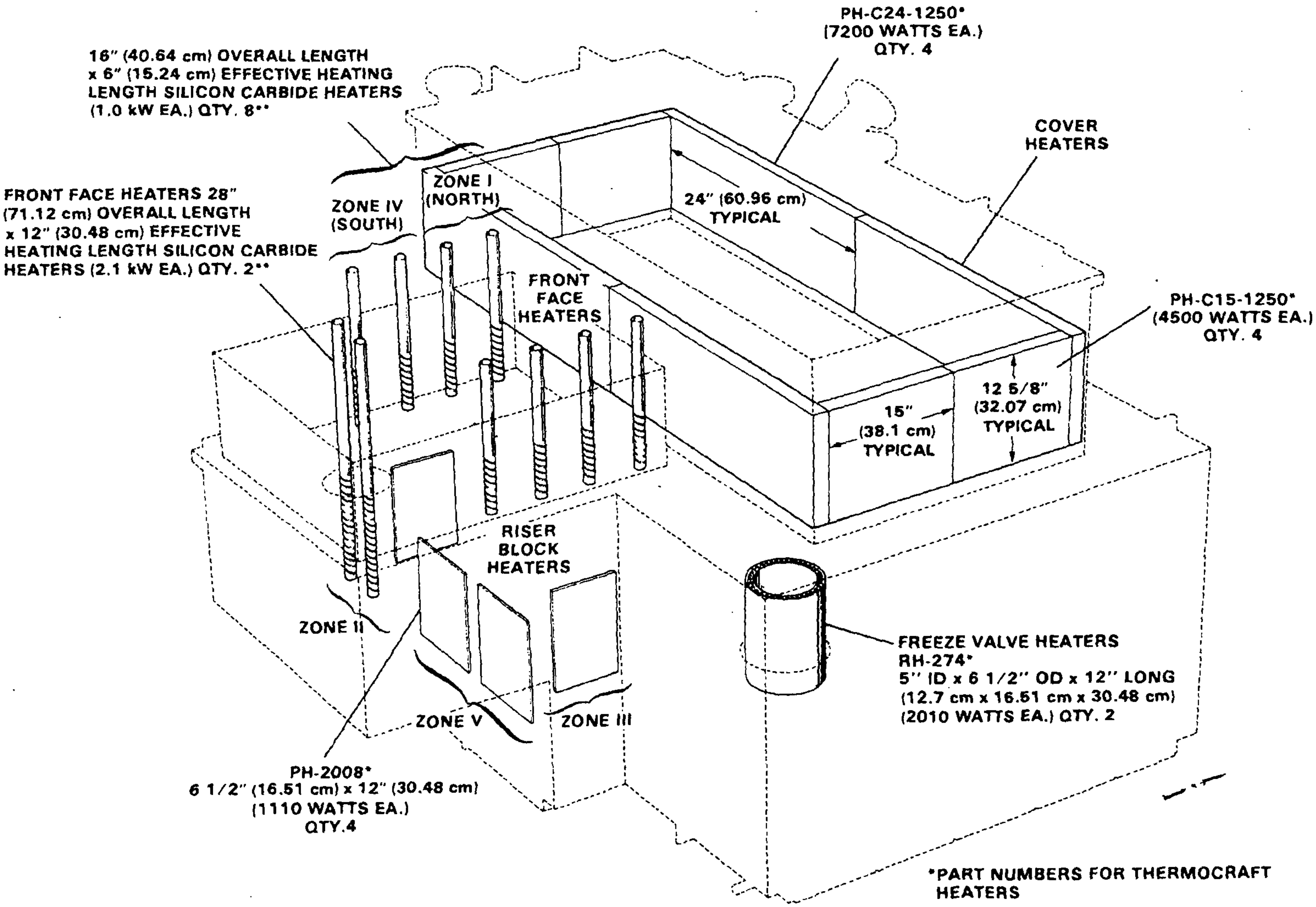


FIGURE 20. Auxiliary Heating Systems

- overflow heaters, Zones I, II, and IV
- riser block heaters, Zones III and V
- lid heaters
- bottom drain heaters.

The lid heaters were used for the initial melter startup and could be used to improve the slurry-fed glass production rate by radiative heating of the cold cap from above.

