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TRANSMITTAL OF THE VITRIFICATION PILOT PLANT MELTER INCIDENT FINAL REPORT

03/05/97

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REPORT

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Department of Energy

Ohio Field Office
Fernald Area Office
P. O. Box 538705
Cincinnati, Ohio 45253-8705
(513) 648-3155

MAR 05 '97

DOE-0626-97

Mr. James A. Saric, Remedial Project Director
U.S. Environmental Protection Agency
Region V - 5HSF-5J
77 W. Jackson Boulevard
Chicago, Illinois 60604-3590

Mr. Tom Schneider, Project Manager
Ohio Environmental Protection Agency
401 East 5th Street
Dayton, Ohio 45402-2911

Dear Mr. Saric and Mr. Schneider:

TRANSMITTAL OF THE VITRIFICATION PILOT PLANT MELTER INCIDENT FINAL REPORT

The purpose of this memorandum is to transmit the *Vitrification Pilot Plant Melter Incident: Final Report*. This report includes the analysis, conclusions, and recommendations of the three teams assembled to examine the key aspects of the incident -- root cause, safety practices, and path forward. These teams included individuals from the Department of Energy, Fernald Environmental Management Project (DOE-FEMP), Department of Energy, Ohio Field Office (DOE-OH), Flour Daniel Fernald (FDF), and technical experts from private industry and other DOE sites. The Independent Technical Review Team, assembled to evaluate the overall path forward for the remediation of the waste residues in the silos, was provided the information in this report as it was generated to assist in their evaluation.

More specifically, this report provides a detailed account of how and why the melter leakage occurred. Further, the safety review provided in this report shows that the Vitrification Pilot Plant (VitPP) operated in full compliance with the safety basis documentation; however, potential safety concerns resulting from deterioration of the melter were not factored into revisions of the safety basis documentation. Finally, this report recommends completing the remainder of Phase I testing and Phase II testing off-site. Therefore, it is recommended that the VitPP be placed in a safe condition, and be used to support the waste retrieval demonstration, new radon treatment system, and be salvaged where appropriate.

Recycled and Recyclable

As we agreed per the weekly teleconference call, DOE recommends that the transmittal of this report to be utilized as the Interim Phase I report until such time the remaining Phase I data is gathered.

If you have any questions or comments concerning this report, please contact Nina Akgunduz at (513) 648-3110, or me at (513) 648-3139.

Johnny W. Reising
Fernald Remedial Action
Project Manager

FEMP:Yockman

Enclosure: As Stated

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United States Government

Department of Energy

Fernald Area Office

memorandum

DATE: MAR 03 '97

REPLY TO ATTN OF: DOE-0629-97

FEMP:Yockman

SUBJECT: TRANSMITTAL OF THE VITRIFICATION PILOT PLANT MELTER INCIDENT FINAL REPORT

TO: Sharon Fauver, EM-42, CLOV

The purpose of this memorandum is to transmit the *Vitrification Pilot Plant Melter Incident: Final Report*. This report includes the analysis, conclusions, and recommendations of the three teams assembled to examine the key aspects of the incident -- root cause, safety practices, and path forward. These teams included individuals from the Department of Energy, Fernald Environmental Management Project (DOE-FEMP), Department of Energy, Ohio Field Office (DOE-OH), Flour Daniel Fernald (FDF), and technical experts from private industry and other DOE sites. The Independent Technical Review Team, assembled to evaluate the overall path forward for the remediation of the waste residues in the silos, was provided the information in this report as it was generated to assist in their evaluation.

More specifically, this report provides a detailed account of how and why the melter leakage occurred. Further, the safety review provided in this report shows that the Vitrification Pilot Plant (VitPP) was operated in full compliance with the safety basis documentation; however, potential safety concerns resulting from deterioration of the melter were not factored into revisions of the safety basis documentation. Finally, this report recommends completing the remainder of Phase I testing and Phase II testing off-site. Therefore, it is recommended that the VitPP be placed in a safe condition, and be used to support the waste retrieval demonstration, new radon treatment system, and be salvaged where appropriate.

The DOE-FEMP, in consultation with the regulators and stakeholders, is using the input and recommendations from both the Melter Incident Report and Independent Technical Review Team to develop a path forward strategy for the remediation of the Silos and also as a resource for the ongoing avoidable cost review being conducted by DOE.

If you have any questions or comments concerning this report, please contact Nina Akgunduz at (513) 648-3110, or me at (513) 648-3101.

Glenn Griffiths

for Jack R. Craig
Director

Attachment: As Stated

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Recycled and Recyclable

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Report No. 40100-RP-00019

VITRIFICATION PILOT PLANT MELTER INCIDENT: FINAL REPORT

FEBRUARY 1997

INFORMATION ONLY

Fernald Environmental

FERMCO
Restoration Management Corporation

(now renamed Fluor Daniel Fernald)
P. O. Box 538704 Cincinnati, Ohio 45253-8704 (513) 648-3000

February 26, 1997

Fernald Environmental Management Project
Letter No. C:OOTP:97-0090

Mr. Jack R. Craig, Director
Department of Energy
Fernald Environmental Management Project
P. O. Box 538705
Cincinnati, Ohio 45253-8705

Dear Mr. Craig:

CONTRACT DE-AC24-92OR21972, VITRIFICATION PILOT PLANT MELTER INCIDENT FINAL REPORT

Please find enclosed, in response to the December 26, 1996 Vitrification Pilot Plant Melter Incident, the Melter Incident Final Report. The report is the result of the investigations of the three teams convened to study the melter incident:

- Safety Review Team
- Data Analysis and Path Forward Team
- Incident Analysis Review Team

The Final Report includes two key components: (1) the main report, which provides an overview of the incident and a summary of each team's report; and (2) three appendices, which provide each team's report in full.

If you have any questions, please contact Robert P. Heck at 648-3051.

Sincerely,

Robert P. Heck, II for
John C. Bradburne
President

RPH:DAN:kdg
Enclosure

c: Nina Akgündüz, DOE-FEMP, MS45
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Project #40100 (Melter Incident Final Report)

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Report No. 40100-RP-00019

VITRIFICATION PILOT PLANT MELTER INCIDENT: FINAL REPORT

FEBRUARY 1997

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Attachment A - Vitrification Pilot Plant Incident Analysis Team Report
Attachment B - Vitrification Pilot Plant Safety Review Team Report
Attachment C - Vitrification Pilot Plant Data Analysis and Path Forward Team Report

Acronyms

AEDO	assistant emergency duty officer
ARARs	applicable or relevant and appropriate requirements
AZS	alumina-ziconia-silica
C	Celsius or centigrade
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
DOE	U.S. Department of Energy
EPA	U.S. Environmental Protection Agency
EPC	engineering, procurement and construction
FDF	Fluor Daniel Fernald
FEMP	Fernald Environmental Management Project
IRT	Independent Review Team
IVB	invitation for bid
kg	kilogram
MT/d	metric tons per day
OEPA	Ohio Environmental Protection Agency
PNNL	Pacific Northwest National Laboratory
RFP	request for proposal
ROD	Record of Decision
SCR	silicon control rectifier
TCLP	Toxicity Characteristic Leaching Procedure
VITPP	Vitrification Pilot Plant
VSL	Vitreous State Laboratory

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EXECUTIVE SUMMARY

This report was prepared in response to an incident involving a leak of non-radioactive glass from the vitrification melter at the Fernald Environmental Management Project (FEMP) on December 26, 1996. This report summarizes the reports of the three teams convened to investigate the incident; it includes a path forward for the Vitrification Pilot Plant (VITPP) Test Program at the FEMP, a U.S. Department of Energy (DOE) facility located northwest of Cincinnati, Ohio.

The VITPP contains the vitrification melter and systems for product-forming and handling, off-gas treatment, melter feed preparation and transfer, wastewater treatment, process support auxiliaries, and a control room. The VITPP melter and support systems were designed to produce approximately one metric ton of glass per day. The VITPP is the third and final tier of an exhaustive treatability program, culminating in the design of facilities and equipment for final remediation of Silos 1, 2, and 3 material. Silos 1 and 2 contain K-65 material (radium-bearing, low-level radioactive waste); Silo 3 contains dry, radioactive metal oxide waste (Silo 4 was never used).

Immediately following the incident, Fluor Daniel Fernald (FDF), the DOE contractor managing remediation of the facility under the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA), initiated two tiers of response: (1) emergency response measures were taken immediately; and (2) three teams were formed to study key aspects of the incident -- incident analysis, safety review, and data analysis and path forward.

Two of the teams evaluated the cause and safety impact of the incident. The third team focused on the technical data needs of the program, then recommended the optimal FEMP vitrification program path forward to meet these needs. Their conclusions provide valuable lessons learned that can be applied during future vitrification program activities and during other remediation activities. The reports are summarized in Sections 2, 3, 4, and 5 of this report; complete reports are provided as Appendices A, B, and C.

Three causal factors, identified during the root cause analysis were identified as the primary contributors for the discharge of glass through the bottom of the melter. The root cause analysis was bounded by the parameters of the as-built melter at the VITPP; alternative designs were not evaluated. Contributing factors to the failure and other potential areas for improvement are included in the lessons learned section of this report. The primary causal factor was the corrosion of the molybdenum disilicide bubbler tubes. Other contributing factors were the generation of stray electrical currents in the underside of the melter and glass migration and/or metals precipitation to the bottom of the melter. (See Appendix A, Vitrification Pilot Plant Incident Analysis Team Report.)

The Safety Review team determined that the discipline gained from all aspects of operational experience and personnel training for the VITPP Test Program ensured that the incident resulted in no personnel injuries nor environmental impact. The VITPP Test Program was executed in full compliance to existing safety basis documentation. However, evaluation of potential safety concerns resulting from the deterioration of melter components was not effectively factored into revisions of the safety basis documents, procedures, and test plans. (See Appendix B, Vitrification Pilot Plant Safety Review Team Report.)

The Data Analysis and Path Forward team identified, evaluated, and recommended facilities capable of providing services to meet data needs of the VITPP Test Program. This detailed technical analysis provides the basis for the Silos Project path forward. (See Appendix C, Vitrification Pilot Plant Data Analysis and Path Forward Team Report.)

As a result of the incident, the Data Analysis and Path Forward Team recommended a practical approach in pursuing further data needs (i.e., 1 MT/d capacity, mini-melter scale surrogate material) for design, construction, and operation of the full-scale vitrification facility -- that is, to focus on glass chemistry in order to allow use of commercially available, proven techniques and materials of construction. This approach can best be accomplished by eliminating the high-sulfate Silo 3 waste from the vitrification program, thus eliminating the need for a three-chamber design, reducing waste loading to target 60 percent, and ensuring an oxidizing environment with close redox monitoring, as well as a means of dealing with molten metals when it occurs.

After evaluating 21 options for obtaining these data, the team recommended that the VITPP melter be placed in a safe shutdown condition, and not be restarted. Alternatively, the needed data should be obtained through a combination of the following:

- Waste retrieval demonstrations
- Melter (or refractory) materials of construction testing using K-65 materials
- Laboratory testing to validate and optimize surrogate formulas that model K-65 residues
- Pilot plant scale-up at a 1 MT/d off-site facility with surrogate materials
- Mini-melter testing with K-65 residues (low temperature, approximately 1,150°C)

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- Design, construction, and operation of the first module (melter train) of a final remedial facility.

Design, construction, and operation of the final remedial facility (additional modules) would be based on successful testing, as indicated above.

Thus, each of the three investigative teams fulfilled their charters in providing data, solid technical analyses, lessons learned, and recommendations to steer the future of remediation of silo material at the FEMP.

A comparison of the data obtained thus far in the VITPP Test Program shows the program to be successful in several ways. The melter produced high-quality surrogate material glass that passed the Toxicity Characteristic Leaching Procedure (TCLP). A considerable amount of information was obtained about the process equipment used in the vitrification program. In addition, critical data about glass chemistry was obtained. The program demonstrated that vitrification is a technology that is feasible for treating silo waste at the FEMP. However, further data are required to reduce the current uncertainty associated with the practicality of operating a full-scale vitrification facility and ensuring cost/schedule predictability. While it is recognized that not all needed data were acquired prior to the incident, the VITPP Test Program was successful in yielding considerable data that can be used in the future design of a vitrification facility for treating FEMP silo waste, or for treating other wastes suitable for vitrification at other sites undergoing remediation. All observations, recommendations, and lessons learned will be incorporated into the sitewide lessons learned database and shared complex-wide.

This report, and the technical analyses on which it is based, provide a solid foundation for DOE when deciding the future direction of the vitrification program at the FEMP. These recommendations will be shared with regulatory agencies and stakeholders, as well as the Independent Review Team (IRT) that DOE-Fernald convened in November 1996, to evaluate the FEMP Silos Project. The IRT is expected to make recommendations after completion of its review later in 1997.

1.0 INTRODUCTION

1.1 Purpose and Organization of This Report

This report is prepared in response to an incident involving a leak of non-radioactive glass from the vitrification melter at the Fernald Environmental Management Project (FEMP) on December 26, 1996. This report summarizes the reports of the three teams convened to investigate the incident. It includes a path forward for the Vitrification Pilot Plant (VITPP) Test Program at the FEMP, a U.S. Department (DOE) facility located northwest of Cincinnati, Ohio.

Section 1.0 provides relevant site history; background information about the VITPP test program; a description of December 26, 1996, incident; and a description of the charter and technical approach of each investigative team. Sections 2.0 through 4.0 provide the results and conclusions of each team. Section 5.0 summarizes overall conclusions. Appendix A is the Vitrification Pilot Plant Incident Analysis Team Report, Appendix B is the Vitrification Pilot Plant Safety Review Team Report, and Appendix C is the Vitrification Pilot Plant Data Analysis and Path Forward Team Report.

1.2 Relevant Site History

The FEMP, a former uranium processing facility, is undergoing remediation under the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA), guided by a series of related agreements between the DOE and the U.S. Environmental Protection Agency (EPA) and the Ohio EPA (OEPA). Compliance with other environmental regulations, such as the Resource Conservation and Recovery Act (RCRA), is integrated into the CERCLA remediation. Fluor Daniel Fernald (FDF) is managing the remediation for the DOE.

According to the terms of the Amended Consent Agreement between DOE and EPA (September 1991), the site was divided into operable units. Operable Unit 4 includes the four waste storage silos, the decant tank system, and berms and soils within the operable unit boundary. EPA approved the Operable Unit 4 Record of Decision (ROD) in December 1994. The ROD specified that Operable Unit 4 remediation include the on-site vitrification of Silos 1, 2, and 3 material, as well as soil excavation, dismantling and demolition of the silos and vitrification facilities, and site restoration. The VITPP Test Program supports Operable Unit 4 remediation. The Silos Project is responsible for remediating Operable Unit 4.

1.3 VITPP Background

The VITPP was designed to develop the detailed requirements to apply the vitrification process to the large-scale remediation of Silos 1, 2, and 3 materials. The test program for the VITPP is being conducted in two phases. Phase I includes processing surrogate materials to verify that systems and components operated as designed, establishing viable glass formulas, identifying operating parameters, and documenting performance limitations. The purpose of Phase II is the re-test of the VITPP, using actual silo waste materials to provide final verification that the vitrification process is a viable method for processing the wastes.

The VITPP's primary component is an electrically powered (joule-heated) melter. The unique three-chamber melter design was constructed to obtain detailed performance data for glass chemistry development, materials of construction selection, and validation of the melter's physical design. Due to the complex chemical characteristics of the silos waste material, the melter design was primarily driven by the need for high-temperature operations (1,350°C) in order to destroy sulfates in concentrations up to 15 percent, combined with lead concentration up to 12 percent that require an oxidizing environment to avoid the formation of a metallic lead phase which can be detrimental to the materials of construction.

1.4 Incident Description

On December 26, 1996, at approximately 2222 hours, a Fluor Daniel Fernald (FDF) Maintenance Supervisor at the VITPP entered the melter room (on the ground floor) and observed a small stream of molten glass flowing from the bottom of the VITPP melter and into the bottom drain container. The maintenance supervisor alerted the system engineer, who directed the control room operator to ramp-down power to silicon control rectifier (SCR) #1, in order to decrease melter temperature. The maintenance supervisor notified fire and safety personnel via radio that smoke was present in the melter room, but no fire was observed. At 2225 hours, the communications center received the first smoke alarm from the VITPP. The communications center operator immediately notified the assistant emergency duty officer (AEDO). The system engineer directed all personnel in the melter room to stand by the exit to the room on the first floor, near the personal protective equipment room door. The shift manager attempted to "freeze" the glass stream (which was about the diameter of a pencil) by applying water to the stream from the water hose. The attempt was abandoned after it was determined to be ineffective in reducing the flow of glass from the melter. At 2234 hours, the shift supervisor directed the control room operator to initiate an emergency shutdown of the melter by depressing the emergency stop button and ordered the evacuation of all personnel from the building. Two electricians were ordered to open the motor control center

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disconnect for SCR 1 and SCR 2, which resulted in the complete electrical isolation of the melter.

As the glass continued to flow from the melter, the hole widened to approximately 2 inches in diameter. Some of the flow was diverted, and fell outside the bottom drain container and onto a concrete floor. The epoxy paint on a two-foot-by-three-foot area of the floor ignited and resulted in a small fire. Two portable fire extinguishers were unsuccessfully expended on the fire. Emergency response personnel arrived on the scene by 2240 hours and extinguished the fire by covering the floor with water.

Just before the incident, the temperature of the melt was 1,150°C. The melter was in an idle condition (no feeding). The molten glass in the melter consisted of a Series A surrogate waste formula. This surrogate was designed to simulate a mixture of the Silos 1 and 2 waste stream and standard glass-forming chemicals.

Approximately three tons of surrogate material (glass) leaked from the melter. No one was injured during the event. The incident occurred approximately halfway through Campaign 4 of the VITPP Phase I Test Program; nine batches of slurry feed had been fed to the melter. Phase I was scheduled to be completed in January 1997. The incident prematurely ended Campaign 4 of the VITPP Test Program. The event was classified as an Unusual Occurrence per DOE Order 232.1, "Occurrence Reporting and Processing of Operation Information." A Notification Report was issued on December 27, 1996 (Appendix A).

1.5 Evaluation Teams

Immediately following the incident, FDF initiated two tiers of response: (1) immediate emergency response measures; and (2) three teams were formed to study key aspects of the incident. Each team was assigned a separate charter; all activities commenced on January 7, 1997. The three teams are:

- The Incident Analysis Team, responsible for evaluating the root cause(s) for the incident and for developing lessons learned from the melter operation and subsequent disassembly.

- The Safety Review Team, responsible for evaluating the appropriateness of the response to the incident and the adequacy of engineering controls regarding safety during the event, and for identifying the safety basis and the safety and health impacts both on and off site.

- The Data Analysis and Path Forward Team, responsible for evaluating data, determining deficiencies for detailed design and operation of the melter, determining the need for low-temperature melts and alternatives to restart of the vitrification pilot plant, identifying post-incident data needs, and developing a path forward for Phase I of the VITPP Test Program.

Team members were chosen based on their areas of expertise and experience with vitrification programs. Team members are identified, with brief biographies provided, in each team's report.

1.5.1 Incident Analysis Team Approach

Four deliverables were identified: a Melter Inspection Plan, results of a Failure Modes Analysis for root cause determination, lessons learned, and a final report.

The first step in the evaluation of the incident involved the development and approval of a Melter Inspection Plan. Using the plan, the melter was partially disassembled and inspected by an inspection team. The inspection team consisted of pilot plant operations and maintenance personnel. Information from the melter vendor, melter refractory vendors, melter inspection team, and documented operational experience gained during melter operation was used to perform a Failure Mode Analysis for the root cause determination.

During the melter disassembly, the inspection team evaluated and examined the melter. The lid and refractory roof of the melter were removed completely. The inspection team documented the conditions found inside the melter. Videos, photographs, physical measurements, and well-documented sampling of the melter were performed and recorded. Core-drilled borings were taken from the failed bubbler and at other strategic locations in the floor of the melter.

Finally, lessons learned from the operation and partial melter disassembly were developed and documented.

1.5.2 Safety Review Team Approach

The Safety Review Team investigation included review of safety documentation, operating procedures, test plan, and emergency procedures.

The team formed three sub-teams to facilitate their investigation. The sub-teams are:

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- **Sub-Team 1- Safety Basis Documentation.** Sub-Team 1 reviewed the adequacy of the Safety Basis documentation. The documentation review included the Preliminary Hazards Safety Analysis Report, the Auditable Safety Record, and the Final Hazards Safety Analysis Report. The review also included the design change control procedures, configuration management, and the Project Execution Plan.

- **Sub-Team 2 - Off Normal Scenarios and Test Corrections.** Sub-Team 2 reviewed the procedures and test plan to determine what materials (i.e., refractory, electrode material, bubbler material, etc.) had been identified for monitoring. The Phase I Test Plan was reviewed to determine if the plan addressed off-normal scenarios such as erosion/corrosion of refractory, breached electrodes, and metal precipitates. Identification and resolution of the off-normal condition by the operations personnel were analyzed.

- **Sub-Team 3 - Response to Incident.** Sub-Team 3 described the melter incident in detail, including initial conditions, discovery of the melter breach, and the incident chronology. Evaluation of the appropriateness of the incident response by the shift team is also provided. Their evaluation documents related facts such as organizational structures, training and qualifications, and standard operating procedures.

1.5.3 Data Analysis and Path Forward Team Approach

The primary objectives of the Data Analysis and Path Forward Team were to evaluate all of the existing data to date (especially from Phase I of the VITPP) and to determine what additional data are needed for preparation of a high-quality melter performance specification and for detailed design of the rest of the facility. While focus was placed on addressing the data that would have been provided by completing the Phase I testing, further evaluation was given to the data needs for Phase II testing. Once data needs were identified, the team developed options to acquire the data. Both on-site (VITPP) and off-site sources were evaluated, and a recommendation for data acquisition was made. The third objective of the team was to recommend the near-term path forward of the VITPP.

The following deliverables were identified and are addressed in this report:

- A data matrix of outstanding data requirements and operations experience that would have been gained from the balance of Campaign 4 to support full-scale detailed design
- Options and recommendations for acquiring outstanding Phase I data, additional data and operating experience, as well as additional data required for full-scale design
- A recommendation for the path forward for the VITPP Phase I Test Program
- A comparison of cost, schedule, and risk analysis for path forward options.

Initially the team developed assumptions to be considered in the determination and evaluation of the data needs and the options to provide the needed data. These assumptions are as follows:

- Evaluation of data needs addresses Silos 1 and 2 materials only.
- Melter(s) design, fabrication, and installation will be procured under a performance specification.
- FDF will design/build/operate the full-scale vitrification facility.
- Toxicity Characteristics Leaching Procedure (TCLP) will be met with low-temperature operations; an acceptable glass can be produced.
- Laboratory testing always is required prior to any scale of melter testing.
- Radioactive testing or testing of actual silo residue will be performed in the form of further glass chemistry development that will support the preparation of design documents. Therefore, some Phase I data requirements are deleted, primarily in the area of off-gas composition.
- A waste retrieval demonstration will be performed. Slurry testing will be included. The demonstration will supplement Phase I slurry hydraulics data.

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- Minimal scale-up results in decreased risk.

- Evaluation of melter options was confined to joule-heated melters only, due to time constraints.

Then, a simplified, more relevant data matrix was developed to provide an understanding of the type of information that was needed. Data needs were grouped as they related to Phase I, Phase II, the waste retrieval demonstration and new (identified as a result of the melter incident). Data needs are identified in Table 2-1 of Appendix C.

Options for acquisition of needed data were then evaluated and categorized by the minimum scale of facility needed to develop sufficient data that would provide confidence for use in the design of the final remediation facility for Silos 1 and 2. In many cases, if the data were obtained in a larger-scale facility, the confidence level regarding potential "scale-up" would be higher. The options were categorized into four categories:

- Laboratory scale - crucible (five options)

- Mini-melter - ~10 kilogram (kg) - 100 kg/day (five options)

- Pilot scale melter - ~1 metric ton per day (MT/d) (nine options)

- Full-scale melter - ~6 MT/d (two options). These data acquisition options are identified in Table 2-1, Table 4-1, and Section 4.0 of Appendix C.

The team's primary consideration for option development was given to scale and the potential dynamics of the equipment used. The detailed alternatives were evaluated for their ability to meet the technical requirements. Where glass composition is the concern, it appears to be appropriate to perform crucible melts. Composition and rate information to support the off-gas design is considered to be much more representative when samples are taken from a melter where a feed can be sustained and steady state operations achieved. A major consideration with regard to the data required and the need for the data reflects the level of risk that is acceptable in the design of the final remediation facility. The level of scale-up required is a factor and it is assumed that with minimal scale-up, there will be minimal risk.

Next, specific facilities (located both on site and off site) that could provide the required data were identified. These specific data acquisition options are identified in Table 4-1 of Appendix C.

Cost, schedule, and risk analysis were compared for each facility. During evaluation of the facility options, emphasis was placed on schedule impact and the technical risk of each specific option. The scope of the data gaps was also considered. This evaluation appears in Section 4.0 of Appendix C.

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2.0 INCIDENT ANALYSIS SUMMARY

2.1 Results

Three causal factors were identified during the root cause analysis (Appendix A) as the primary contributors for the discharge of glass through the bottom of the melter. The root cause analysis was bounded by the parameters of the as-built melter at the VITPP; no alternative designs were evaluated. Contributing factors to the failure and other potential areas for improvement are included in Section 2.2. The primary causal factor was the corrosion of the molybdenum disilicide bubbler tubes and the underlying refractory materials. Other contributing factors were the generation of stray electrical currents in the underside of the melter and glass migration and/or metals precipitation to the bottom of the melter. This section explains these three causal factors.

The corrosion of the bubbler tubes and the underlying refractory materials result from the following mechanisms:

- Corrosion of the molybdenum disilicide bubbler tubes in the oxidized and/or lead-bearing surrogate glass melt. Two mechanisms for the corrosion of the bubbler tubes were identified:
  - Reaction of molybdenum disilicide with available oxygen-forming molybdenum trioxide and silicone dioxide
  - Reaction of molybdenum disilicide with lead oxide in the glass melt, forming molybdenum trioxide, silicone dioxide, and elemental lead.
- Reduction of lead oxide from the glass melt produced lead metal, which may have accelerated the corrosion of the underlying refractories. The mechanism for reducing the lead oxide involves the molybdenum disilicide tubes chemically reacting with lead oxide in the glass melt to produce molybdenum trioxide and elemental lead. Also, the molybdenum metal electrodes chemically reacted with the lead oxide contained in the center chamber glass that leaked into the electrode side chambers, producing molybdenum oxide and elemental lead.
- Another bubbler corrosion mechanism investigated was the possibility that molten lead could alloy with molybdenum disulfide forming a

molybdenum-lead-silicon alloy at glass melt temperatures. However, laboratory tests did not support this mechanism.

Glass migration and/or metals precipitation to the bottom of the melter resulted from bubbler tube corrosion, migration through the bottom drain assemblies, and migration through refractory joints. The migration initiated corrosion of the alumina-zirconia-silica (AZS) refractory and may have been followed by the precipitation of metallic lead.

Electrical current and ground paths generated inside the melter created localized current and hot spots with resulting increased corrosion. The current paths may have been the result of glass migration through the refractory material to the inner and outer shells. Metallic lead precipitation at the inner and the outer shell may have provided electrical paths. The lead may have been the result of the reaction mechanisms described above.

2.2 Conclusions

These phenomena are generally known in the glass industry. They are considered the primary conditions affecting the service life of the refractory lining and the length of melter life. There is a general consensus that the corrosion rate(s) of the materials of construction are unique to the corrosive properties of the waste glass melt and the corrosion resistance of the refractory lining. Based on the uncertainty of these rates, a breach of any melter cannot be predicted accurately with a high degree of certainty. The K-3 refractory layer within the melter indicated reasonable wear and corrosion resistance while circumstances occurred that accelerated the failure of the AZS material. A detailed root cause analysis is included in Section V of Appendix A. This section identifies the recommendations and observations, followed by lessons learned, made by the Incident Analysis Team.

2.2.1 Observations

The first observation is that the bubbler tube assemblies were not resistant to corrosion by the glass, or metal phases in the glass melt, nor was the molybdenum disilicide tube resistant to an oxidation reduction reaction with lead oxide. Because of this, Molybdenum disilicide should be treated as a consumable material. Designs must consider the need to continuously replenish it.

The second observation is that the bonded AZS refractory, although possessing excellent thermal shock properties, was not resistant to downward drilling by metal nor did it possess a high corrosion resistance to the glass

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melt. The failure of this barrier contributed to the glass migration to the bottom of the melter and also may have contributed to the formation of stray electrical current paths to below the main melt pool. As a result, refractory design should consider use of material with exceptional corrosion resistance to glass melts and to metal attack. A graded layering of chrome-bearing fused cast refractories such as Monofrax K-3 or fused cast AZS refractory (instead of bonded) should be considered in future refractory designs. It is important that the selected material be continuous from the glass pool to the outer shell around any melter penetrations.

The third observation is that penetrations through refractory containing the melt pool (i.e., bottom drains, bubbler assemblies and thermowells) resulted in extensive migration of the glass melt and metals to sub-layer refractory. As a result, penetrations through melt pool refractory should be minimized to the extent possible in future melter designs.

The fourth observation is that materials of construction testing for melter components and refractory lining was not conducted. As a result, careful consideration should be given to materials of construction testing on melter components and refractory with both glass surrogate materials and actual silo waste. Chemical reactions, including redox, should be considered.

The fifth observation concluded that glass migration and metals precipitation to the inner shell and to areas between the inner and outer shells resulted in the formation of a disruptive current path. The formation of the electrical current path was facilitated by the presence of inner and outer shells, because the two shells were constructed of electrically conductive material. The disruptive electrical current path may have contributed to the heating of the sub-surface glass pool and prevented the glass from solidifying below the surface refractory. As a result, future melter design(s) should consider the elimination of electrically conductive shells enclosing the refractory. The materials of construction should provide a temperature profile from melt pool to the outer melter surface that freezes migrating glass well in advance of the glass reaching the outer container.

The final observation is that although the project was performing reduction/oxidation (redox) monitoring in accordance with project procedures, process experts stated that better redox monitoring could be accomplished via analytical analyses. Improved redox monitoring and controls may minimize metals precipitation at the bottom of the melter. As a result, project management should re-evaluate the monitoring system in place to control

reduction/oxidation (redox) chemical reactions to determine if the monitoring system meets the needs of the project.

2.2.2 Lessons Learned

The following lessons learned are the result of the Event and Causal Factor Chart developed for the operational analysis. These items were determined by other events and conditions which were not directly related to the failure of the melter, but were deemed as contributing causes and/or potential areas for improvement.

The first lesson learned is that project personnel identified a concern about bubbler tube erosion, and the concern was addressed in the initial Phase I Test Plan and Hazards and Operability Report. The concern was omitted from the Final Hazard Analysis Report without mitigating measures identified or implemented. As a result, the project organizational structure should include an independent external technical review by industry experts. In addition, concerns that arise must be captured and maintained until formal resolution is reached through an approval process.

The second lesson learned is that the project personnel's level of knowledge in melter design, components and operations was less than adequate. Project personnel were not trained to recognize the limitations of the unique melter design and the components used for construction of the melter. As a result, managers, engineers, operators, and maintenance personnel should be trained by an expert in melter design, operation, and unique phenomena associated with the melter to develop a basic understanding. Industry experts should be used as consultants in the beginning of the project. Research of industry publications of material suppliers must be conducted to verify the quality of contracted designs.

The third lesson learned is that the invitation for bid did not satisfactorily address existing procedural requirements. The design change control process did not start until construction began. As a result, contracts must identify the site and functional area requirements in contract specifications prior to Invitation for Bid (IFB) and Request for Proposal (RFP) to assure that quality products are delivered (i.e., the design change control process must be formalized and documented).

The fourth lesson learned is that the engineering, procurement and construction (EPC) process was compromised by the project's desire to recover from incurred schedule slippages. Concurrent design led to design

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changes, adaptations, additional schedule slippages, and cost over-runs. As a result, design integration is essential and should not be compromised to maintain schedules. Increased design controls are required when more than one design organization is responsible for deliverables. Design change control should be applied during the Title I Design to verify and justify deviations from the design functional requirements.

The fifth lesson learned is that numerous design deficiencies were identified during melter operations and during the post-incident melter inspection. As a result, the following recommendations would enhance melter design performance:

- Materials of construction must be evaluated for form, fit, function, reactions to each other, and life expectancy (e.g., the interaction of molybdenum disilicide and lead, riser block, AZS, etc.)
- Consider alternate melter design (i.e., gas, low-temperature, electrical)
- Minimize or eliminate refractory penetrations
- Develop reliable temperature monitoring of the glass pool
- Install ground current monitoring of electric melters
- Improve reliability of the melter discharge chamber
- Improve reliability of the melt pool level indication
- Redundancy of support and backup systems need to be evaluated (e.g., lack of redundancy for SCR 1)
- Performance specifications should be developed with the aid of industry experts during the conceptual design phase, subsequent design reviews, and technology selection.

The sixth lesson learned is that, due to a lack of timely analytical results, operational productivity was impacted, reaction to changing conditions was delayed, and assessment of the impact of operational adjustments could not be ascertained. The project had to rely on three remote laboratory facilities. As a result, adequate resources should be provided to improve the turn

around time of chemical analytical results. Project personnel should inform vital support organizations in advance that increased services may be required, depending on variables experienced during operation. This advanced notice will enable the support organization to plan for increased demands for services by the project.

The final lesson learned is that critical components were not evaluated collectively for operational impact. Breakdown of level indicators and thermal wells were detected during operations, but other components made of the same material, such as air lifts and bubblers, were not evaluated for potential failure. As a result, a material failure and trending process should be developed to identify deficiencies that potentially can affect similar processes or materials prior to a failure; a failure mode analysis should be developed for all systems and components prior to operations and develop casualty procedures to minimize the effects of an abnormal event or condition; data quality objectives should be thoroughly developed that include the materials of construction, additives, products, by-products, lubricants and solutions to assure that all process materials and components are baselined before start-up and monitored during operations.

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3.0 SAFETY REVIEW SUMMARY

3.1 Results

The Safety Review Team found the following results to address the investigation areas in the team's charter:

- **Safety Basis.** FDF executed this project in accordance with applicable DOE safety-related requirements and within the established safety basis for the VITPP Test Program. These requirements, and related guidance, include DOE 5480.23, Nuclear Safety Analysis Reports; Draft DOE-STD-3005-YR, Evaluation Guidelines for Accident Analysis and Safety Structures, Systems, and Components; DOE-STD-1027-92, Hazard Categorization and Accident Analysis Techniques for Compliance with DOE Order 5480.23, Nuclear Safety Analysis Reports; DOE Order 5480.7A, Fire Protection; DOE-STD-5502-94; the Safety Performance Requirements manual; RM-0021; SPR2-1; and 29 CFR 1910.120, Hazardous Waste Operations and Emergency Response.

- **Implementing Controls.** The team evaluated whether the Phase I Test Plan addressed issues relevant to the incident. The test plan addressed redox, contamination of the side chambers, erosion/corrosion of electrodes, and metal precipitates, but did not address bubbler tube erosion/corrosion and a melter leak.

  The VITPP Test Program had implementing controls in place for off-normal, or emergency, conditions. An example of project personnel responding to an unrelated, unusual operational situation was given. In addition, several safety documents recognized the potential for a leak in the melter. The probability of occurrence was determined to be "unlikely" with "moderate" consequence; however, the basis for these determinations is unclear.

- **Response to Incident and On-Site Safety and Health Impacts.** The actions taken to combat the casualty were appropriate and timely. The discipline gained from all aspects of operational experience and personnel training ensured that the incident resulted in no personnel injuries and minimal material damage.

- **Safety Impacts Off Site.** Safety impacts off site were evaluated in terms of the potential environmental impact of the incident. During

Campaign 4, lead and barium compounds were included in the surrogate. These metals, in addition to chromium leaching from the melter refractory, were the only RCRA hazardous substances present in significant amounts in the melter at the time of the event. No radionuclides were present in the surrogate materials during Campaign 4. Mass balance analyses accounted for most of the lead and barium in the glass, itself; most of the remainder can be accounted for as particulate released to the melter off-gas system. All the glass that drained from the melter was containerized, and was characterized to determine proper management, including disposal, in accordance with RCRA requirements. Releases via air or wastewater pathways were minimized because of the nature of the released material (glass which quickly solidified), and binding of the metals into the relatively insoluble matrix of the glass. Detailed pathway analyses are provided in Appendix B. The incident resulted in no violations of the Operable Unit 4 applicable or relevant and appropriate requirements (ARARs).

3.2 Conclusion

The Safety Review Team identified the lessons learned and recommendations, as a result of the investigation and resulting report.

- **Safety Assessments and Hazard Analyses.** The safety assessments and hazard analysis processes for the VITPP inadequately assessed the potential for casualties resulting from incompletely understood wastage in the melter. (Wastage is the combined effects of corrosion, erosion, and electrochemical reactions which results in degradation of material.) The team recommended that:

    - Project management must assure that periodic independent technical review and direction is provided during all phases of the project. This must include safety, design, and operational considerations, and it must be available during early phases of the project.

    - Because of the technical complexity of this project, project management must assure that a thorough, detailed hazard assessment is performed by an integrated team with adequate technical resources to establish the safety basis, and that this safety basis is revised as new data and facts become available.

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- **Design Change Control.** The design change control process existing at the time of the bottom drain container design was ineffective in assuring that changes are adequate to maintain operations within the safety basis. The team recommended that project management must ensure that management control processes are effectively implemented throughout the life of the project to maintain integrity of the safety basis. Not only must this include a consideration of the FDF design change control process, but also the design and safety interface between FDF and subcontractor scope of work.

- **Emergency Response.** Good emergency response to casualty conditions requires good design, disciplined operations, and continuous training. The team recommend that:

  - The discipline gained from all aspects of operational experience and personnel training ensured that the incident resulted in no personnel injuries. Project management must ensure that such training is a continuing requirement for a project of this complexity.

  - Project management must ensure that there are appropriate designs and/or effective administrative controls and/or project procedures to mitigate and confine the consequences of a potential breach of the VITPP melter which could spill molten glass onto the floor of the melter room, as well as, the consequences resulting from emergency response. For example, the melter room design did not include: (a) curbs or dikes to contain large quantities of water, and prevent it from leaving the pad; or (b) curbs or dikes to mitigate flow of molten glass into the sump.

4.0 DATA ANALYSIS AND PATH FORWARD SUMMARY

4.1 Results

From the data needs evaluation and the evaluation of the options for obtaining the required data, eight specific recommendations for the path forward for the vitrification program at the FEMP were developed. These recommendations are based on the analysis and conclusions of the team as defined in Section 5.0 of Appendix C.

The basis of the recommendations is related to the melter design and reasons for that design. The VITPP's unique three-chamber design was envisioned to address the following two specific design considerations as it applies to waste glass chemistry:

- The high-sulfate concentration in the Silo 3 residues requires high temperatures to destroy sulfates, thus improving leachability performance in the final waste.

- High lead, which requires an oxidizing environment to avoid lead reducing out and forming a metallic phase, would eventually cause electrical shorting of the melter.

In theory, the three-chamber design was well suited for the glass chemistry that resulted from combining Silos 1, 2, and 3 material. However, during practical operation, the result was somewhat different. When the molybdenum electrodes were subjected to the highly corrosive glass that migrated into the electrode chambers from the center waste glass chamber, the molybdenum experienced accelerated corrosion and caused lead to form a metallic phase in the reducing environment required to protect the molybdenum. The unique design for the glass chemistry provided equally unique conditions that potentially can be catastrophic in their impact on the melter materials of construction used.

A practical approach is to focus on glass chemistry in order to allow usage of commercially available, proven techniques, and materials of construction. This approach can be accomplished best by eliminating the high-sulfate Silo 3 waste from the vitrification program and a waste loading target of 60 percent, and ensuring an oxidizing environment with close redox monitoring, as well as a means of dealing with molten metals when they are produced.

4.2 Conclusions

The Data Analysis and Path Forward Team has developed a path forward for the vitrification program at the FEMP that is based on the recommendation that the VITPP melter does not restart (Recommendation 1). Once this premise is accepted, the next five recommendations (Recommendation 2 through 6) can be performed concurrently, but may have varying durations. These recommendations (Recommendation 2 through 6) collectively meet the data needs required for design, construction, and operations of Module 1 of the Remedial Facility (Recommendation 7) and subsequent module(s) (Recommendation 8).

Recommendation 1 - Vitrification Pilot Plant Safe Shutdown. Based on the fact that the three-chamber design, with molybdenum electrodes and multiple bottom penetrations, is unsuitable for vitrifying Silos 1 and 2 waste, it is recommended that the VITPP melter not be restarted, but be placed in a safe shutdown condition. This recommendation is further supported by the cost and schedule required for design, construction, and operational readiness involved in a VITPP restart.

In the event the VITPP melter is not restarted, the balance of data not yet supplied by VITPP operations (needed to support design of the remedial facility), will need to be gathered at alternative facilities. The primary vitrification program data needs are addressed by the following recommendations for acquisition of this data:

Recommendation 2 - Perform Waste Retrieval Demonstration. Waste retrieval and feed system data should be supplied in the VITPP as part of the planned comprehensive waste retrieval demonstration following detailed rheology testing in an off-site laboratory. The waste retrieval demonstration is included in the Silos Project approved baseline.

Recommendation 3 - Perform Materials of Construction Testing with K-65 Residues. As a result of the melter incident, further materials of construction testing is needed for electrodes, refractory materials, and ancillary equipment. This testing typically would be performed at a qualified laboratory, using actual residues, in accordance with standard ASTM tests.

Recommendation 4 - Perform Laboratory Testing of K-65 Residues to Validate Surrogates. The K-65 residues should be analyzed to gain a better understanding of the chemical composition of the waste. The analysis would be used to validate or modify the recipe for silo surrogate materials.

Additional physical data consisting of the rheology and hydraulic data of the slurry will be developed in the laboratory, with simulated conditions confirmed in the waste

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retrieval demonstration. It is important to prove that the behavior of the actual material closely resembles the surrogate material, therefore enabling various tests to be performed, using differing variables to optimize the slurry-handling aspects of the facility.