3.2.10 Instrumentation

The LFCM has been constructed with instrumentation for extensive data acquisition. Temperatures are continually monitored and recorded for the electrodes, for various locations within the refractory floor and walls, amid the auxiliary heaters in the overflow, on the lid, and on the riser section. Thermocouples are also located in the bottom drain and on the receiving canisters. Cooling flow to the various sections of the melter is also monitored for temperature and flowrate. In addition, the gross canister weight is continually recorded for monitoring the glass-production rates.

3.2.11 Feed Systems

Because of the different operating modes of this melter, both liquid and calcine feeding systems are required. Simulated liquid waste is metered by an air lift from a head tank pot up to an air/liquid disengagement chamber. From that chamber, the liquid flows by gravity to the melter, as shown in Figure 21.

During calcine feeding, solids are fed from a volumetric feeder through a central flange in the cover plate of the melter. During the early tests with the LFCM, solids were introduced near the rear wall of the melter and were distributed over the entire surface with a batch distributor (Chapman et al. 1979). This distributor system process was eliminated to reduce the amount of moving parts in the calcine-fed mode.

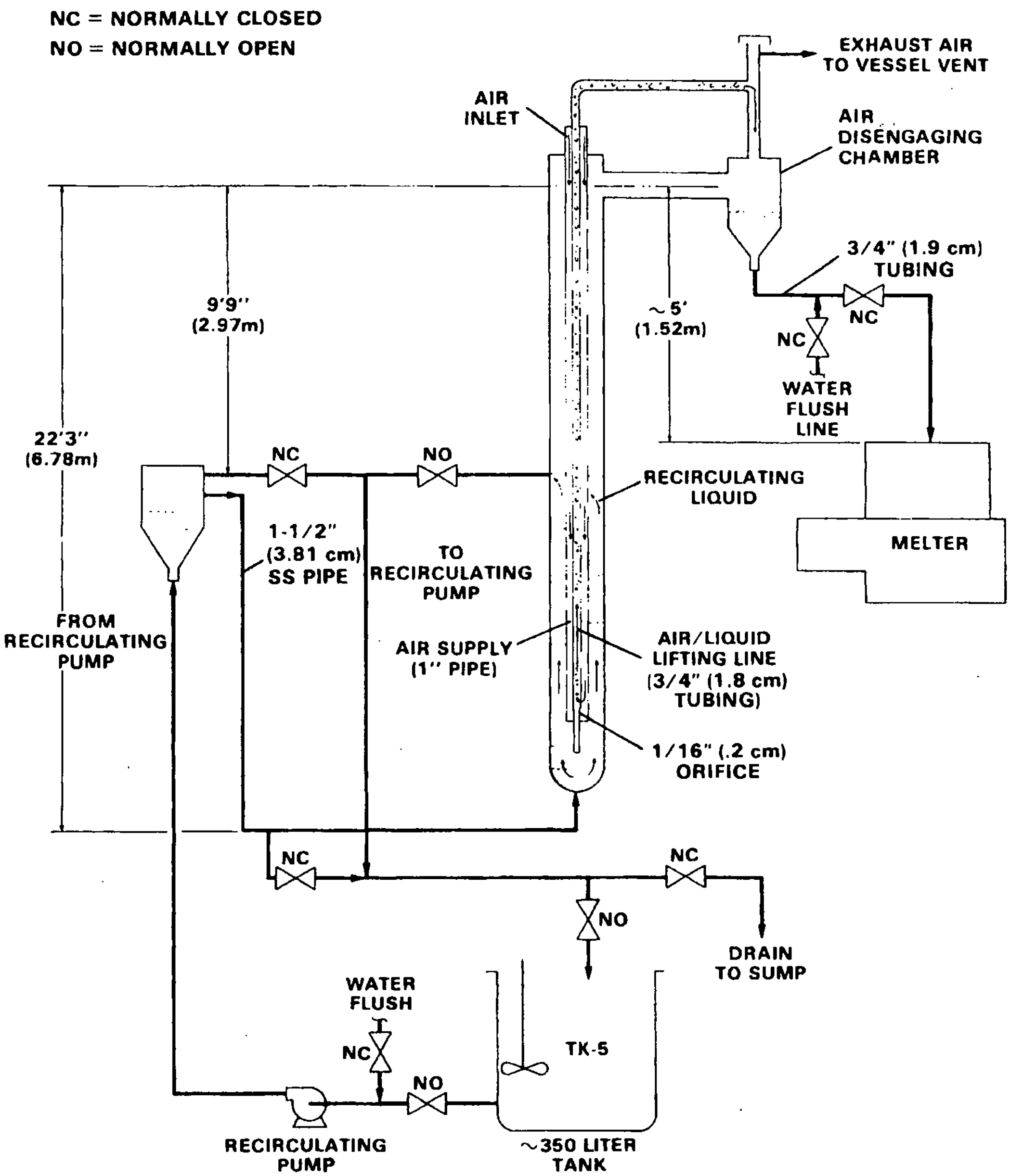


FIGURE 21. Liquid Feed System

4.0 OPERATING EXPERIENCE

Construction of the LFCM was completed in February 1977. The melter was started up by filling it with premelted glass granules (cullet). Using the lid heaters to melt sodium hydroxide placed on the cullet surface, joule-heating was initiated. The melter was heated to operating temperature over a 22-h period and was operated continuously, either in the processing or idling mode, until it was shut down in February 1980 for modifications and examination. Thus, the melter was in operation for about three years.

During its operating life, 19,000 L of slurry and 54,000 kg of calcine with glass formers were fed to the melter. This produced 80 tonnes of glass with about 780 operating hours under feeding conditions. A summary of the melter operation is presented in Appendix B.

Specific operating characteristics of the LFCM (and ceramic melters in general) during glass-production testing has been well documented in earlier reports, and is beyond the scope of this report. The documents by Brouns et al. (1980) and Chapman et al. (1979) are recommended for the reader interested in specific melter operational data.

The following are significant events that occurred during the operating life of the melter that may have affected the condition of the melter, or are notable from a design/operation viewpoint:

- rapid initial startup--The LFCM was initially brought to operating status in about 22 h. This rapid heatup rate exceeds the recommended temperature-increase rate for the refractories and probably produced excessive thermal stresses in the refractory layers.
- overflow-drain section rapid heatup--The overflow-drain refractory was also heated rapidly to operating temperature on several occasions following the replacement of failed silicon carbide heating elements. As above, rapid temperature changes can produce damaging thermal stresses.

- foaming events--The melter refractory also was exposed to thermal shock conditions during periods of glass-foam generation. Although glass foam is generated by several mechanisms (Blair and Lukacs 1980), foam generation following stable cold-cap development would expose the cool Monofrax K-3 refractory above the cold cap to high-temperature glass. Depending on the initial Monofrax K-3 surface temperature, this could produce refractory heating much faster than the $<40^{o}C/h$ recommended by the Monofrax K-3 manufacturer.

- slurry feed initiation transients--Typical melting-cavity temperature decreases of $150^{o}C/h$ have been measured during slurry feeding initiation, and maximum rates of $\sim300^{o}C/h$ have been recorded. Audible cracking has been reported at these high rates of refractory temperature change.

- power outages--The LFCM operating power was suspended for many short periods, and two extended power outages (maximum of 17 h) were encountered. On each occasion, the melter was returned to operating status by joule-heat generation in the melting cavity by the electrodes.

- elimination of melter containment-box cooling--The melter-containment-shell water cooling was discontinued (with the exception of the electrode walls) in August 1979. The temperature profile changes in the refractory may have affected the refractory cracking and produced the bowing observed in one of the coolant channels (Section 5.1).

- coolant interruptions of the ionic booster electrodes--The coolant for the ionic booster electrodes was interrupted on two occasions, producing $200^{o}C$ higher plenum temperatures than normal. These elevated temperatures may have increased the warpage of the melter lid and cover plate.

- melting cavity pressurization--The melting cavity pressure varies during glass production testing because of changing offgas production, especially during slurry feeding. Routine minor pressure variations were observed, and one event produced ~3.7 MPa melting cavity pressure. Large pressure variations may affect the mechanical deformation of the melter lid.