Recommendation 5 - Perform Pilot Plant Scale-up with Surrogate at a 1 *mt/day* Off-Site Facility. The primary Phase I Campaign 4 data gap left by the VITPP incident is the lack of empirical scale-up data (throughput versus melt pool surface area) as it applies to a low-temperature formulation. After evaluating data needs, the team determined that data is needed at a 1 MT/d or greater (surrogate glass) scale. Various facilities are available, including the 1 MT/d, Inconel electrode, melter at The Catholic University Vitreous State Laboratory (VSL) or possibly the 2 MT/d, high-temperature melter at Pacific Northwest National Laboratory (PNNL). Both VSL and PNNL could perform the low-temperature testing with a surrogate that has been validated against analysis of actual silo material. Both options could provide needed Phase I data at a lower cost and in a shorter time frame.

Recommendation 6 - Perform Mini-Melter Testing with K-65 Residues. The primary data that were to be supplied by Phase II (radioactive VITPP tests) were the radioactive glass properties at a 1 MT/d scale, off-gas composition, and health physics data. After careful evaluation of data needs for full-scale remedial design, the team recommends that radioactive testing with actual silo residues can be performed in mini-melter tests and correlated against pilot-scale runs with surrogates that have been validated against K-65 elemental and compound analysis. Radioactive mini-melter testing would demonstrate low-temperature vitrification parameters, as well as provide off-gas composition and dose measurements that could be utilized to validate dose modeling. In summary, all Phase II data needs could be met at the mini-melter scale, excluding radioactive operation at the larger scale (1 MT/d), and prototypical off-gas and feed system testing.

Recommendation 7 - Module 1, Remedial Facility Design, Construct and Operate. To reduce the risk of proceeding to full-scale design with limited pilot-scale testing, a sequential approach should be adopted. It is recommended that the first of several melter modules be used as a verification step, prior to hot operations, as confirmation of the melter design. The risk of scale-up is reduced in the fact that the plan will include extended comprehensive testing at full scale with surrogate material prior to operations with radioactive materials or procurement of the remaining full-scale modules. Operation of the first module will provide significant experience and serve as "proof of process testing," with the opportunity to incorporate "lessons learned" into the production modules.

Recommendation 8 - Additional Remedial Modules Design, Construct, and Operate. Following successful operation of the first module, larger-scale prototypical modules could be designed, constructed, and operated in parallel to the initial module, to fully stabilize the silo contents.

Each recommendation is discussed in detail in Section 5.0 of Appendix C.

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5.0 CONCLUSION

Each of the three investigative teams fulfilled their charters in providing data, solid technical analyses, lessons learned, and recommendations to guide the future of remediation of silo materials at the FEMP. This report documents each of the three teams' conclusions regarding the December 26, 1996, melter incident at the FEMP:

- The Incident Analysis Team identified the root causes(s) for the molten glass drainage. (See Section 3.0 and Appendix A.) Three causal factors were identified during the root cause analysis as the primary contributors for the discharge of glass through the bottom of the melter. The root cause analysis was bounded by the parameters of the as-built melter at the VITPP; no alternative designs were evaluated. Contributing factors to the failure and other potential areas for improvement are included in the lessons learned section of this report. The primary causal factor was the corrosion of the molybdenum disilicide bubbler tubes. Other contributing factors were the generation of stray electrical currents in the underside of the melter and glass migration and/or metals precipitation to the bottom of the melter (Appendix A).

- The Safety Review Team evaluated the response to the incident, where safety and health impacts of the incident on and off site, developed lessons learned, and provided recommendations to improve the safety analysis for VITPP operations. (See Section 4.0 and Appendix B.) The team concluded that the discipline gained from all aspects of operational experience and personnel training for the VITPP ensured that the incident resulted in no personnel injuries nor environmental damage. The VITPP Test Program was executed in accordance with DOE requirements, and within the safety basis established for this project. However, thorough scientific and engineering discipline was not exercised at the beginning of, or during, the project. Continuous attention to potential safety concerns resulting from the deterioration of melter components was not effectively factored into revisions of the safety basis documents, procedures, and test plans.

- The Data Analysis and Path Forward Team evaluated the data obtained in Phase I of the VITPP Test Program and identified what additional data are needed to prepare a high-quality melter performance specification and for detailed design and operation of the balance of the plant. This detailed technical analysis provides the basis for the program's path forward. (See Section 5.0 and Appendix C.)

As a result of the incident, the Data Analysis and Path Forward Team recommended a more practical approach in pursuing needed data for design, construction, and operation of a vitrification facility -- that is, to focus on glass chemistry in order to allow use of commercially available, proven techniques and materials of construction. This approach can be accomplished best by eliminating the high-sulfate Silo 3 waste from the vitrification program, reducing waste loading to target 60 percent, and ensuring an oxidizing environment with close redox monitoring, as well as a means of dealing with metallic lead when it occurs.

After evaluating 21 options for obtaining these data, the team recommended that the VITPP melter be placed in a safe shutdown condition, and not be restarted. Alternatively, the needed data should be obtained through a combination of:

- Waste retrieval demonstrations
- Melter (or refractory) materials of construction testing using K-65 materials
- Laboratory testing to validate and optimize surrogate formulas that model K-65 residues
- Pilot plant scale-up at a 1 MT/d off-site facility with surrogate materials
- Mini-melter testing with K-65 residues (low temperature, approximately 1,150°C)
- Design, construction, and operation of the first module (melter train) of a final remedial facility

Design, construction, and operation of the final remedial facility (additional modules) would be based on successful testing, as indicated above.

A comparison of the data obtained thus far in the VITPP Test Program shows the program to be successful in several ways. The melter produced high-quality surrogate material glass that passed the TCLP. A considerable amount of information was obtained about the process equipment used in the vitrification program. In addition, critical data about glass chemistry was obtained. The program demonstrated that vitrification is a technology that is feasible for treating silo waste at the FEMP. However, further data are required to reduce the current uncertainty associated with the practicality of operating a full-scale vitrification

facility and ensuring cost/schedule predictability. While it is recognized that not all needed data were acquired prior to the incident, the VITPP Test Program was successful in yielding considerable data that can be used in the future design of a vitrification facility for treating FEMP silo waste, or for treating other wastes suitable for vitrification at other sites undergoing remediation. All observations, recommendations, and lessons learned will be incorporated into the sitewide lessons learned database and shared complex-wide.

This report, and the technical analyses on which it is based, provide a solid foundation for DOE when deciding the future direction of the vitrification program at the FEMP. These recommendations will be shared with regulatory agencies and stakeholders, as well as the Independent Review Team (IRT) that DOE-Fernald convened in November 1996, to evaluate the FEMP Silos Project. The IRT is expected to make recommendations after completion of its review later in 1997.

ATTACHMENT A

VITRIFICATION PILOT PLANT

INCIDENT ANALYSIS TEAM REPORT

Report No. 40100-RP-0001
Revision 0
Issue Date: February 1997

Vitrification Pilot Plant Melter Incident: Incident Analysis Review Team

Prepared for
Vitrification Pilot Plant Incident Investigation

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REFERENCES

1. Glass Industry, "Can the corrosion of tank bottoms be prevented?," P.P. Boggum, June, 1974.

2. Ceramic Engineering and Science Proceedings, Vol. 9, No. 3-4, pp. 253 - 263, "First Campaign of a Lead Crystal Glass Electric Furnace in Poland," G.A. Warren, T. Sasiak, R.E. Davis, March/April 1988.

3. Glastech, "Corrosion of Refractory Bricks Resulting from Metal Drop Boring," Ber. 57 No. 4, pp. 81 - 95, J. Lexow and R. Bruckner, 1984.

4. Journal of the Society of Glass Technology, "VIII. - Some Distinctive Contours Worn on Alumina-Silica Refractory Faces by Different Molten Glasses: Surface Tension and the Mechanism of Refractory Attack," pp. 90 - 100, H.J. Tress.

5. Glass Industry, "Building Longer Life into Electric Melters," A. J. Merchant, September 10, 1987

6. Standard Oil Engineered Materials, "Carborundum Products - Monofrax Bottom Paving System - Product Information," September 1987.

7. Ceramic Industry, "Selecting Refractories for Glass Tank Superstructures," J. Thompson, February, 1988.

8. Monofrax Application Brief, "Exudation and Corrosion Behavior of Fusion Cast AZS Refractories," S.M. Winder, K.R. Selkregg, A. Gupta, D. Walrod, September, 1996.

9. Monofrax Fused Cast Refractories, "Exudation and Corrosion Behavior of Fusion Cast AZS Refractories," Carborundum, October, 1994.

10. "Monofrax Refractory Systems for Glass Furnaces," Carborundum Products, September, 1989.

11. Monofrax Application Brief, "Refractory Performance in Oxy-Fuel Glass Tanks," A. Gupta, S.M. Winder, September, 1996.

12. D. Nixon to E. Tooney letter, C:WMTSP(SP):96-0010, "Duratek 1000HT, Acceptance Issues," dated September 12, 1996.

13. Electric Melting in the Glass Industry, Chapter 28, "Electric Melting of Glass," Section 8, p.118.

14. Glass Furnaces, "Electric Tank Furnaces," English Translation by the Society of Glass Technology, Chapter 8, Section 8.5.1 p. 207.

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NOTICE TO READER

The Incident Analysis Review Team utilized the data available to determine the root cause for the failure of the Vitrification Pilot Plant Melter. All additional analyses, not yet received but requested, in the Vitrification Pilot Plant Melter Inspection Plan should be taken and utilized in the development of a path forward for the Silos Project.

As additional information and data are received, addenda to this report may be issued if the data are determined to have an impact on the outcome of this report.

EXECUTIVE SUMMARY

This report summarizes the methodology and conclusions of the Incident Analysis Team, formed to identify the root cause(s) of a leak in the vitrification melter at the U.S. Department of Energy (DOE) Fernald Environmental Managment Project (FEMP) on December 26, 1996.

The Vitrification Pilot Plant (VITPP), a unique three-chamber melter design, was constructed to obtain detailed performance data for glass chemistry development, materials of construction selection, and validation of the melter's physical design. The melter is being tested for use in vitrifying uranium processing waste contained in silos in the FEMP's Waste Storage Area. Vitrification is the selected remedy for treating these wastes, as stated in the FEMP Operable Unit 4 Record of Decision. Due to the complex chemical characteristics of the silos waste material, the melter design was primarily driven by the need for high-temperature operations (1,350° C) in order to destroy sulfates in concentrations up to 15%, combined with lead concentration up to 12% that require an oxidizing environment to avoid the formation of a metallic lead phase which can be detrimental to the materials of construction. The knowledge and experience gained through the operation of the pilot plant has provided invaluable insight and lessons learned required for the design, construction, and operations of the full-scale facility.

EVENT SUMMARY

On December 26, 1996, at 2222 hours, a maintenance supervisor at the VITPP entered the melter room (on the ground floor) and observed a small stream of molten glass flowing from the bottom of the VITPP melter and into the bottom drain container. The maintenance supervisor alerted the system engineer, who directed the control room operator to ramp-down power to silicon control rectifier (SCR) #1, in order to decrease melter temperature. The maintenance supervisor notified fire and safety personnel via radio that smoke was present in the melter room, but no fire was observed. At 2225 hours, the communications center received the first smoke alarm from the VITPP. The communications center operator immediately notified the assistant emergency duty officer (AEDO). The system engineer directed all personnel in the melter room to stand by the exit to the room on the first floor, near the personal protective equipment (PPE) room door. The shift manager attempted to "freeze" the glass stream (which was about the diameter of a pencil) by applying water to the stream from the bottom drain water hose. The attempt was abandoned after it was determined to be ineffective in reducing the flow of glass from the melter. At 2234 hours, the shift supervisor directed the control room operator to initiate an emergency shutdown of the melter by depressing the emergency stop button and ordered the evacuation of all personnel from the building. Two electricians were ordered to open the motor control center disconnect for SCR 1 and SCR 2, which resulted in the complete electrical isolation of the melter.

As the glass continued to flow from the melter, the hole widened to approximately 2 inches in diameter. Some of the flow was diverted, and fell outside the bottom drain container and onto a concrete floor. The epoxy paint on a two-foot-by-three-foot area of the floor ignited

and resulted in a small fire. Two portable fire extinguishers were unsuccessfully expended on the fire. Emergency response personnel arrived on the scene by 2240 hours and extinguished the fire by coating the floor with water. Approximately three tons of surrogate material (glass) leaked from the melter. No one was injured during the event. The event was classified as an Unusual Occurrence per DOE Order 232.1, "Occurrence Reporting and Processing of Operation Information." A Notification Report was issued on December 27, 1996. (Appendix A)

ROOT CAUSE SUMMARY

The draining of the glass melt through the bottom of the VITPP Duratek 1000-HT three-chambered, high-temperature melter was the result of corrosion of the number 3 bubbler tube assembly in association with severe exudation and corrosion of the bonded (Zirmul) alumina-zirconia-silica (AZS) sub-layered refractory beneath the Monofrax K-3 hot-face surface block. The corrosion phenomena were exacerbated with the introduction of lead oxide into the glass melt during Silos 1, 2, and 3 surrogate waste test campaigns (Phase I, Campaign 2 and Campaign 4), thus significantly reducing the life expectancy of the melter. The attack on the materials of construction (both the bubbler tube assembly and the underlying refractory materials) were through the following mechanisms:

- Corrosion of the molybdenum disilicide bubbler tube in an oxidized and/or lead-bearing surrogate glass melt. Corrosion resulted in the bubbler tubes receding into the less corrosion resistant underlying refractories. The chemical measurement for the erosion are:

  $2MoSi_2 + 7O_2 \rightarrow 2MoO_3\uparrow + 4SiO_2$ (Equation 1)
  $MoSi_2 + 7PbO \rightarrow MoO_3\uparrow + 2SiO_2 + 7Pb\downarrow$ (Equation 2)

- Downward migration of the glass melt through the bottom penetrations (bubbler tube and bottom drain assemblies) and refractory joints, initiated erosion/corrosion of the AZS refractory.

- Reduction of lead oxide from the glass melt produced lead metal. The molten lead may have accelerated the erosion/corrosion of the underlying refractories by a downward drilling action. Probable chemical reactions for the production of the lead metal are:

  - The molybdenum disilicide tubes chemically reacting with lead in the glass:

    $MoSi_2 + 7PbO \rightarrow MoO_3\uparrow + 2SiO_2 + 7Pb\downarrow$ (Equation 2 above)
    $1g + 10.27g \rightarrow 0.95g + 0.79g + 9.53g$

    According to equation 2, 1 gram of molybdenum disilicide produces 9.53 grams of molten lead metal. The silicone dioxide produced is absorbed into and becomes part of the glass. The molybdenum

trioxide is a gas at molten glass temperatures. Therefore, some molybdenum trioxide may leave the glass melt before being absorbed by the molten glass. Overnight crucible melts performed at Fernald show that when a molybdenum disilicide rod is placed in lead-bearing glass, a molten lead puddle forms in the bottom of the crucible.

- The molybdenum metal electrodes chemically reacting with lead-bearing center chamber glass that has leaked into the electrode side chambers:

$$\begin{array}{l} Mo + 3PbO \rightarrow MoO_3\uparrow + 3Pb\downarrow \\ 1g + 6.98g \rightarrow 1.50g + 6.48g \end{array} \qquad \text{(Equation 3)}$$

According to Equation 3, 1 gram of molybdenum metal will produce 6.48 grams of molten lead metal. Molybdenum trioxide is a gas at molten glass temperatures. Therefore, some molybdenum trioxide may leave the glass melt before being absorbed by the molten glass. Overnight crucible melts performed at Fernald show that molybdenum metal is placed in lead-bearing glass, a molten lead puddle forms in the bottom of a crucible.

- "Reboil" of surrogate glass passing through the E-brick walls can precipitate free lead. At 1,250°C the conductivity of E-brick is about 0.075 siemens per centimeter (S/cm). Some surrogate glasses, due to the inleakage of side chamber frit, approached conductivities in the range of 0.25 to 0.30 S/cm. The higher conductivity of the glass channels more electrical current, with resulting heating, in the cracks in the E-brick walls. Some operators commented that they saw the cracks near the glass melt surface glowing. The extra heat causes a reductive environment which was evident by the "boiling" seen at the E-brick walls while current was applied. The "boiling" quickly disappeared when the current was turned off or greatly reduced. Inspectors noted that they saw what looked like solidified lead-metal beads that formed from sweating from the pores of the E-brick wall cracks.

Reduction studies show that simple reduction of the surrogate glass (e.g., through the use of urea) will produce lead sulfide because of the presence of dissolved sulfur (sulfate) in the glass. It takes a high (localized) reduction potential (such as molybdenum metal, molybdenum disilicide or "reboil") to produce molten metal. No lead sulfide precipitation was identified during the melter inspection, only lead metal was observed. Glass samples from the lead-bearing campaigns where analyzed and showed $Fe^{+2}/(Fe^{+2}+Fe^{+3})$ ratios ≤ 0.33 in the side chambers and < 0.20 in the center chamber. Precipitated lead metal or lead sulfide is not known to happen under these redox conditions.

- Turbulent air abrasion, oxidation, and molten glass circulation via compressed air flowing from corroded bubbler tubes receding below the corrosion resistant K-3 refractory floor.

- Disruptive electrical current paths as a result of glass migration and metal precipitation at the inner and outer shell. Localized current creates hot spots with resulting increased corrosion.

- Upward drilling on the refractories by the glass melt through vapor phase formation.

- Lack of corrosion resistance of KAO-TAB95 castable insulating refractory to molten glass.

Another mechanism was suggested by the investigating group that could have led to the erosion of the bubbler tubes. This was the possibility that molten lead could alloy with molybdenum disilicide forming a molybdenum lead silicon alloy at glass melting temperatures. However, this does not appear plausible based on a recent crucible melt that was performed in the Fernald lab. Molten lead was left in contact with a molybdenum disilicide rod. Glass was poured over the lead and rod to protect them from the air and the crucible left overnight in a furnace at glass forming temperatures. The rod was pulled out of the molten lead at the end of the test. The molten lead did not even "wet" the molybdenum disilicide rod which indicates there appears to be no tendency to form the speculated alloy.

These phenomena are generally known in the glass industry. They are considered the primary conditions affecting the service life of the refractory lining and the length of melter life (References 1 - 11, 13 and 14). There is general consensus that the corrosion rate(s) on materials of construction are unique to the corrosive properties of the waste glass melt, the corrosion resistance of the refractory lining, and the reduction rate of molten metals. Based on the uncertainty of these rates, the breach of a melter cannot be predicted with any degree of certainty.

A detailed root cause analysis is included in Section V of this report.

I. TEAM SCOPE

On December 26, 1996, the melter in the VITPP failed, releasing approximately three tons of surrogate material into a bottom drain container and onto the floor surrounding the melter. Three teams were formed by FDF using cognizant industry professionals, operational personnel, and DOE oversight (Appendix B). The teams formed were:

1) Safety Review Team
2) Data Analysis Team
3) Incident Analysis Review Team

Each team was assigned a separate charter and all activities commenced on January 7, 1997.

The charter of the Incident Analysis Review Team was divided into two separate tasks: 1) evaluate the root cause of the melter failure; and 2) develop lessons learned from the melter operations and disassembly. In order to achieve these objectives, four deliverables were identified:

1) Prepare a Melter Inspection Plan (Appendix C)
2) Perform Failure Modes Analysis for root cause determination
3) Develop lessons learned
4) Prepare final report.

The Incident Analysis Review Team evaluated and examined equipment and results of core borings, conducted interviews, and reviewed documents pertinent to determine the root cause for the failure in the area of Bubbler #3 located in the bottom of the melter. To fulfill the assignment, the team set the parameters and concentrated on the incident and the factors that contributed to the ultimate failure of the melter.

Although the conclusions of the review may have some impact on future design, materials, or operational guidelines of melters, it was not within the scope of the team's analysis to proceed beyond the root cause of the event. The melter is a one-of-a-kind design developed as a pilot project. The team did not concentrate its efforts on verifying the validity of previous reviews, design specifications, or materials employed in determining the melter's operating parameters, life expectancy, or intended purpose.

Additional information may be gained by the inspection and further analysis of the melter, and this information may enhance the future path forward of the project. However, the path forward was beyond the scope of the Incident Analysis Review Team.

II. BACKGROUND INFORMATION

The VITPP was designed to develop the detailed requirements to apply the vitrification process to the large-scale remediation of Silos 1, 2, and 3 materials. The test program for the VITPP is being conducted in two phases. Phase I includes processing surrogate materials to verify that systems and components operated as designed, establishing viable glass formulas, identifying operating parameters, and documenting performance limitations. The purpose of Phase II is to re-test the VITPP using actual silo waste materials to provide final verification that the vitrification process is a viable method for processing the wastes. The VITPP's primary component is an electrically powered (joule-heated) melter. The remainder of this section provides the melter operational and design requirements, as specified in technical specification No. RHN02-40-062 - Vitrification Furnace, issued by FDF for this project:

Design Specifications

Minimum Operating Life	1 year
Design Life	3 years
Operating Temperature	1,100 - 1,350 degrees C
Minimal Production Rate	1 tonne/day of vitrified surrogate material discharging from the furnace without agitation
Furnace Design Temperature	1,400 degrees C
Feed Solids Content	50% by wt. approx.
Feed Moisture Content	50% by wt. approx.
Feed Rate	0.1 - 1.0 gpm of slurry (intermittent flow)
Feed Temperature	2 - 26 degrees C
Available Feed Pressure	up to 5 psig
Feed Slurry Density	90 - 100 lb/ft^3
Bath Surface Area	9 ft^3 (Nominal)
Bath Volume	27 ft^3(Nominal)
Furnace Pressure	-0.5 to -0.25" W.C.

In addition to these design requirements, the overall design of the furnace shall allow for operation both with and without agitation. Even though the furnace system shall produce a minimum of one (1) metric ton of glass per day without agitation, the overall design of the furnace, especially the following items, shall be capable of meeting the demands of three (3) times the minimum production rate:

a. Electrodes
b. Electrical power supplies and controls
c. Slurry feeding mechanism and controls
d. Vitrified material discharge chamber.

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Finally, the overall design shall be suitable for remote operations, but will permit hands-on maintenance.

1. **Furnace Assembly**

   a. **Outer Furnace Containment Shell** The shell shall be of welded steel base frame, and will integrate the melting and discharge chambers. It shall be designed to support and hang the refractories as well as to accommodate:

      1) A water jacket or coil panels for cooling (where required)
      2) Electrode holders
      3) Removable but fastened lid(s) for both the melting and discharge chambers
      4) Connections for a bottom drain, an overflow drain, and a vitrified material product discharge.

   b. **Refractory and Insulation** Provide with the proposal, the refractory and insulation thicknesses and specifications including commercial grades. Be prepared to supply after receipt of an order: Refractory specifications complete with manufacturer's data indicating chemical composition and physical properties including density, cold crushing strength, modulus of rupture at room temperature and at elevated temperatures, reversible and permanent thermal expansion, porosity, linear change after reheating, load subsidence or creep, thermal conductivity (530 - 1,650 degrees C), abrasion and corrosion resistance, specific heat for temperature ranges, permeability, modulus of elasticity, melting point, and the quantity of field installed refractory complete with scope of work for field installation.

   c. **Electrodes** The electrodes shall be suitable for the application and surrogate feed material. Bench-scale tests have shown graphite to be unacceptable because of the reduction of lead in the melt. Electrode holders shall insulate electrodes from the outer furnace shell.

   d. **Vitrified Material Discharge Chamber with Heaters** The vitrified material discharge chamber shall be heated with electric heaters to maintain molten vitrified material temperatures at the selected operating temperature of the melting chamber.

   e. **Melting Chamber Plenum Heaters** The plenum may need to be heated with electric heaters (seller is to determine) to maintain plenum at selected operating temperature.

   f. **Bottom Drain, Overflow Drain, and Vitrified Material Discharge Spout** Since the silo materials contain heavy metals which may settle out, the furnace shall have the capability of draining heavy metals or conductive sludges from

the floor or lower portion of the furnace. The supplier shall provide one bottom drain tap with manual remote on-off control capable of shutting off flow of molten vitrified material to prevent draining the furnace when tapping these metallic deposits. Since the silo materials contain sulfates, a molten salt or surface scum layer may form on the top of the melt surface, particularly when no agitation is applied. A drain tap which provides manual remote on-off control shall be provided for removing this layer when necessary. In addition, the design shall provide a tapping mechanism to permit manual remote shut-off of molten product discharge from the melting chamber to the tapping chamber. Normal operation will be continuous overflow through the discharge chamber.

g. **Removable but Fastened Lid Assembly(s) with Connections for Feed, Off-Gas, Viewing Cameras, Instrumentation, Plenum Heaters, and Agitation Device** Silo material is a source of hazardous and radioactive particulates and gases (i.e., radon). Consequently, a furnace lid with access/instrument ports shall seal the furnace (as air-tight as practicable) to provide containment of the furnace off-gasses and particulates. The furnace off-gas will be exhausted to an external scrubber/HEPA filter system which is to be provided by FDF. The lid shall include connections for a slurry feed nozzle, an exit nozzle for the off-gas, access ports for thermowells, a pressure tap, nozzles for remote viewing capability, and an access port for the agitator assembly. The supplier is to size suitable off-gas and feed nozzles and provide nozzle location drawings. The lid shall accommodate remote viewing capability at a minimum of 4-inch diameter flanged nozzles and tubes at two locations: melting chamber and discharge chamber. The view ports shall be positioned to allow full view of the melt surface and the pour spouts and risers. Remote viewing will be by closed circuit television cameras. The remote viewing system will be furnished by FDF.

h. **Slurry Feeding Mechanism and Controls** The feed slurry will be provided through a pipeline terminating with a single flanged connection to interface with the equipment.

2. **Agitation**

A means of agitation shall be supplied with the furnace to perform the following functions:

a. Apply shear forces to the molten vitrified material as a means of improving the vitrified material matrix, while maintaining the cold cap.
b. Prevent the settlement of heavy metals and the formation of a sulfate layer.
c. Homogenize the molten vitrified material by thoroughly mixing the melter contents. Any agitation components in contact with the molten vitrified material or plenum gases shall be fabricated from materials suitable for the

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application and surrogate feed materials specified. The overall design of the melter shall allow operation both with and without the agitation. Provisions must be made in the furnace design to allow for the removal of the entire agitation assembly from service, without removing the furnace lid.

3. **Cooling Systems (if required)** Skid mounted cooling unit(s) complete with heat exchangers, pumps, liquid reservoirs, and control:

   a. The agitator (if applicable)
   b. The outer furnace shell (if required) excluding the lid
   c. The feed nozzle (if required)
   d. The sulfate drain skimmer.

   The supplier shall provide cooling systems and insulation as required to ensure the outer surface temperature does not exceed temperatures indicated in the design specifications over the entire operating life of the furnace. All cooling fluid shall be in closed loops. Cooling unit(s) shall be connected to the furnace by non-electrically conductive piping.

4. **Instrumentation** Instrumentation shall be furnished in accordance with requirements as specified. The supplier shall provide adequate access ports and instrumentation to include:

   a. Temperature measurement of:

      1) Molten bath (up to 1,400 degrees C)
      2) Plenum
      3) Off-gas at exit
      4) Each drain/pour point
      5) Discharge Chamber
      6) Cooling Fluid Inlets and Outlets.

   b. **Differential Pressure Measurement of Furnace** FDF to provide furnace differential pressure measurement and control. The supplier shall provide all other control and monitoring instrumentation, including cabling and connectors to junction box, to ensure safe and proper operation of the furnace. FDF will connect to the supplier's junction box and provide cabling to the remote pilot plant Data Acquisition Control System (DACS). The overall design shall be suitable for remote operation, but will permit hands-on maintenance.

5. **Control** Control shall be furnished in accordance with the requirements as specified. Instrumentation shall provide capability for controlling and monitoring:

   a. Furnace:

      1) Temperature of molten vitrified material
      2) Power to electrodes
      3) Current through electrodes
      4) Voltage across electrodes
      5) Temperature of plenum
      6) Skimmer position
      7) Opening/Closing all drain/pour points
      8) Melt bath level.

   b. Agitator (if applicable):

      1) Agitator speed
      2) Agitator position.

   c. Cooling Systems: (if applicable)

      1) Cooling fluid temperatures (inlets and outlets).

6. **Electrical Power System** Electrical Power System shall be furnished in accordance with the requirements as specified.

7. **Services** The services of an on-site technical representative(s) shall be provided to advise or consult during furnace assembly, furnace installation, start-up, and training. The schedule for this work will be assumed to be ten (10) hours per day, five (5) days per week, for ten (10) weeks, which includes the training schedule. The technical representative shall certify the work was done in accordance with the representative's company's requirements.

   The seller's personnel assigned to the site must comply with the FEMP site training requirements.

   A separate hourly rate shall be quoted for a technical representative should the time be required for services that exceed the time specified.

8. **Testing** In addition the melter had to demonstrate its operational capability to process to the surrogate material specified.

As-Built Configuration

The joule-heated melter designed for the project operates at 1400°C using molybdenum electrodes. Unlike other designs, this melter protects the molybdenum electrodes. The triple chamber design is such that the wastes and glass additives are fed directly into the center melt chamber that is separated from the two side chambers containing the electrodes by refractory brick walls. To further protect the electrodes, the side chambers contain an unstirred, borosilicate glass that decreases the amount of oxygen in contact with the molybdenum. Heat dissipation from the side chambers to the center chamber is promoted by the high conductivity refractory brick walls that transfer the energy necessary to melt the waste, without allowing the corrosive elements of the waste to contact the electrodes.

Figure II-1 shows the exterior profile of the melter. The larger area is the furnace section and the smaller area is the discharge section. The outer containment shell of the melter is welded steel. It supports the electrodes, electrode cooling jackets, lids for the melter and discharge chambers, surface drain, bottom drains, bubbler tubes and various thermowells. The contents of the melter are represented in Figures II-2 - II-6. The glass contact refractory is fused cast Monofrax K-3 which is backed by a bonded Zirmul AZS refractory and a castable aluminum oxide refractory. The total glass containment refractory is housed in an internal shell constructed from Inconel 690. Between the internal and external shells is a refractory ceramic fiberboard insulation package to inhibit excess heat loss and wicking of the glass to the outer shell. Above the glass containment area of the melter, the refractory consists of Monofrax H refractory and multiple layers of insulation from inner area to the outer shell. The roof refractory is the same.

To meet the high operating temperature requirements, the melter utilizes molybdenum electrodes and three glass chambers. The center chamber is approximately 27 cubic feet and the two side chambers are sized to contain five electrodes each while immersed in glass. The three chambers are formed by walls of a high conductivity refractory brick, Monofrax E (Figures II-4 & II-5). The three chamber system segregates the waste and glass additives in the center chamber from the molybdenum electrodes in the side chambers. The side chambers contain a borosilicate glass to protect the electrodes and slow down degradation that would occur rapidly, if they were subjected directly to the waste materials.

Figures II-2 and II-4 identify the five air bubblers, the air lift lance, and the bottom drains. The air bubblers were provided as an agitation system for the center chamber glass pool. The design consisted of five molybdenum disilicide tubes placed uniformly through the floor of the melter down the middle of the center chamber. The bubblers provide inlets to the glass pool that permit compressed air to be introduced to the glass pool. Operation of the bubblers disrupts the glass pool by generating a curtain of bubbles. The bubbler system was to provide an oxidizing environment to promote oxidation of metallic components in the waste and prevent forming a detrimental metallic layer on the bottom of the melter glass pool, disrupting the formation of a sulfate layer on top of the molten glass, and increasing convection within the glass pool to improve the production rate.

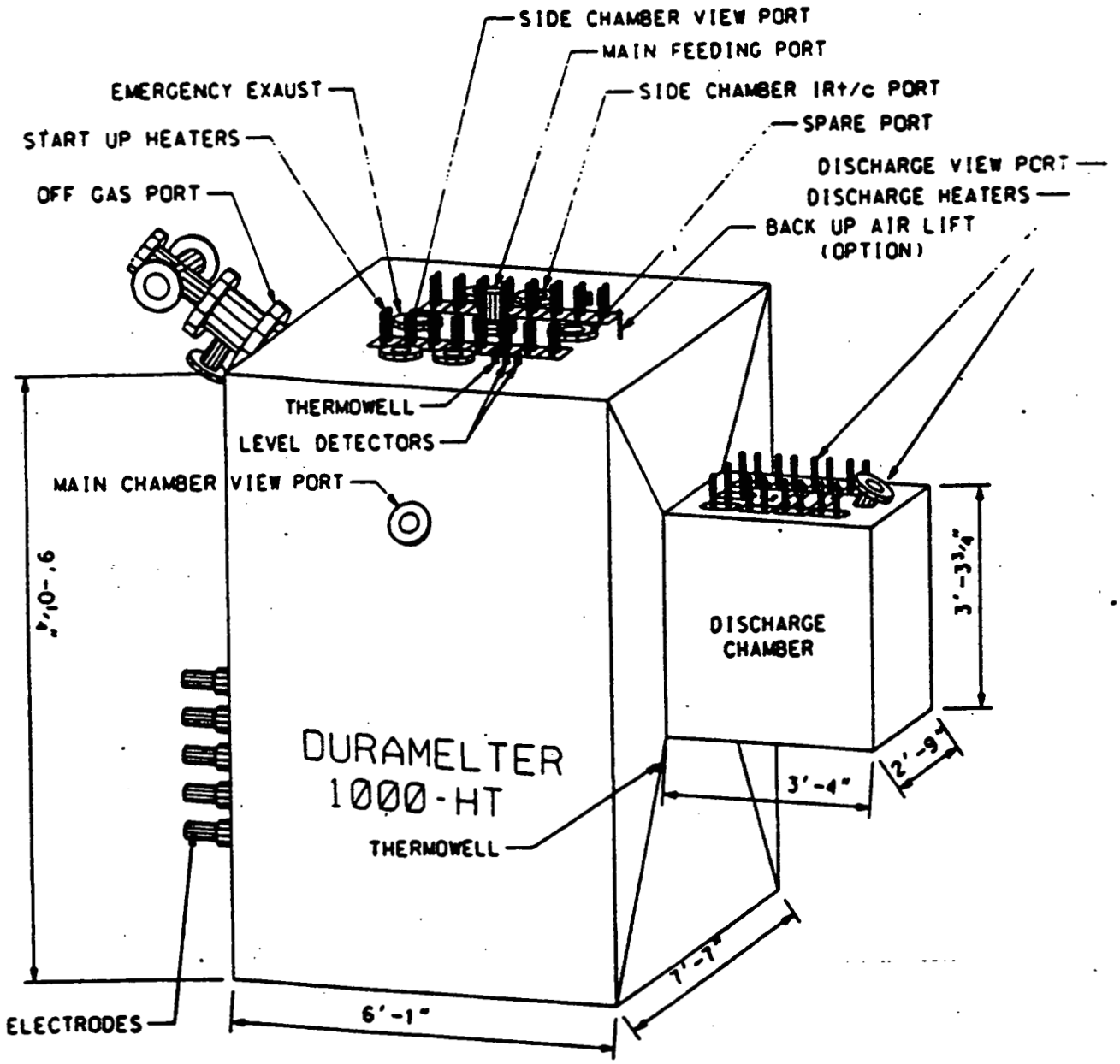
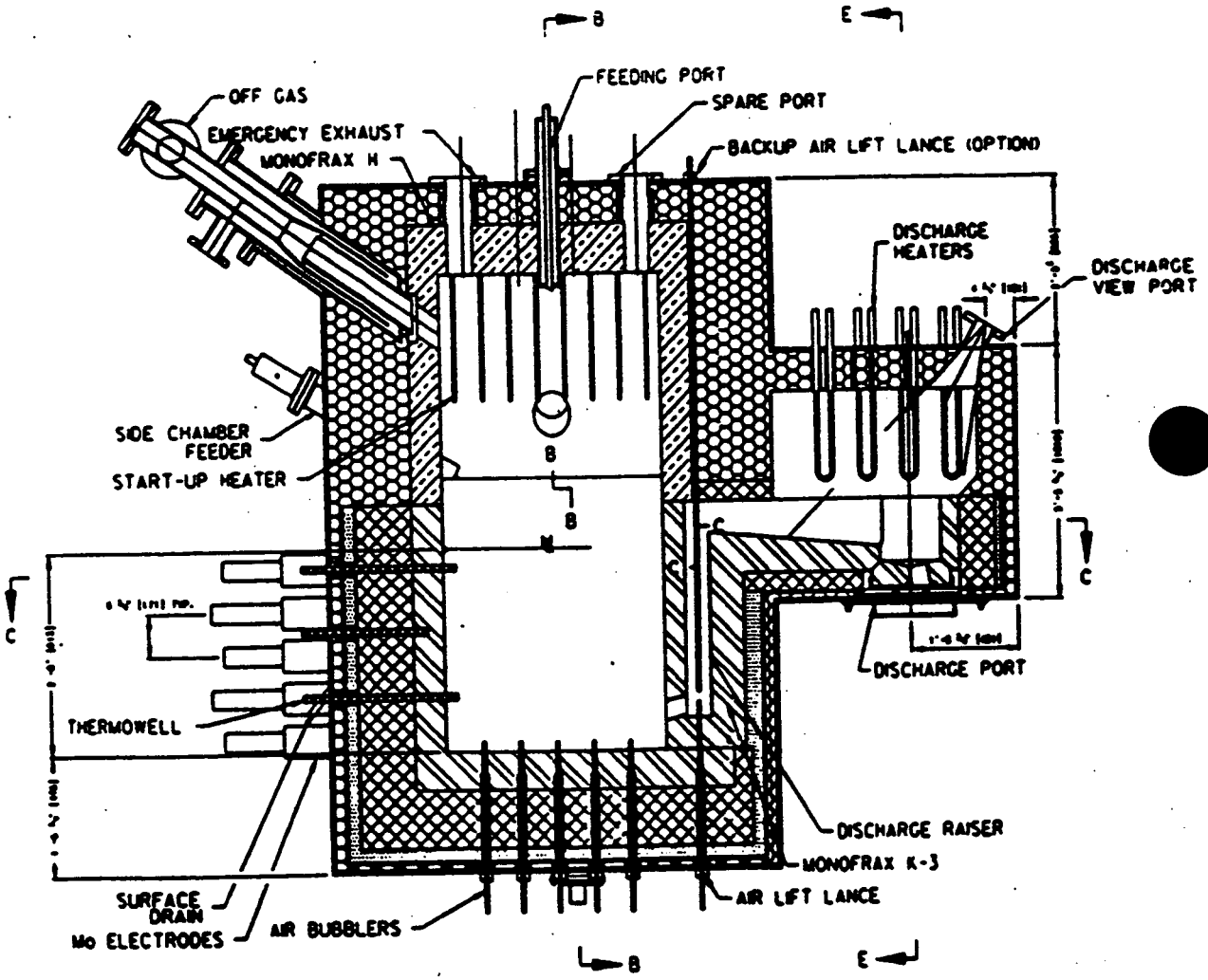


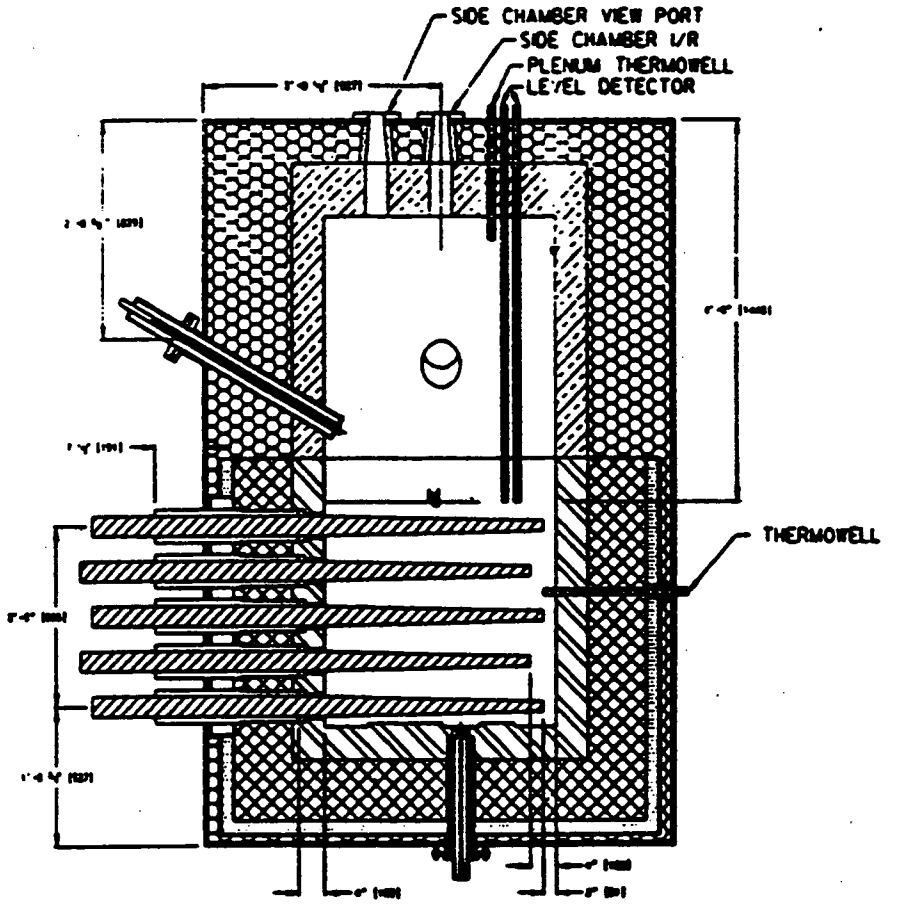
FIGURE II-1

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SIDE VIEW (SECTION A-A)

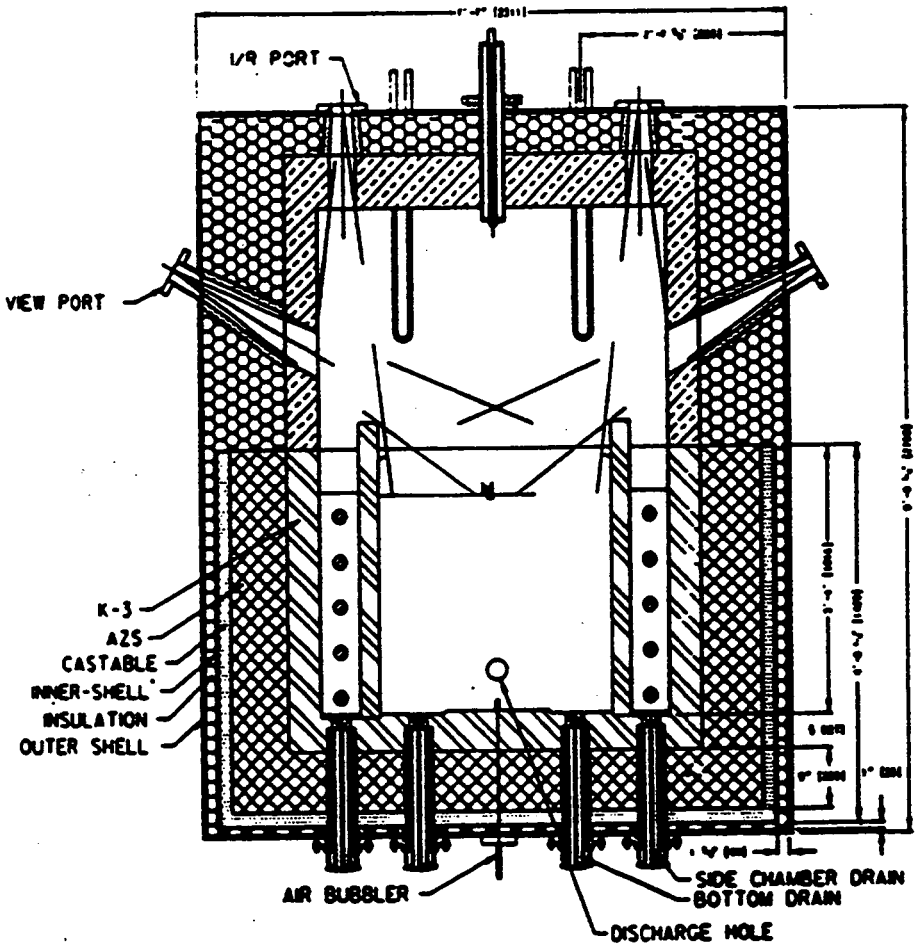
FIGURE II-2



SIDE VIEW (SECTION D-D)

FIGURE II-3

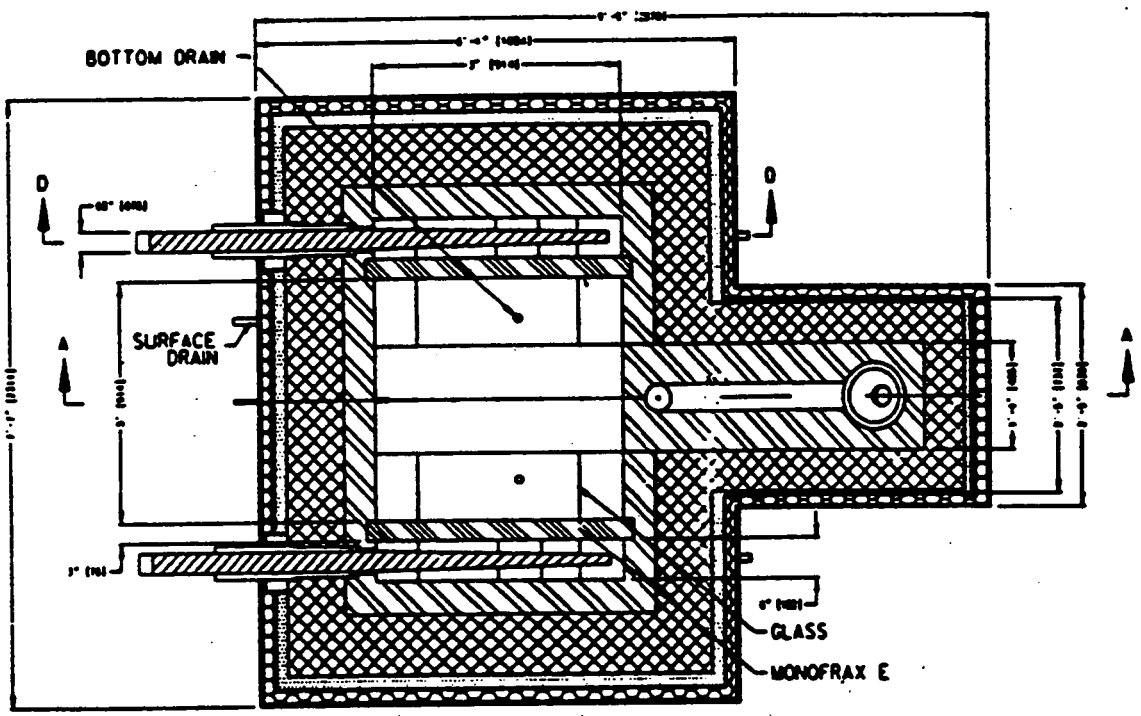
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BACK VIEW (SECTION B-B)

FIGURE II-4

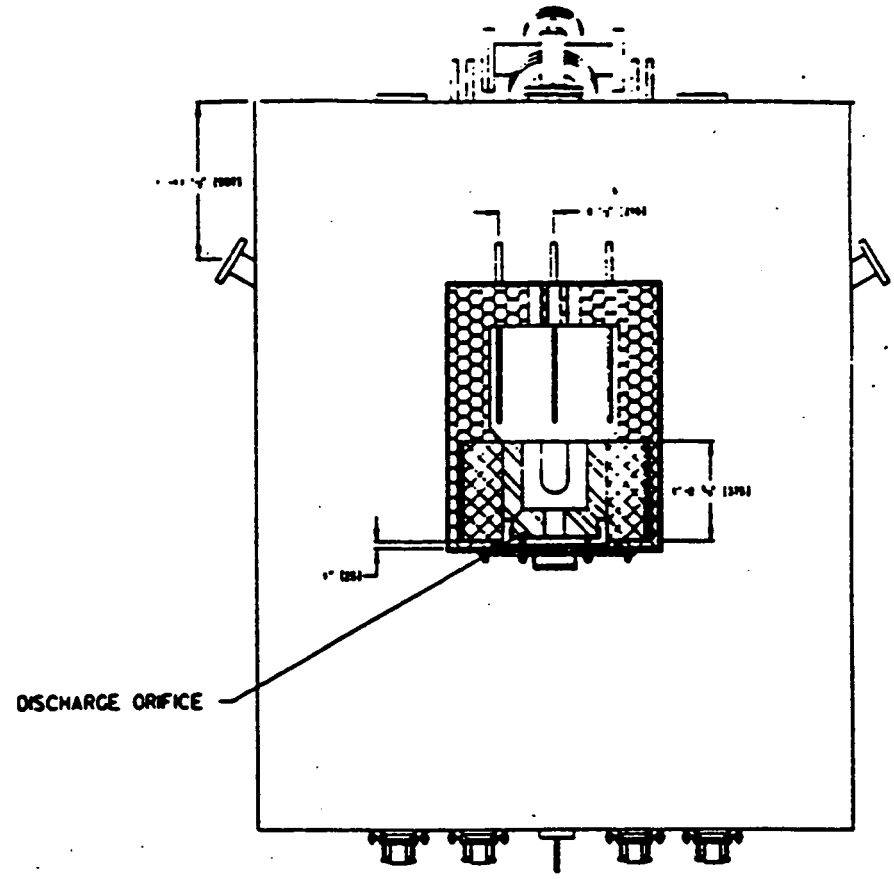
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TOP VIEW (SECTION C-C)

FIGURE II-5

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FRONT VIEW (SECTION E-E)

LEGEND:

- MONOFRAX E (3")
- MONOFRAX H (5")
- MOLYBDENUM (03")
- K-3 (5")
- AZS (9")
- CASTABLE (2")
- INCONEL 690 (1/4")
- INSULATION (1 3/4")
- STEEL (1/2")
- SUPER TUBE

FIGURE II-6

PLENUM VOLUME: 48 FT ^{3}
GLASS VOLUME: 24 FT ^{3}
SIDE CHAMBER GLASS VOLUME: 7 FT ^{3}
(NOT INCLUDE ELECTRODES)

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The air lift lance is also a molybdenum disilicide tube. The air injected through it raises the molten glass up the discharge riser to a level high enough for the glass to flow down the pour spout and out the discharge port.

Four bottom drains were provided to empty the melter. There are two drains for the center chamber and one provided for each side chamber. These have the ability to be remotely activated and could be used only once.

The design process with the melter supplier involved iterations of conceptual presentations with both parties agreeing on a final configuration. In general, the melter provided met the intent of the performance specifications. Final acceptance, was dependent on the successful completion of a performance test with surrogate materials. The test was conducted during the Phase I Campaign. This melter was conditionally accepted (Reference 12).

III. STATEMENT OF FACTS

SUMMARY OF CONDITIONS AND EVENTS

This section of the report describes the operational experiences of the VITPP from May 18, 1996, to December 26, 1996. The report highlights anomalies associated with the melter operations, in accordance with the Event and Causal Factor Chart for Melter Operation Experiences (Appendix D), during Phase I Bakeout and Campaigns 1, 2, and 4. Campaign 3 was deleted because the vitrification of Silo 3 waste exclusively was too problematic due to its high sulfate content.

Tables III-1 and III-2 identify the chemical composition of the surrogate materials being fed to the melter during Phase I testing.

BAKEOUT

Bakeout of the melter began on May 18, 1996. During bakeout, the temperature of the melter was slowly increased to an operating temperature range of 1,150-1,250° Celsius. The purpose of the bakeout session was to cure the refractory material through a specific temperature curve, charge the melter with benign glass, and insert the molybdenum electrodes.

THERMAL EXPANSION AND MELTER PRESSURE TRENDING

Shortly after bakeout started, a loud bang was heard in the melter room (0330 hours, June 6). The source of the sound could not be pinpointed, but it was believed that it may have been caused by thermal expansion of the melter. During the same time period, the center chamber frit feed tube plugged, but the condition was determined to be coincidental to the loud bang. The clog was cleared after mechanical agitation of the feed tube was performed. A trend analysis was generated on pressure indicator PDIC-250 readings to identify if there was a possibility of melter pressurization, but no positive pressures were observed.

AUTO DISCHARGING

Chronic auto discharging of glass from the melter began on June 7. From that date through the end of bakeout, problems with auto discharging caused glass accumulation on the discharge opening and in the discharge chamber. Actions taken to combat the auto discharge problems initially involved the reduction of air flow to the air lift from 2.0 standard cubic feet per hour (scfh) to 1.5 scfh.

One auto discharge was attributed to scheduled maintenance activities. The discharge of glass from the melter eventually resulted in the formation and build-up of solidified glass on the diverter trough. As the glass exited the melter, it cooled and froze on the discharge trough, and clogged the discharge opening. Corrective

Table III-1

	1	2	3	4	5	6	7	8	9	10	11	12
Campaign	Bakeout	1	1	2	2	2	2	2	Post 2	Post 2	Post 2	Post 2
Batches	melter filled	01 to 13		01	02 to 04		05 to 11		12 to 15	16	17 to 18	
Dates	6/6-6/24	6/24 to 7/31		8/26-27	9/4 to 9/8		9/10 to 9/24		10/5 - 10/18	10/20-10/21	10/20 to 11/1	
Compound	Startup Glass Frit	Benign Wet Batches	Glass 16 Glass	Transistion Batch as oxides	Acceptanace Test Batches	Glass	Series D Batches	Glass	Stockpiled Sludges as oxides	Wet 12 as oxide	Wet 16 Batches	Glass
Al2O3	10.92	10.92	10.92	19.40	13.98	15.08	13.40	14.03	14.03	11.00	10.92	10.92
B2O3	10.58		10.58	9.20		10.44		8.82	8.82	10.50		10.58
H3BO3					1.92		6.00					
BaO				4.90		2.60		2.80	2.80			
BaCO3					1.02		1.15					
BaSO4					2.75		2.90					
CaO	14.56		14.56		0.15	7.42	0.14	13.84	13.84	28.00		14.56
CaCO3		25.99			10.64		22.18			5.00	25.99	
CaSO4					3.19		2.70					
Fe2O3				11.50	4.81	5.37	4.04	4.45	4.45			
Fe2(SO4)3					0.79		0.67					
K2O				2.30		1.06		0.85	0.85			
K2CO3					0.75		0.59					
KNO3					1.06		0.89					
Li2O				0.30		0.13		1.15	1.15			
Li2CO3					0.32		2.85					
MgO				10.40	0.24	5.02		3.40	3.40			
MgCO3					1.05		2.33					
MgSO4					1.70		1.43					
Mg3(PO4)2					7.11		4.68					
MnO2					0.18	0.19	0.15	0.16	0.16			
Na2O	14.53		14.53			6.76		6.28	6.28			14.53
Na2B4O7		15.29			13.52		7.88				15.29	
Na2SO4					1.18		1.00					
Na2CO3		20.14			1.90		4.54				20.14	
NaNO3					2.01		1.73					
NiO				0.70	0.14	0.14	0.12	0.12	0.12			
P2O5				7.90		3.84		2.53	2.53			
PbO				10.20	5.09	5.09	5.46	5.46	5.46			
SiO2	45.41	45.41	45.41	23.20	32.16	36.69	32.90	35.49	35.49	45.50	45.41	45.41
TiO2						0.02		0.01	0.01			
V2O5					0.15	0.15	0.13	0.13	0.13			
ZrO2	4.00	4.00	4.00					--	--		4.00	4.00
Bentonite					6.84		3.88	--	--			
CO(NH2)2					3.89		3.32					
SO3								0.50	0.50			
Total Solids	100.00	121.75	100.00	100.00	118.54	100.00	127.05	100.00	100.00	100.00	121.75	100.00

Column comments:

2. and 3. ZrO2 was replaced with SiO2 in batches of campaign because "refined ore-grade" ZrO2 used was slightly contaminated with radioactive thorium.
5. and 6. BaSO4 was used in the first batch. CaSO4 was stoichiometrically substituted for BaSO4 in the remaining batches.
7. and 8. Because thickening problems, bentonite was substituted with chemical equivalents after batch D-4 and CaSO4 was stoichiometrically substituted for BaSO4. Also, borax (Na2B4O7) was found to cause slurry thickening problems. It was replaces with stoichiometric amounts of boric acid (H3BO3) and sodium carb LiO2 contents of the actual glass in the melter was high because of side chamber inleakage. W% Li2O in batches were : 1-2.32, 2-3.27, 3-2.55, 4-2.67, 5-2.18, 6-2.72, 7-2.7, 8-2.48, 9-4.36, 10-3.98, 11-2.86.
9. These batches were previously aborted batches. The chemistry of these batches was "pulled" to approximate the Series D already in the melter. batches typically had less lead and more alumina.

Note: Each batch produced approximately 1 MT glass. Quantities in table columns are those required to produce 100 parts of glass by weight.

000066

	13	14	15	16	17	18	19	a	b
Campaign	4	4	4	4	4	4	4	na	na
Batches	1 to 2	03 to 06		07 to 08		09	Planned	na	na
Dates	11/30-12/1	12/5 to 12/11		12/11 to 12/16		12/22-12/24	not done	6/8-11/29	12/5-12/26
Compound	Translation Batches as oxides	Series A Batches	Glass	Series A Modified Batches	Glass	Transition Batch as oxide	Low Temp Series A Glass	Side Chamber Frit	BaO Side Chamber Frit
Al2O3	3.50	6.05	6.05	6.15	6.15		3.54	12.70	11.43
B2O3	15.00				10.51	18.60	14.29		
H3BO3		23.44	13.20	18.67					
BaO	5.70		4.52		4.09	3.40	4.19		15.75
BaCO3		1.86		inleakage					
BaSO4		4.68		4.24					
CaO	2.00	0.22	5.00	0.20			1.95	6.40	
CaCO3		8.44		23.32	13.31				
CaSO4		0.11		0.10					
Fe2O3	5.10	3.98	3.98	3.60	3.60	2.50	3.69		
Fe2(SO4)3									
K2O	0.60		0.65		0.58		0.60		
K2CO3		0.95		0.86					
KNO3									
Li2O	0.90	3.79	1.53	inleakage	1.28	6.30	4.08	10.10	9.09
Li2CO3				1.58					
MgO	1.40		1.36		1.23	0.80	1.36	4.10	3.69
MgCO3		1.75		1.59					
MgSO4									
Mg3(PO4)2		1.14		1.03					
MnO2									
Na2O	4.10		5.80		7.33	6.80	7.26	5.30	4.77
Na2B4O7									
Na2SO4									
Na2CO3		9.65		12.29					
NaNO3		0.43		0.39					
NiO	0.60								
P2O5	0.90		0.82		0.56	0.30	0.57		
PbO	11.60	8.82	8.82	7.98	7.98	7.60	8.19		
SiO2	47.30	47.96	47.96	43.38	43.38	52.40	50.28	55.30	49.78
TiO2									
V2O5									
ZrO2								6.10	5.49
Bentonite									
CO(NH2)2		0.80		1.45					
SO3	1.10		0.51			1.30			
Total Solids	100.00	124.07	100.00	126.83	100.00	100.00	100.00	100.00	100.00

Column comments:

14. thru 17. The inleakage of BaO and Li2O from the side chambers into the center chamber was accounted for and subtracted from the next batch. Therefore, unlike Campaign 2 high Li2O concentrations, the Li2O concentration in the actual glass was close to the recipe.

16. and 17. 50 lbs of LiCO3 was added during Campaign 4 Batch 7 to pull the viscosity close to the expected Series A modified glass recipe. This was done to reduce excess foaming in the melter center and side chambers.

a. and b. These are the side chambers glass frit used during Phase I. The BaO glass was used only during Campaign 4. Note that these are not glass concentration in the side chambers because of communication between chambers. Late in Phase 1, the side chambers (especially, the West chamber) started to approach the Center Camber's glass composition.

Note: Each batch produced approximately 1 MT glass. Quantities in table columns are those required to produce 100 parts of glass by weight.

actions for these events included raising the discharge chamber temperature to melt the glass and/or flushing molten glass from the melter to clear the chamber and the opening. These actions also resulted in lowering the level of the glass pool, which also reduced the possibility of auto-discharging. In one case, the discharge hole was completely bridged and had to be cleared using an acetylene torch and air lifting glass from the melter. With the auto discharging of glass, the discharge chamber high-level alarm actuated several times. The level detector was adjusted to two inches above the bottom of the discharge chamber trough.

CRACKS IN THE WEST E-WALL

On June 22, cracks in the west E-wall were discovered. The following day it was recorded that cracks were observed in both melter isolation walls. The cracks were represented in sketches drawn in System Operability Test book 25-SU-0034.

CAMPAIGN 1

Campaign 1 began on June 24. The intent of Campaign 1 was to establish continuous operation of the melter, verify melter temperature and power control, synchronize operation of the melter and gem machine, and increase glass output to three metric tons per day at an operating temperature range of 1,150° to 1,250° C.

SPARKS AND FALSE CONCERNS OF OXIDATION IN THE SIDE CHAMBERS

Soon after benign slurry was fed to the melter during the last week of June 1996, sparks were observed at the surface of both side chambers. More specifically, flashes of bright yellow light about ¼ inch in diameter and occurring every 5 seconds or so were observed through the side chamber sight glasses[1]. The sparks appeared to result from bubbles of gas breaking at the surface of the melt. These sparks are noted in the logs as "signs of oxidation" in the east and west side chambers. Initially, it was felt that the molybdenum electrodes were oxidizing (or partially oxidizing) and/or flaking off small bits that were carried to the surface where they competed burning (molybdenum is pyrophoric; it can rapidly oxidize at 400°C). This theory does not explain the gas bubble, that breaks to produce a spark. Molybdenum trioxide is the only one of several molybdenum oxide compounds that is a gas at glass melting temperatures and could form a bubble. However, this does not explain the combustion at the surface because molybdenum trioxide is the most oxidized molybdenum oxide compound and cannot "burn" any further.

One possible explanation for the sparks is that, because the side chambers are starved form oxygen, sodium oxide in the melt may be reduced to elemental sodium. Since sodium metal boils at 892°C, microgram quantities might be forming bubbles and flashing (burning) when the bubbles break and contact the air. The mechanism for the reduction of sodium involves a weak electrolytic potential forming between two electrodes in the same chamber but at different temperatures.

The sparks caused no operational difficulties and would disappear for periods of time, but at the time it was felt this was a sign that the electrodes were oxidizing. The sparks were more frequent in the west side chamber. Both side chambers were fed with frit after the sparking condition was observed. Because of the concern for corroding electrodes, all new glass samples were tested for molybdenum content. Glass analysis results were received on June 28, which indicate that molybdenum concentrations were below detection limits.

CORROSION OF MOLYBDENUM DISILICIDE COMPONENTS

Operations was required to adjust the height of the glass level indicators in the side chambers. These level indicators were molybdenum disilicide rods that completed an electrical circuit when contacting the molten glass. Molybdenum disilicide is conductive, especially at high temperatures.

On June 28, the east side chamber level indicator was found to be one inch lower than west side chamber. The east side chamber probe was removed to inspect for wear. The probe had not lost length, but was visibly tapered from corrosion. Operations continued to make adjustments to the level indicators until July 29 when the level probes were removed from both sides and measured. Corrosion was noted on both level indicators and the corroded tips were cut off to make the ends square again. The west side level indicator was broken and replaced with a new probe. On August 2, the east chamber level probes were not functioning properly. The electrode was found almost fully exposed and well above the glass level and coated with glass. At this time, no concerns were raised about the possibility of other molybdenum disilicide components eroding in the melter, such as the bubbler tubes.

GLASS MIGRATION IN THE DISCHARGE OPENING

As a result of adding side chamber frit to combat the oxidation process of the molybdenum electrodes, the melter experienced frequent auto discharging events. On July 5, the discharge chamber high-level alarm actuated intermittently. Operations did not believe the alarm was valid because raising the discharge chamber temperature did not clear the alarm. On July 12, a large chunk of glass was removed from the discharge opening. A large amount of glass still remained. It was determined that the glass was exiting the melter from an unknown location. After removal of the remaining glass, it was noticed that the discharge orifice brick had a large crack in it. For the remainder of Campaign 1, Operations had to take actions to remove glass from the discharge chamber. After using a torch to melt the glass from the discharge chamber on July 29, Operations documented that the glass was coming from the northeast corner between the brick and the shell, under the discharge opening. On August 2, glass continued to migrate to the area below the opening, but no auto discharge was witnessed. The same day voltage measurements were taken for SCR 1.

BUBBLER 5 INOPERABLE

Another problem discovered in Campaign 1 was oil in the airflow indicator to Bubbler 5. The oil forced Operations to increase air flow to the bubbler to keep indication from going to zero. On July 10, the tubing was partially pulled from the ferrule and re-tightened. Bubbler air flow indication immediately went to zero and Operations concluded that the previous air flow indication was false. The air leak was located downstream of the air flow indicator and upstream of the bubbler. It could not be determined how long Bubbler 5 had not been functioning.

BOTTOM DRAIN 3 COOLING JACKET LEAK

A blue flame was noticed in the east side chamber on July 28 at 1800 hours. Operations could not determine the reason for the flame, but actions to combat the problem were to add frit to the east side chamber. The blue flame returned on August 1 and Operations speculated the broken piece from an electrode was burning and was the source of the flame. Operations added 100 pounds of frit, but the flame was not eliminated. Later in the shift, the flame was drastically reduced during an outage to support maintenance activities. Continued efforts of troubleshooting included shutting down SCR 1, which had little effect on the flame; therefore, Operations concluded that an oxygen source was providing the conditions for the flame. Later that day, the east bottom drain temperatures were found to be out of specifications (high) and Operations performed a visual inspection and observed what appeared to be glass (later determined to be graphite paper) around the bottom drain. Operations also saw water on the plunger shaft and observed that the thermocouple was broken. Operations increased cooling water flow to the east bottom drain and observed the flame increased in intensity. This confirmed that the cooling jacket of the east bottom drain was leaking coolant (containing glycol, chlorides and sulphur) into the melter. Operations decided to isolate the East bottom drain from the rest of the melter cooling system and then provided it with a separate cooling system. The installation was completed on August 2 and the system was comprised of a 55-gallon drum filled with distilled water, a pump, and supply and return piping. This corrective action stopped the flame inside the melter. The cooling system was modified on August 3 from a closed-loop system to a one-time-through system. The closed-loop configuration did not allow sufficient cooling without changing the water inventory every hour.

ELECTRICAL SHORT TO GROUND

During the inspection of the east bottom drain thermocouple on August 1, inspectors found that when glass contacted the east bottom drain, the drain went to the voltage potential of the melter bath and burned the thermocouple at the point where it contacted ground potential. On August 3, Operations noticed that bottom drain 1 temperature was also rising. Cooling water to the drain was increased to maximum flow and cooling water flow to all other components outside of the glass pool was

decreased. The east bottom drain was repaired with a cooling jacket insert on August 23 and the jacket was reconnected to the permanent cooling water lines.

A potential of approximately 80 volts was measured across the buss, and 40 volts were measured between leg to ground. Current checks of the ground wires indicated there was a current flow of up to 20 amps to the ground buss. This indicated that there may have been a melt pool to ground short in the melter. A discharge chamber chute liner was fitted into place on August 4 and an Inconel shoe was installed on August 18. Despite these efforts, the discharge chamber still filled with glass migrating from the cracks in the discharge orifice brick.

The glasses in the melter at this time were the startup glass in the center chamber and the electrode glass in the side chambers. Neither of these glasses contained lead, so molten lead could not cause the short.

CAMPAIGN 2

Campaign 2 began on August 26. During this campaign, the intent was to displace benign glass of Campaign 1, perform a 36-hour acceptance test, an 8-hour gem machine acceptance test, and run a Series D surrogate material at an operating range from 1,250° - 1,350°C.

Urea was added to the slurry feed to aid in the destruction of sulfates, and thus, maintain glass production rates. A concern with using reductants to destroy sulfates is that the concentration and reduction power may be too strong, and thus produce molten metalic lead or lead sulfide in the bath. Urea was the reductant chosen from several tried during minimelter (bench-scale) testing because it is a simple organic compound that virtually produces only gasses when oxidized and leaves no carbon residue that can be carried down into the glass melt and continue to reduce the glass bath further than intended. Therefore, urea can only apply its reduction power at or above the surface of the glass bath.

During the campaigns, the color of the glass was monitored for signs of reduction. All the glasses contain hues of brown in them, which is an indicator that they are oxidized, not reduced. The ratio of Fe^{+2}/Fe^{+3} or $Fe^{+2}/(Fe^{+2}+Fe^{+3})$ is used as an indicator of redox in the glass. An oxidized glass looks brown because Fe^{+3}, which is the oxidized species of iron, produces a brown color. Whereas, Fe^{+2}, which is the reduced species of iron, produces a blue-green color. Therefore, if the brown hue leaves the glass there is concern that the glass may become too reduced and metalic lead or lead sulfide is possible.

Reduction studies show that simple reduction of the surrogate glass (e.g., through the use of urea) will produce lead sulfide because of the presence of dissolved sulfur (sulfate) in the glass. It takes a high (localized) reduction potential (such as molybdenum metal, molybdenum disilicide or "reboil") to produce molten metal. No lead sulfide precipitation was identified during the melter inspection; only lead metal was observed. Glass samples from the lead-bearing campaigns where also analyzed and showed only oxidized conditions

with $Fe^{+2}/(Fe^{+2}+Fe^{+3})$ ratios ≤0.33 or ≤33% reduced. Precipitated lead metal or lead sulfide is not known to happen under these redox conditions.

LID HEATERS

On August 27, Operations discovered that the lid heaters were not functioning properly. During troubleshooting, it was found that the two most northern lid heaters on the east side were directly shorted to ground. The failure of the lid heaters was anticipated by project personnel and the failure had no significant effect on operations and glass chemistry.

AUTO-DISCHARGING

Throughout Campaign 2, auto discharging events and glass migration in the discharge chamber continued to be a problem. On August 29, it was recorded that glass appeared to be coming out above the "dam" in the northeast and southeast corners. After cleaning, a thin, one-inch-long partially molten glass sliver remained in the northeast corner of the dam where the dam meets the orifice retaining plate. The discharge chamber opening plugged again on several more occasions during September and October.

BOTTOM DRAIN 1 COOLING JACKET LEAK

On August 28, flashing and bubbling were observed in the west side chamber. Operations discovered that the problem was a failure of the cooling jacket for Bottom Drain 1. The same temporary cooling system that was used for the east bottom drain was installed and leakage was decreased, but some bubbling in the west side chamber persisted. A new cooling jacket sleeve was installed on September 1; the same modification was made to Bottom Drain 2 and the spare bottom drain as preventative measures.

FOAMING AND TEMPERATURE EXCURSIONS

During the acceptance test (performed during Campaign 1), foaming in the center chamber was observed. The foaming was expected due to the addition of sulfates to the melter. On September 4, the actions taken to combat the foaming in the melter resulted in several temperature excursions. The maximum temperature in the melter (determined by modeling) was estimated to have been 1,407° C. After the completion of the acceptance test, other foaming events were experienced, primarily in the west side chamber. On September 18, the foaming was so extensive that foam flowed over the west E-wall. The following day, foaming was noticed in the east side chamber.

Actions taken in response to the extensive foaming observed in the melter during this period included adding water, adding frit, and varying temperature. On September 25, an additional crack was discovered in the west E-wall. The crack was described

as a "large crack which runs from the top of the wall down to the glass."

BOTTOM DRAIN 3 "GLOW" OBSERVED

From September 24 until the end of Campaign 2, Operations attempted to abate an observable glow at Bottom Drain 3 and continued high temperatures at Bottom Drain 1. Operations determined that the heat in Bottom Drain 3 was from joule heating. When the discharge chamber temperature was reduced (SCR 2) to 1,170° C, the intensity of the glow was reduced. On September 26, it was reported that the intensity of the glow varied during the day and that the variance seemed to depend on the power of SCR 1 and discharge chamber temperature. Initial efforts to combat the glow in Bottom Drain 3 were the application of air for external cooling and increased cooling water flow. On October 30, VITPP personnel decided that the path forward for bottom drains was to leave them "as is" until after Campaign 4 was completed. This decision was reached after attempts to remove and replace the cooling sleeves were unsuccessful.

CAMPAIGN 4

Campaign 4 began on November 29. Campaign 4 was intended to simulate the chemistry of Silos 1 and 2 materials through a Series A surrogate. This campaign was divided into four parts:

Part 1 included running Series A glass at 1250° C,
Part 2 ran Series A at 1150° C,
Part 3 ran Series B glass (Series A plus bentonite), and
Part 4 tested maximized melter efficiency and capacity.

PHASE SEPARATION IN DISCHARGE CHAMBER

On December 1, 1996, Operations discovered some sulfates in the discharge chamber opening. This led personnel to believe that there must be a hole somewhere in the south wall of the melter. On December 11, Operations pulled the air lance to see if the hole in the South wall could be seen. During the inspection, no hole was observed. After the December 26 event, it was discovered that a very large cross-shaped crack developed in the south wall, below the level of the melt pool.

FOAMING EVENTS

Just after Campaign 4 began, Operations added 100 pounds of frit to mitigate early signs of side chamber foaming. However, foaming occurred in all of the chambers on December 3. The foaming caused difficulties in maintaining center chamber temperature. The foaming was reduced in the east and center chambers, but west chamber foaming persisted until December 9. On that day, it was noted that frit additions did not seem to help reduce the foaming, and control of melter temperature

was very difficult. Operations noted that foaming was a problem when operating above 170 kilowatts (KW). When power was reduced to below 140 KW, the low melter temperature then became the primary problem. On December 10, the foam flowed over the west E-wall during third shift. The deterioration of both E-walls was noted and concerns about their structural integrity were documented.

After December 11, only minor foaming events were documented in the log. The glass chemistry was changed at this time. As noted in the Campaign 2 Operable Unit 4 Vitrification Pilot Plant Phase I Interim Treatability Study Report, it was felt that the Series A glass run in the minimelter may be too viscous to run in the melter. Therefore, the modified Series A glass recipe in the study report was tried. This glass was less viscous and appeared to help reduce the foaming because the bubbles popped as they broke the surface without build-up of foam. Also, the urea was increased in attempt to further reduce the sulfate concentration and reduce the high levels of sulfates off-gassing, which is attributed to causing most of the foaming.

The new recipe lowers the viscosity of the bath, but it would take quite some time for the new recipe to have an effect on the bath. This is because it takes days to flush the old glass out of the melter and replace it with the new recipe. Therefore, lithium carbonate was added directly to the melter to lower the bath's viscosity before feeding the new surrogate recipe.

BOTTOM DRAIN OVERHEATING

VITPP Operations continued to have problems with the cooling system for the bottom drains. It was anticipated that problems would still be experienced, based on the decisions made during Campaign 2 and the inability to remove the modified cooling jackets from the bottom drains.

MELTER PRECIPITATION

On December 16, metal was discovered on top of the coupling of Bottom Drain 3. The metal was removed and sent to the lab for analysis. On December 19, the lab results were received and it was determined that the metal from the bottom drain was 58% lead (Pb). Cooling water leaks, bottom drain overheating, and plugging of the lines continued to be problems until the melter drained on December 26.

GLOW OBSERVED AT BUBBLERS 2 AND 3/MELTER SELF-DRAINS TO THE BOTTOM DRAIN CONTAINER

On December 26, at the beginning of day shift, the operations manager and an electrician observed a glow in the areas of Bubbler 2 and Bubbler 3. Immediate actions to cool the bubbler area included directing extra cooling air toward the glow and increasing monitoring frequency of the melter bottom. Operations determined that the glow was not the result of an electrical short, because melter power had no effect on the intensity of the glow. At 2222 hours on December 26, as operations was continuing to formulate an action plan to combat the glow at the bubblers, the melter self-drained through a hole formed at Bubbler 3.

III. STATEMENT OF FACTS

CHRONOLOGICAL LISTING OF FACTS ASSOCIATED WITH MELTER

The following chronological listing of events are melter excerpts taken from the VITPP project logs and records for analysis of root cause, contributing causes, and operational lessons learned. The asterisked (*) items were considered potentially significant to the event being analyzed, and notes in parenthesis are provided for clarification.

5/18 *COMMENCED BAKEOUT.

6/5 *Introduced glass and inserted electrodes.

6/6 Witnessed a loud bang in the melter room at approximately 0330. May be due to melter expansion. Could not identify source of sound. The only coincidental condition was the the Center Chamber frit feed tube was plugged and required hammer jolt to free frit. All instruments read normal; no positive pressures were recorded.

6/7 *Discovered that melter was auto discharging. The trickle flow to the air lift was reduced from 3.8 scfh to 2.0 scfh. The discharge was stopped.

*Discovered that melter was again auto discharging. The trickle flow to the air lift was reduced from 2.0 scfh to 1.5 scfh. The discharge was stopped.

*A third auto discharge was observed. Trickle flow was readjusted from below 1 scfh back to 1.5 scfh. The rotometer may have been bumped and changed.

*Some unplanned glass has auto discharged during the shift as the melter/gem housing was breached from time to time during several scheduled maintenance activities.

*Discharge chamber high-level alarm. Frozen glass must be removed from the probe.

6/11 A plug of glass slowly built up on the diverter trough due to a combination of very slow discharge and increased cooling on the diverter. This plug eventually closed off the orifice of the discharge chamber. The discharge chamber temperature was raised 25°C and the diverter cooling reduced. The glass plug melted and the discharge chamber drained within 30 minutes.

6/12 Flushed a small quantity of glass through the discharge chamber to the startup drum to remove solidified glass which had built up around the orifice.

Discharge chamber level detector still needs to be adjusted.

6/12 Melter auto discharged. Trickle air increased to 3 scfh for 1to 2 minutes then returned to 1.5 scfh. This was done to lower the main chamber level to stop the auto discharge.

6/12 *Melter auto discharged again; same response action taken.

*Discharge chamber level probe removed.

6/13 Flushed a small quantity of glass through the discharge chamber to the startup drum to prevent solidified glass from building up around the orifice.

*Begin the practice of "burping" the melter approximately once a shift or when trickle discharge is observed. This will keep the orifice from plugging and also familiarize the operations staff with the normal mode of operations when running.

6/14 *Discharge chamber level alarm in continuous alarm.

Melter auto discharged for approximately 1/2 hour. Air lifted for 15 minutes to discharge glass from melter.

6/15 Melter auto discharged. Increased trickle air for 10 minutes to discharge glass from melter. Discharge chamber plugged, reduced trickle air to stop discharge. Unplugged discharge chamber. Increased trickle air for 10 minutes to discharge glass from melter.

*Removed old discharge chamber level probe. Aluminum oxide sleeve broke.

*Installed new discharge chamber level probe, which broke.

6/17 Identified glass forming in the discharge orifice. Verified discharge orifice clear.

6/20 Two auto discharges.

6/21 Discharge chamber is completely bridged over and is gradually filling due to some auto discharge. The discharge chamber had been freezing over for several hours before the entire opening completely closed.

6/22 Auto discharge. In about 2 hours, the discharge hole was completely bridged and auto discharge continued as the discharge cavity began to fill. Discharged using an acetylene torch and air lifting at 2.5 scfh.

*Contractor representative and system engineer discovered a crack in the west E-wall.

Discharge chamber plugged and cleared.

6/23 *There are now cracks in both melter isolation chamber walls. See sketch in SOT book 25-SU-0034.

6/24 *COMMENCED CAMPAIGN 1 Batch 1

*Noted evidence of oxidation in the west and east side chambers throughout the shift. The oxidation was more frequent in the west side chamber. The chambers were fed after each oxidation per contractor representative's direction (What was thought to be oxidation appeared as bright bubbles rising through the side chamber glass).

*Set side chamber level detector warning probes to 2" above the electrodes per contractor representative.

6/25 *Raised side chamber level probes 1" above previous setting.

*Added molybdenum to list of metals analyses for glass samples.

*West Plenum temperature increased to approximately 1,400°C after working on side chamber level probes.

6/26 *Removed Infrared (IR) detectors from east and west side chambers.

6/28 *Pulled TI-7S (discharge chamber level probe), found it broken in two pieces.

*Glass conductivity of the center chamber is too high according to system engineer and contractor representative.

*All samples requiring molybdenum concentrations (east and west chamber glass) resulted in below detectable levels.

*East side chamber glass level detector found to be 1 inch lower than west side chamber glass level detector.

*Cracked section of west side E-wall found to be shifted toward main chamber. Piece no longer lines up with the edge of the wall.

6/29 Glass trickled from the discharge orifice for most of the night. May be auto discharging.

7/1 *East and west side chamber level probes adjusted to 1" above the top electrode.

7/5 Auto discharge after center chamber frit addition.

*Discharge chamber high level alarm is alarming intermittently. Does not appear to be valid. Raising discharge chamber temperature did not clear alarm.

7/9 *Pulled east side chamber level probe to inspect for wear.

*The oil in FI-005 (Air Flow Bubbler 5) is starting to cause problems. The oil is forcing the floating ball down to almost zero then comes back to six scfh. Flow was increased to nine scfh to keep it from going to zero.

7/10 *The tubing on FI-005 (Air Flow Bubbler 5) was partially pulled from the ferrule. Tubing was placed back into ferrule completely and tightened. FI-005 then went to zero flow. This indicates that the bubbler is plugged since all of the air was coming out of the tubing leak.

*0415: Bubbler 5 air flow is fluctuating up & down. Appears to have oil in line.

0503: FI-005 was changed (from 6 scfh) to 9.0 scfh to compensate for oil in line.

7/11 *0600: FI-005 loss of air flow. Prior readings were false due to air leak at B-nut on rotometer.

7/12 *Removed large chunk of glass from below the discharge orifice. A large amount of glass is still in place. The glass is getting out of the melter from an unknown location. After removing the glass it was noticed that the discharge chamber brick has a crack in it.

7/13 Took picture of glass formation below discharge chamber.

7/15 Cleared glass from the discharge chamber orifice using torch. Found the source of the glass to be on the west side of the orifice between the refractory and the steel. The north, east, and south seem to be clear.

*West melter wall has developed an array of cracks similar to that of a windshield hit with a rock. These are thermal stress cracks based on contractor representative's opinion.

7/25 *Probed both side chambers to determine the top electrode length. Determined that the top electrode on the east side has eroded back approximately 12 inches (Recorded measurement; took no further action).

7/27 There is a build-up of glass that appears to be coming from the south side of the discharge chamber that requires constant attention and frequent cleaning.

7/28 *Melter feed was terminated in order to remove frozen glass from the discharge orifice, dissolve the cold cap, and add sodium carbonate and center chamber frit directly to the center chamber to adjust the glass viscosity.

A blue flame was noticed in the east side chamber at around 1800 hours. Seventy-five pounds of frit were added to the east side and the flames were no longer seen. At 0415 hours, the east side chamber was measured to be 2.75" higher than the west side. Soon after the measurement, flames were noted on the east side. Fifty pounds of frit were added to the east side and the flames were no longer seen.

7/29 *Sampled both side chambers and took levels. The east side was 58.25" and the west side was 57" to the top of the glass. The east side glass was 3" above the electrode. Probed the east side and confirmed that the top electrode is broken.

*The level probes were removed from both sides and measured. The eroded tips were cut off to make the ends square again.

Used torch twice to melt the discharge chamber glass plugs. The glass between the brick and the shell is coming from the northeast corner under the orifice.

7/30 *East side chamber level probes were lifted one inch each.

The electrodes remain covered with glass.

7/31 Discharge chamber level probe sheath has dropped off of the refractory and is sitting on the bottom of the discharge chamber chute.

*Crack found in the lower end of the discharge chute.

*Inspected underneath melter. No leaks, drips, or loose tubing fittings were found.

8/1 *There is a flame in the east side chamber. Added 100 lbs. of frit but flame was not eliminated.

*Shut down SCR 1, observed flame to determine if changes could be seen with the power off. Subtle changes were noted.

*Checked east bottom drain temperatures on round sheet and found that after 0000 hr. 8-1-96, the temperature reading was noted as out of service.

*Visually inspected east bottom drain. Also saw water on the shaft and found the thermocouple broken.

*Increased cooling water flow to east bottom drain and determined that the blue flame increased in intensity. Adjusted cooling water flow lower to see if the flame could be reduced or stopped. This was not effective. Isolated the east bottom drain from the rest of the melter cooling system and provided a separate cooling system.

Developed inspection plan to measure bottom temperatures, replace thermocouple, measure bottom drain to ground resistances, and performed the inspection. Found that when glass contacted the east bottom drain it went to the voltage potential of the melter bath and burned the thermocouple at the point where it contacted ground potential (bottom drain plunger thermocouple, which is isolated from ground, had burned in two where it contacted the bottom drain gear box, which is grounded).

8/2 *Completed installation of a temporary cooling system. The blue flame stopped immediately. The phenomenon has subsided since.

*At 0300, the east chamber level probes are not functioning properly.

*Glass was migrating into the area below the orifice. Attempted to clear glass from discharge chamber. Due to the constant migration of the glass in this area and the temperature of the discharge chamber (1,300°C) all of the glass in this area was not able to be removed, so this attempt was stopped. When the activity was stopped the orifice was completely open, but there was a large quantity of glass in the area just below and behind the orifice.

*Contract representative made voltage measurements at SCR 1. Current checks of the ground wires indicate that there is as much as 20 amps, on part of the ground buss.

8/3 *Modified the temporary spare bottom drain cooling system so it operates as a one-time-through system rather than a closed loop.

8/3 *Increased cooling water flow to maximum to bottom drain 1 and then decreased all other non-essential users.

*Installed volt ohm meter on Bottom Drain 1 thermocouple to monitor potential to ground.

8/4 *Switched the thermocouples for Bottom Drain 3 and the spare bottom drain. They were swapped below the melter by the thermocouple. Switch position 16 and 17 have the correct numbered wires per the drawing and now read the correct drain as currently labeled on the melter control panel.

Fixed the labels for the valves and flow indicator for Bottom Drain 3 and the spare bottom drain to make it match the drawings.

*Installed discharge chamber chute liner.

*Attempted to get side chamber level probes to work. The alarm only comes on when the level probe is moved.

8/7 Double checked utility rack labels for Bottom Drain 3 and spare. Labels found to be correct. The piping was hand-over-handed for a verification.

Took additional resistance and voltage readings from melter test panel.

The circulation pump for Bottom Drain 3 cooling tripped the breaker. Temperature reached 400°F. Steam was emitted for less than two minutes, then cooling water was returned.

8/8 The bottom drain thermocouples were verified to be correctly identified at the melter control panel.

8/10 *East side chamber thermowell installed with five thermocouples.

West plenum thermocouple was found to be defective. Maintenance has replaced it with a spare.

8/13 Performed inspection of east side chamber thermocouple installation.

8/15 The discharge chamber seems to be leaking again.

8/17 *The east lid heaters, two most south elements failed.

8/18 *Installed the Inconel shoe in the discharge chamber. The shoe has drifted downward due to the heat used during the orifice glass burnout process. There are only two bolts holding the shoe in position (north end) and the entire orifice frame has dropped down on the south end.

*Found that the discharge chute shoe had dropped approximately 1/4" on the south end. Glass was noted to have filled the space on the east side of the discharge chute.

8/19 *Removed start-up thermocouple from east side chamber. All but approximately two feet of the thermowell was gone! Burnt off portion appears to be standing in the east side chamber northwest area leaning on the E-wall.

8/20 Installed glass stream flow pin below discharge orifice.

8/23 *Installed new insert in Bottom Drain 3, re-connected permanent utility rack water cooling lines.

8/26 *COMMENCED CAMPAIGN 2 (Acceptance Test Initiated)

*Center chamber top glass thermocouple is defective.

8/27 *The two most northern heaters on the east side read a direct short to ground.

8/28 Troubleshot lid heaters. Found two heaters out of service in east heater bank (See 8/17) and one out of service in west bank. Jumpered out the two bad heaters on the east side and 1 bad/1 good on the west to balance the electrical load.

Flashing and bubbling was noticed in west side chamber. Problem determined to be Bottom Drain 1. Installed temporary cooling system on Bottom Drain 1.

8/29 *Bubbling is still occurring in west side chamber. Leakage is significantly lower than observed with normal cooling system in service.

8/29 *Bulbous glass formations are building below discharge orifice. It appears that glass is coming out above the "dam" in northeast and southeast corners. After cleaning, a thin 1" long partially molten glass sliver remained in northeast corner of dam where it meets the orifice support plate.

8/31 Found cooling water temperature on Bottom Drain 1 to be warmer (134°F) than that of the previous Bottom Drain 3 water temperature.

9/1 *Installed new Bottom Drain 1 insert.

*Bottom Drain 1 temperature continued to increase with rotometer not exceeding 0.5 gallons per minute (gpm) with valve fully open. To better control the temperature and to obtain a better flow, the temporary cooling system was connected. With melter temperature at 1,250°C, Bottom Drain 1 temperature was 550°C.

*Bubbles continue to be observed in the west side chamber.

*Side chamber level indicators did not alarm. Electrodes were exposed.

9/2 Discharge chamber continues to fill with glass.

*Installed new sleeve in Bottom Drain 2.

The supply and return lines were swapped on Bottom Drain 3. All supply lines furnish flow through the cooling tube inside the sleeve rather than the return line being connected.

A low flow condition exists at the Bottom Drain 1 cooling line. Temperature is acceptable.

Discharge chamber orifice is open, however there is some glass coming out from the side of the discharge chute.

9/3 *Installed new sleeve in spare bottom drain.

*Side chamber level indicators are unreliable.

Discharge chamber orifice has 30% blockage.

9/4 *Melter temperature excursions experienced several times.

*INITIATED 36 HOUR MELTER ACCEPTANCE TEST

9/5 *Contractor representative reports that Bottom Drain 3 is reading high -- 651°F. Contractor representative recommends that this drain temperature be kept below 400°F due to the mastic used during sleeving the drain. Recommended increasing flow from 1 to 2 gpm and lowering the flow to some of the other components on the melter.

9/7 *COMPLETED 36 HOUR MELTER ACCEPTANCE TEST.

*Added 50 lbs of lithium carbonate and 25 lbs of center chamber frit to make main chamber glass more conductive. Side chamber temperatures were approximately equal to main chamber temperatures.

9/10 Glass buildup in box under discharge orifice.

*Experienced heavy foaming from the west side chamber. Foaming continued for two hours. Water was added in the interim to help reduce the foaming but with little effect.

9/14 Melter discharge orifice still contrary.

9/15 *Experienced foaming in the west side chamber for several hours preventing feed operations.

9/16 *Foaming continued from the west side chamber to the center chamber.

000084

9/18 *Foaming in the west side chamber required the addition of 520 pounds of frit to the west side chamber.

*Three times during the shift foaming in the west side chamber was extensive to the point of causing foam to flow over the west E-wall. SCR 1 power was reduced in each case to reduce the foaming and frit was added in an effort to change the glass chemistry to prevent foaming.

SCR 3 failed to restart after being turned off.

9/19 Need to reinstall thermocouples around the melter.

*Experienced foaming in the east side chamber.

*There is no side chamber temperature indication.

(Initiated effort to contract a melter maintenance vendor to perform specialized tasks, and provide project maintenance staff with training.)

9/20 Slurry feed halted due to melter level and sulfate content.

*Side and center chamber foaming.

New direction is to control melter operating level 1" to 3" below the E-wall reference seam.

9/21 *Had a great deal of foaming in the center chamber. Minor foaming was observed in west side chamber.

Found middle plug in top of discharge chamber to be partially dislodged and damaged. Plate with plug penetrations also appears to be loose.

9/22 *Ramped melter temperature to 1,175°C. Observing refractory temperature only 10°C less than indicated melter temperature and side and center chambers very bright. Melter temperature was 1,263°C, actual temperature (melt pool surface temperature measured with infra-red heat detector) was 1,343°C.

9/24 *Discovered a 685°F hot spot at the point the Bottom Drain 3 transitioned into the bottom of the melter. Determined that the heat was caused by joule heating. (Glow identified at Bottom Drain 3, however unable to pin-point source as it is above the bottom drain assembly flange which is attached to the outer shell. Glow registers 685°F on the infra-red camera.)

Lid Heater 4 opened up during ramp-up to help maintain heat in the melter while SCR 1 is off.

000085

9/25 Experienced one auto discharge.

Implemented new electrical monitoring plan.

9/25 *Found an additional crack on west E-wall. This is a large crack which runs from the top of the wall down to the glass.

*Bottom Drain 3 glow re-appeared.

*Due to the glow on Bottom Drain 3, the drum cooling system was connected. Bottom Drain 3 is now isolated from the rest of the melter cooling system.

The failed heaters on SCR 3 and 4 were jumpered and they were powered up to aid in the heating of the melter while SCR 1 was off. During the ramp-up SCR 3 failed.

*Bottom Drain 3 cooling system was configured to a feed and bleed arrangement. Dry ice (HCO_3) was added to the cooling drum to lower the supply water temperature.

*After re-powering SCR 1, the glow of Bottom Drain 3 was re-established.

9/26 *Reduced the temperature of the discharge chamber (SCR 2) to monitor the effect on the "glow." At discharge chamber temperature of 1,170°C, the intensity of the glow had reduced.

*Bottom Drain 3 glow varied in intensity during the day. Seemed to depend on the power of SCR 1 and the discharge temperature.

9/27 *Bottom Drain 3 glow returned at 1030 hr. It faded away at 1300.

9/27 *Bottom Drain 3 glowed at 2345 . It was dim compared to yesterday's glow. The glow went away by 0015 hr. but reappeared at 0430 and became brighter with time.

9/28 *Ramped down SCR 1 to 0 KW and restored to its previous set point to evaluate SCR 1 impact on the Bottom Drain 3 glow. The glow subsided when power was ramped down and has not returned as of turnover.

*Bottom Drain 3 glow reappeared at approximately 0400.

(Since the melter operates on an ungrounded system, it takes two points to make a circuit. An attempt was made between September 26 and September 29 to locate another electrical contact point.)

9/30 *Restored Bottom Drain 3 cooling water back to temporary cooling water pump.

Bottom Drain 3 temporary cooling water pump tripped. Process water being utilized for cooling water.

Tested the utility rack's ability to provide adequate flow to the bottom drain. The flow rates are limited by the pressure drops through the bottom drain plungers and supply/return tubing.

10/1 Discharge chamber orifice is plugged.

10/2 Took amp, reading on Bottom Drain 3 below and above the isolation coupling.

The west thermocouple is grounded.

*Experienced a dim glow all shift at Bottom Drain 3.

Installed wire to west side chamber thermowell to terminal bar on the west side of the melter room.

10/3 *Experienced a dim glow (though somewhat brighter than yesterday) all shift at Bottom Drain 3.

10/4 *Removed west thermocouple. It was a supertube with a thermocouple in it. It would not pull out, so we drilled into the glass/brick/thermocouple about 3". We then put the castable material into the hole. The south side of the melter was cooled with water for about 15 minutes. The melter was then started back up and the voltage readings taken. The resistance readings came up slightly.

*Experienced a brighter glow (brighter than yesterday) at Bottom Drain 3 as we ramped up in temperature in preparation for the OWI to cap the east thermowell.

Continued to take infrared temperature measurements of the Bottom Drain 3 area.

Discharge chamber plugged.

10/5 *Removed east thermowell. The glass behind it was frozen and chipped back then castable material was placed into the hole.

A lot of glass migrating from the south towards the discharge hole. (Below the discharge trough brick flowing around and out south side of discharge orifice.)

*Experienced a brighter glow (brighter than yesterday) at Bottom Drain 3 as we ramped up in temperature in preparation for feeding the melter.

*Experienced one foaming event. Foam got within 2" of the top of the west E-wall.

10/6 *Bottom Drain 3 glow has been bright all day. Close observation has determined that the glow is in the area of the material between the inner and outer shell.

*Temperature of Bottom Drain 3 increased to 550°F. Water was set to backflow through the bottom drain. The temperature dropped to below 350°F. Later in the day, the water flow for Bottom Drain 3 had stopped; found the pump had lost its prime. The flow was left in the reverse direction. Flow was measured at 2,000 milliliters in 1 minute and 11 seconds.

Re-evaluated the process for measuring the resistance to ground for east and west buss. The old data is not valid.

10/7 *Foaming in west side chamber.

10/7 SCR 4 is at zero amps. SCR 4 is malfunctioning.

Discharge orifice is plugged.

10/8 Water flow through Bottom Drain 3 is being provided by the process water connection on the pad.

Meeting was held with contract representative to review all known information relating to the Bottom Drain 3 glow.

*Bottom Drain 3 temperature increased slightly to 389°F.

Disconnected Bottom Drain 3 from temporary drum cooling system and reconnected it to utility rack.

10/10 *Dropped discharge chamber temperature to 600°C.

Configured Bottom Drain 1 to the separate cooling recirc system on the east wall of the melter room.

Cooled the south wall of the melter with tap water (Chlorine introduced) in an effort to cool the identified hot points.

The supply and return lines to Bottom Drains 1, 3, and spare leaked at the electrical isolation fitting. Bottom Drain 1 reached over 700°F after the leak was fixed.

Reconfigured Bottom Drain 1 cooling so that the water supply is going into the supply tube and the drain is coming out of the drain tube.

*Cooled the south wall of the melter with water. Took a baseline with the thermal imager and electrical readings. Cooled the west side down to approximately 85°F and then took another set of readings. Repeated process with the east side. The electrical readings showed no change. The wall was reheated back to approximately 350°F within one half hour and continued to climb back to initial readings. (Follow-up trouble-shooting attempt from September 26 initiative.)

10/14 *Lowered power from 214 KW to 120 KW, side chambers getting bright. Let temperature coast down to 1,200°C.

10/16 Added frit to side chambers to control foaming.

*Glow on Bottom Drain 3 reappeared for approximately 30 minutes.

10/17 *Noticed material (sulphur?) at the discharge orifice. Sample was taken and sent to the lab for analysis.

10/19 *Glass suddenly appeared brighter in main and side chambers.

*Glow reappeared on Bottom Drain 3.

10/20 *Surface of main and side chambers is still bright.

10/21 *Brightness is back to normal for this indicated temperature (1,250°C).

10/25 Air lines under the melter were satisfactorily tested for leaks.

*Slight glow returned at Bottom Drain 3.

10/27 *Started bottom drain sleeving. Procedure was discontinued as the existing insert on Bottom Drain 1 could not be removed.

10/30 *Attempted to remove Bottom Drain 3 cooling sleeve. Could not get it to break free. Attempt was abandoned. Current path forward is to leave bottom drains as is until after Campaign 4.

000089

Placed Bottom Drain 1 back on the utility rack cooling water.

10/31 SCR's 1 and 2 tripped. Reset breakers and continued operation. Cause of trip not yet determined.

11/2 During glycol addition, melter cooling water back-up system (treated water) was initiated due to low pressure in the system. There was flow at the rotometers, at a reduced rate. A random selection of temperatures of the components being cooled by the system was monitored and an appreciable increase of temperature was not indicated. The system was reset and placed back on the closed loop system.

11/3 Bottom Drain 1 temperature trended up unsatisfactorily. Placed Bottom Drain 1 on the temporary cooling water system.

*Performed thermal image survey of bottom drains. Bottom Drain 1 was indicating much higher by thermocouple, image results for the bottom drains were all reading about the same.

11/19 Tried to swap Bottom Drain 1 back to utility rack cooling. Temperature elevated and could not be restored. Went back to temporary cooling.

11/21 *Adjusted side chamber level probes to compensate for consumption of probes.

11/24 Bottom Drain 1 and 2 cooling water fitting under the melter is leaking a few drips a minute. Found that the temperature at the pipe nipples may exceed its rating.

Cleaned utility rack flow indicator for Bottom Drain 1.

Replaced pipe nipple on Bottom Drain 2. During the switch out, the steam coming from the cooling sleeve started to melt the new nipple. Re-applied the cooling water to Bottom Drain 2 and found the leak still existed. Replaced the new (melted) nipple with a used Teflon™ nipple.

11/25 Cleaned all utility rack rotometers.

Attempted to place Bottom Drain 1 back on the utility rack cooling. Found that the existing tubing fittings were not compatible with the tubing jumper that needed to be installed. Bottom Drain 1 was allowed to stay on the treated water cooling system (hose).

11/25 Varied the flow (on and off) of water to Bottom Drain 2 while it was on utility rack cooling and determined that even though there is no "indicated" flow, there is a definite cooling effect when the flow is re-established.

000090

Bottom Drain 2 temperature increased, swapped to temporary cooling. Tried to locate the missing part from the Bottom Drain 2 flow indicator. Back flushed Bottom Drain 2 water lines to try to locate part.

Reliability of thermocouple for Bottom Drain 2 temperature to the utility rack had erratic readings. Replaced the thermocouple with a new Type K thermocouple supplied by maintenance. Reading in the normal range (approx. 335°F). Previous thermocouple was abandoned in place.

Cleared the Temporary Alteration that was on the compressed air cooling for Bottom Drain 3 (for the "Glow" problem). The tubing was removed.

11/26 Decided to place Bottom Drain 1 back on the utility rack cooling. Prior to placing on the utility rack cooling, noticed that there were several leaks on the tubing connections and at the Teflon™ insulator pipe for the supply and return. The bottom drain insert was wet below the point where it passes into the outer sleeve (above the 1/4" tubing penetrations). The sleeve is leaking up in the bottom drain and the water is leaking down the insert.

A decision was made not to place Bottom Drain 1 back on the utility rack due to the fitting leaks (no replacement parts were on hand tonight) and did not want to risk steaming out the bottom drain insert again.

11/28 Removed cage and looked at Bottom Drain 1. Could not see any signs of water or leak. There was some white residue on the insert and the rest of the equipment but no signs of any water.

11/29 *COMMENCED CAMPAIGN 4.

11/30 Added 100 lbs. of Barium frit, 75 lbs. to the east and 25 lbs. to the west side. (It was visaully observed that glass from the side chambers was flowing to the center chamber through cracks in the E-wall. To counteract this flow, barium frit was introduced to the center chamber, which had a higher density of 2.8 grams per cubic meter. It was hoped that the higher denisity center chamber frit would reduce the flow of the lower density side chamber frit.)

12/1 Powered SCR 1 down to replace electrical isolation at the east side chamber glass level indicators.

Found some sulfate in the discharge chamber orifice.

12/3 *Experienced foaming in the west, east, and center chambers.

12/7 *West and center chamber foaming. Had trouble maintaining temperature in center chamber.

000091

12/8 *West side and center chamber still foaming. Had trouble maintaining temperature in center chamber.

12/9 *West chamber foaming control is continuing to be a problem.

Plugged the discharge chamber during an auto discharge.

12/10 *Foaming over the west E-wall was a problem all night. The structural integrity continues to show increasing deterioration of the E-walls.

*Added 50 lbs of lithium carbonate and 25 lbs of frit to the center chamber.

Plugged the discharge chamber four times.

12/11 *Pulled air lance to see if the hole that was reported to be in the south melter wall could be observed.

Cleared residual glass from the discharge orifice. Sampled glass and film on the gem window.

Experienced what seemed to be air in the surface drain rotometer on the utility rack. The temperature on Bottom Drain 2 cooling had increased 300°C since the last round sheet log. Before the pipefitter started work, steam and water breached thru the bottom drain insert.

Established a temporary cooling water supply to Bottom Drain 2. Bottom Drains 1 and 2 are isolated from the utility rack and are being cooled by an open loop system using process water.

12/12 *Minor west side chamber foaming. Occasional difficulty with melter temperature increases when KW increased.

12/14 SCR 2 dropped to zero. Replaced discharge chamber heaters.

12/15 Castable insulation from top of discharge chamber fell and obstructed discharge chamber camera view. De-energized discharge chamber heaters and removed castable insulation.

12/16 *Bottom Drain 3 went up to 1,760°F. The return tubing was removed, no flow was evident. Hooked up a treated water hose to return line and allowed water to flow out supply tubing.

*Bottom Drain 3, there was a small amount of smoke coming from the thermocouple.

*There was molten metal on top of the coupling of the bottom drain.

12/17 *Performed first half of melter lid heater removal. East lid heaters were removed and an inspection of the melter lid refractory was completed. Due to a crack across the middle of the center lid refractory block, a determination was made that work would stop until a meeting was held to determine the path forward.

*West lid heaters were removed and an inspection of the melter lid refractory was completed. Insulation board and blanket were installed in the opening and the cover plate was installed. The refractory plugs were not installed at this time.

12/18 Repaired the flexible conduit related to the west side glass chamber level probe.

12/19 *Bottom Drain 3 "metal" from 12/16 was 58% lead.

*Bottom Drain 2 broke loose with a BANG! and is bleeding off steam.

12/20 *Bottom Drain 3 heated up to 1,579°F. Attempted to blow out with air and found that the center tube was plugged with black "tar-like" substance. Tried to blow out with 80 psi air forward and reverse and could not clear. During this effort the temperature dropped to 1559°F.

Bottom Drain 1 lost water flow. Bottom Drain 2 was heating up. Bottom Drains 1 and 2 are isolated from the utility rack and are being cooled by an open loop system using process water.

*Implemented a temporary alteration for Bottom Drain 3 to have an air supplied cooling media until Maintenance provides a permanent fix. Drilled a hole in the bottom drain insert to accept a 1/4" copper tube. This lowered the temperature from 1,660°F to 1,435°F. Bottom Drain 3 being cooled from the air supplied to the bubbler tubes.

12/21 Installed east side and west side melter ceiling refractory strong backs.

*No cooling water flow through Bottom Drains 2 and 3.

*Repairing Bottom Drain 3 cooling water. Installed 1/2" tubing approximately 6-7 inches up into the insert. This is being used as the supply. Also installed 1/2" tubing on the shell of the insert. The two 1/4" tubes were soldered shut.

*There is a blue flame that appeared in the east chamber after Bottom Drain 3 was repaired.

Electrode H return water keeps going in and out of alarm. (Designation H is the bottom east electrode.)

12/22 Installed Temp Alt 85 to supply Bottom Drain 3 with treated water to eliminate glycol from being introduced into the melter.

Completed Bottom Drain 2 repair by installing new cooling water drain connection.

*Bottom Drains 2 and 3 were leaking into the melter enough to cause excessive agitation in respective melter chambers. After establishing effluent flow path for Bottom Drain 2 and installing TA-85 on Bottom Drain 3 excessive agitation subsided. (Cleared cooling water plug in Bottom Drain 2, and placed Bottom Drain 3 on alternate temperature cooling system.)

12/22 *Foaming was constant on east side.

12/23 *Installed temporary alternative to supply spare bottom drain with treated water to eliminate glycol from being introduced into the melter.

Repaired leak on Bottom Drain 2.

*The ends of the top two west side chamber electrodes are gone.

12/24 *Feed tube cooler leaking glycol into the melter. Placed feed tube on temporary water system.

12/25 Applied high temporary RTV to the melter lid plates to lower in leakage.

12/26 Placed plastic water cover over the bottom drain containers.

*Event.

IV. ROOT CAUSE ANALYSIS

The following sections provide a Failure Modes Analysis (FMA) to determine the root cause(s) for the loss of the glass melt contents from the Duratek 1000-HT Melter at 2222 hours, December 26, 1996. This section highlights anomolies associated with the breach of the melter in accordance with the Event and Causal Factor Chart for Melter Failure (Appendix E).

Summary

The draining of the glass melt through the bottom of the VITPP Duratek 1000-HT three-chambered, high-temperature melter was the result of corrosion of the number 3 bubbler tube assembly in association with severe exudation and corrosion of the bonded (Zirmul) alumina-zirconia-silica (AZS) sub-layered refractory beneath the Monofrax K-3 hot-face surface block. The corrosion phenomena were exacerbated with the introduction of lead oxide into the glass melt during Silos 1, 2, and 3 surrogate waste test campaigns (Phase I, Campaign 2 and Campaign 4), thus significantly reducing the life expectancy of the melter. The attack on the materials of construction (both the bubbler tube assembly and the underlying refractory materials) were through the following mechanisms:

- Corrosion of the molybdenum disilicide bubbler tube in an oxidized and/or lead-bearing surrogate glass melt. Corrosion resulted in the bubbler tubes receding into the less corrosion resistant underlying refractories. The chemical measurement for the erosion are:

  $2MoSi_2 + 7O_2 \rightarrow 2MoO_3\uparrow + 4SiO_2$ (Equation 1)
  $MoSi_2 + 7PbO \rightarrow MoO_3\uparrow + 2SiO_2 + 7Pb\downarrow$ (Equation 2)

- Downward migration of the glass melt through the bottom penetrations (bubbler tube and bottom drain assemblies) and refractory joints, initiated erosion/corrosion of the AZS refractory.

- Reduction of lead oxide from the glass melt produced lead metal. The molten lead may have accelerated the erosion/corrosion of the underlying refractories by a downward drilling action. Probable chemical reactions for the production of the lead metal are:

  - The molybdenum disilicide tubes chemically reacting with lead in the glass:

    $MoSi_2 + 7PbO \rightarrow MoO_3\uparrow + 2SiO_2 + 7Pb\downarrow$ (Equation 2 above)
    $1g + 10.27g \rightarrow 0.95g + 0.79g + 9.53g$

    According to equation 2, 1 gram of molybdenum disilicide produces 9.53 grams of molten lead metal. The silicone dioxide produced is

absorbed into and becomes part of the glass. The molybdenum trioxide is a gas at molten glass temperatures. Therefore, some molybdenum trioxide may leave the glass melt before being absorbed by the molten glass. Overnight crucible melts done at Fernald show that when molybdenum disilicide rod is placed in lead-bearing glass, a molten lead puddle forms in the bottom of the crucible.

- The molybdenum metal electrodes chemically reacting with lead-bearing center chamber glass that has leaked into the electrode side chambers:

$$Mo + 3PbO \rightarrow MoO_3\uparrow + 3Pb\downarrow \qquad \text{(Equation 3)}$$
$$1g + 6.98g \rightarrow 1.50g + 6.48g$$

According to Equation 3, 1 gram of molybdenum metal will produce 6.48 grams of molten lead metal. Molybdenum trioxide is a gas at molten glass temperatures. Therefore, some molybdenum trioxide may leave the glass melt before being absorbed by the molten glass. Overnight crucible melts done at Fernald show that molybdenum metal is placed in lead-bearing glass, a molten lead puddle forms in the bottom of a crucible.

- "Reboil" of surrogate glass passing through the E-brick walls can precipitate free lead. At 1,250°C the conductivity of E-brick is about 0.075 siemens per centimeter (S/cm). Some surrogate glasses, due to the inleakage of side chamber frit, approached conductivities in the range of 0.25 to 0.30 S/cm. The higher conductivity of the glass channels more current, with resulting heating, in the cracks in the E-brick walls. Some operators commented that they saw the cracks near the glass melt surface glowing. The extra heat causes a reductive environment which was evident by the "boiling" seen at the E-brick walls while current was applied. The "boiling" quickly disappeared when the current was turned off or greatly reduced. Inspectors noted that they saw what looked like solidified lead-metal beads that formed from sweating from the pores of the E-brick wall cracks.

Reduction studies show that simple reduction of the surrogate glass (e.g., through the use of urea) will produce lead sulfide because of the presence of dissolved sulfur (sulfate) in the glass. It takes a high (localized) reduction potential (such as molybdenum metal, molybdenum disilicide or "reboil") to produce molten metal. No lead sulfide precipitation was identified during the melter inspection, only lead metal was observed. Glass samples from the lead-bearing campaigns where analyzed and showed only oxidized conditions with $Fe^{+2}/(Fe^{+2}+Fe^{+3})$ ratios ≤ 0.33 or $\leq 33\%$ reduced. Precipitated lead metal or lead sulfide is not known to happen under these redox conditions.

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- Turbulent air abrasion, oxidation, and molten glass circulation via compressed air flowing from corroded bubbler tubes receding below the corrosion resistant K-3 refractory floor.

- Disruptive electrical current paths as a result of glass migration and metal precipitation at the inner and outer shell. Localized current creates hot spots with resulting increased corrosion.

- Upward drilling on the refractories by the glass melt through vapor phase formation.

- Lack of corrosion resistance of KAO-TAB95 castable insulating refractory to molten glass.

Another mechanism was suggested by the investigating group that could have led to the erosion of the bubbler tubes. This was the possibility that molten lead could alloy with molybdenum disilicide forming a molybdenum lead silicon alloy at glass melting temperatures. However, this does not appear plausible based on a recent crucible melt that was performed in the Fernald lab. Molten lead was left in contact with a molybdenum disilicide rod. Glass was poured over the lead and rod to protect them from the air and the crucible left overnight in a furnace at glass forming temperatures. The rod was pulled out of the molten lead at the end of the test. The molten lead did not even "wet" the molybdenum disilicide rod which indicates there appears to be no tendency to form the speculated alloy.

These phenomena are generally known in the glass industry. They are considered the primary conditions affecting the service life of the refractory lining and the length of melter life (References 1 - 11, 13 and 14). There is general consensus that the corrosion rate(s) on materials of construction are unique to the corrosive properties of the waste glass melt, the corrosion resistance of the refractory lining, and the reduction rate of molten metals. Based on the uncertainty of these rates, the breach of a melter cannot be predicted with any degree of certainty.

Barrier Analysis

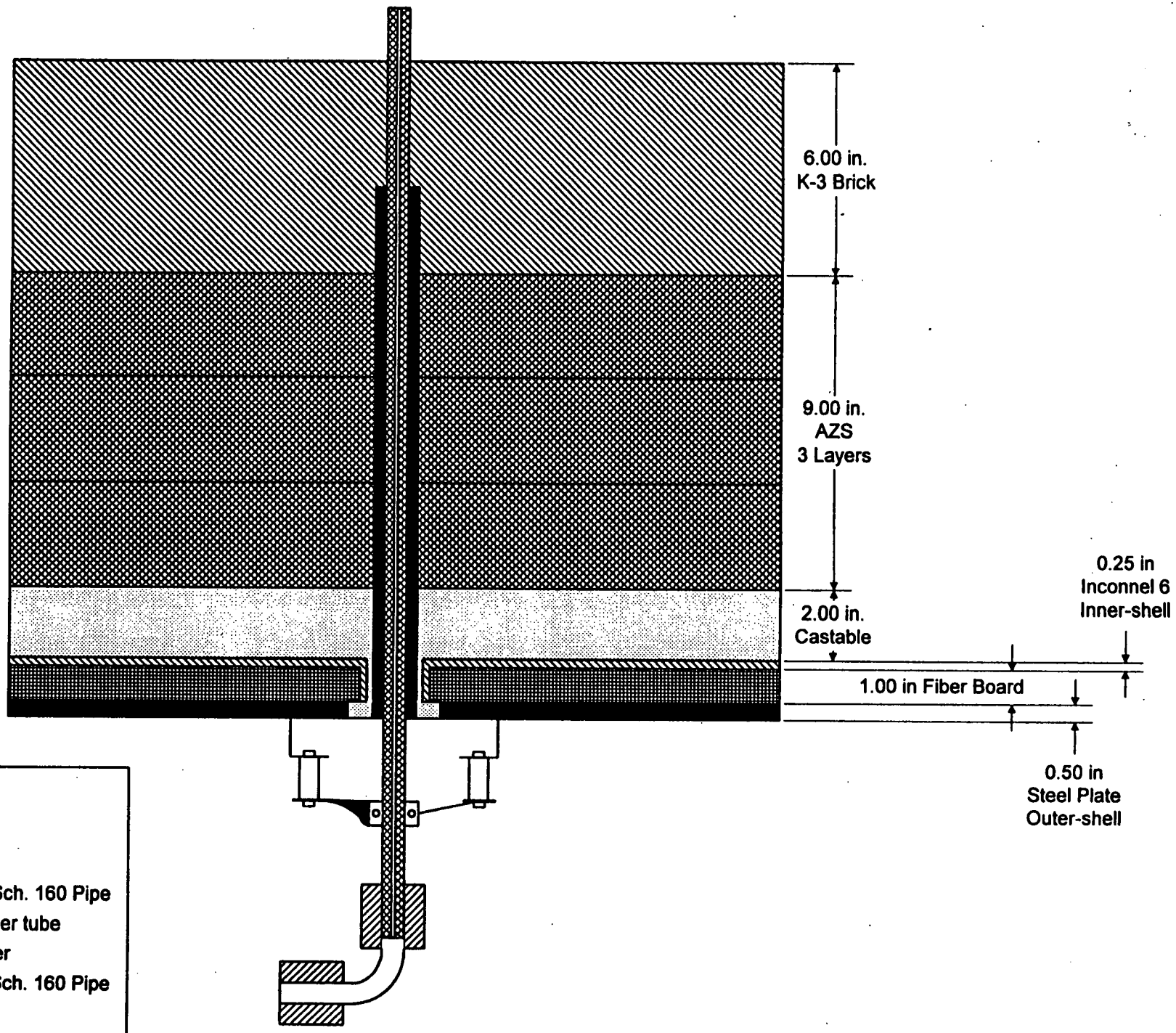
Figure IV-1 shows the configuration of the bubbler tube assembly and the engineered barriers required to be breached before the glass melt contents can be expelled from the bottom of the melter:

- ***Refractory Lining*** (Appendix F)

  - Monofrax K-3 refractory - one 6" block (Photograph IV-1)
  - AZS refractory - 9" (3 blocks each 3") (Photograph IV-2)
  - KAO-TAB95 (AL_2O_3) castable refractory - 2" (poured refractory) (Photograph IV-3)

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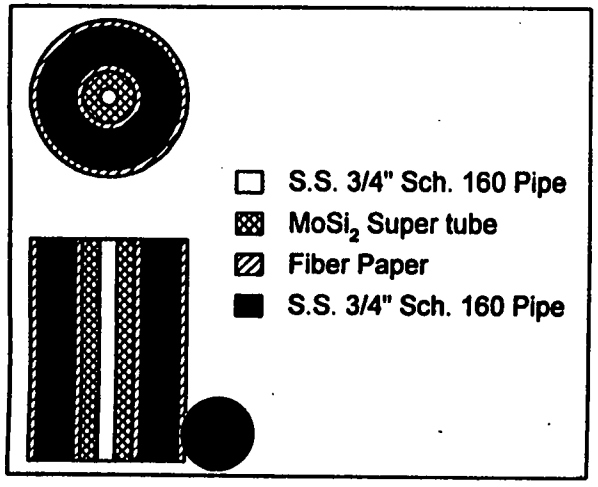
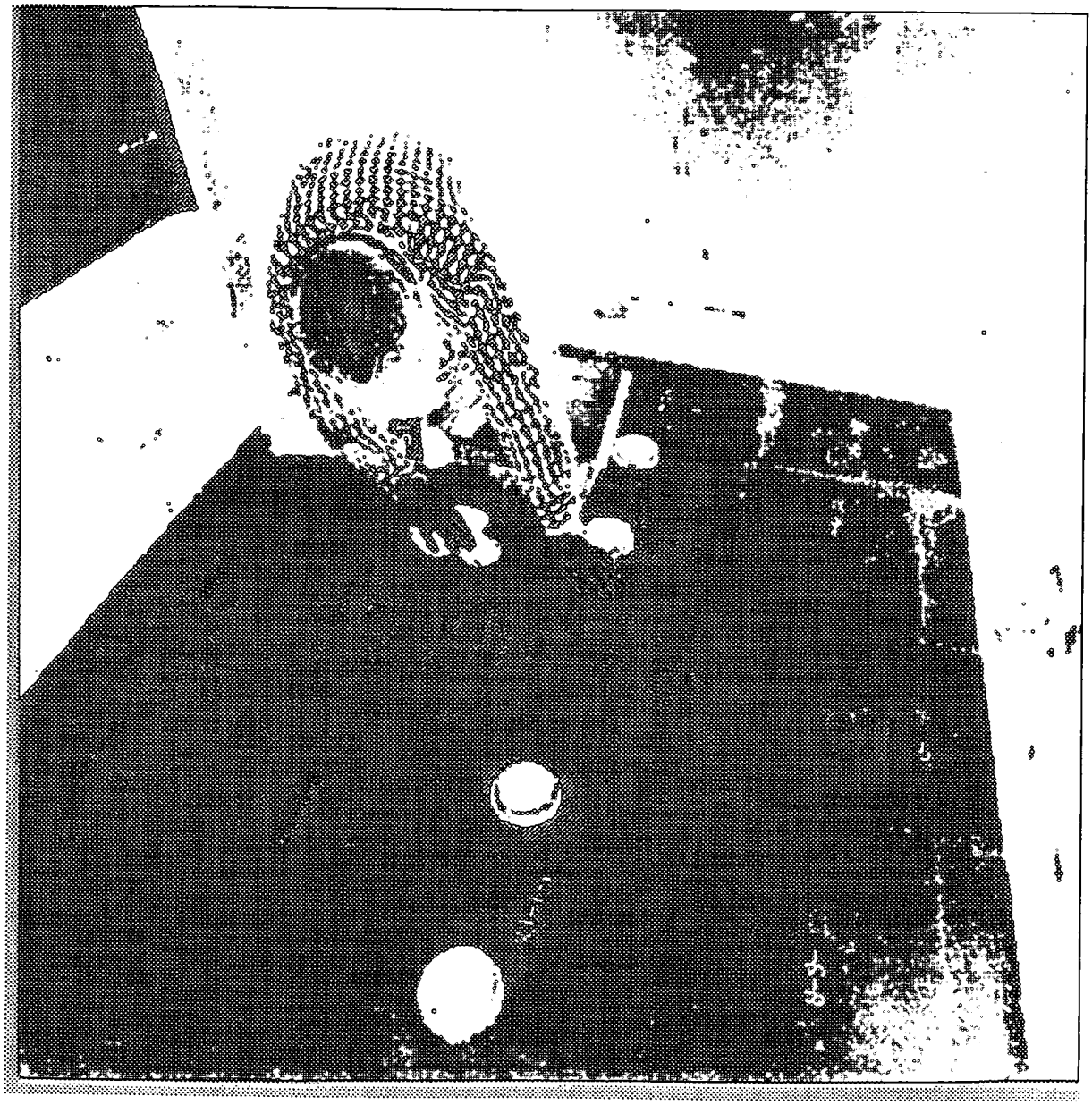


Figure IV-1 Bubbler #3 As Installed

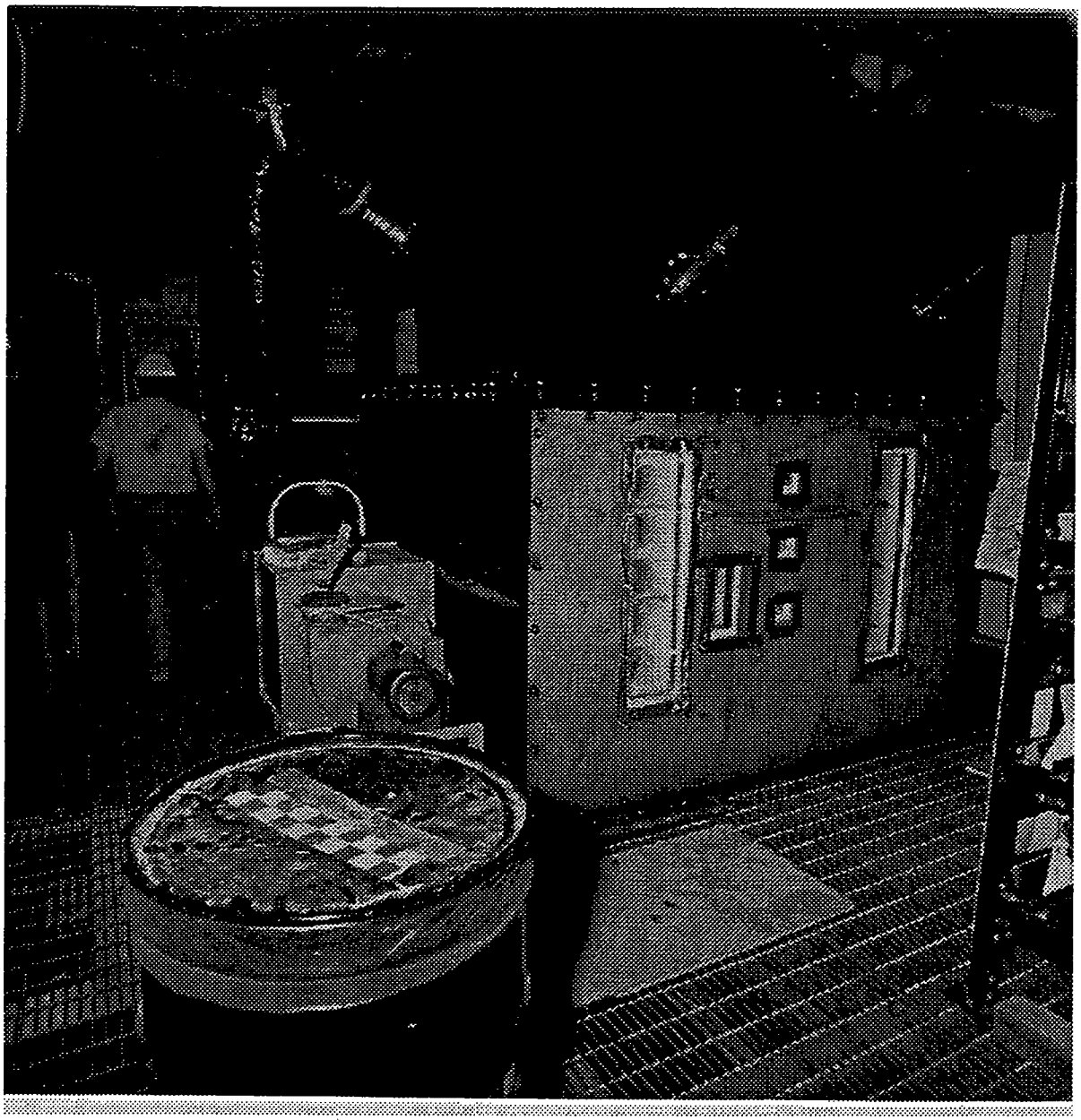
PHOTOGRAPH IV-1



PHOTOGRAPH IV-2



PHOTOGRAPH IV-3



- KAO-TAB95 castable plug - 1-3/4" (electrically isolates the bubbler assembly, the Inconel 690 1/4" inner shell and the ½" steel outer shell from each other)

- ***Bubbler Assembly***

  - 3/4" O.D. schedule 160 stainless steel pipe - 15-1/4" long
  - 1/2" O.D. Molybdenum disilicide bubbler - 20-1/4" long

- ***Inner Shell/Insulating Fiberboard/Outer Shell***

  - There is no inner shell, fiberboard, or outer shell in the area of the bubbler tube assembly. (Photograph IV-4)

The molybdenum disilicide bubbler tube originally extended 1-½" above the Monofrax K-3 hot-face refractory. This tube began to corrode and wear back upon introduction of glass melt into the main chamber on June 6, 1996 (Photograph IV-5). This is postulated since it is known that the molybdenum disilicide bubbler tube will corrode over time. Other melter components, such as level indicators fabricated from the same material, were observed to be experiencing some corrosion during Campaign 1 (June 28, 1996). The glass melt began to migrate downward around the bubbler filling in the void space between the bubbler and Monofrax K-3 refractory (hole through Monofrax K-3 is 5/8" I.D., counter bored 1-1/4" I.D., up 2-1/2" from bottom and bubbler O.D. is 15/32"). The rate of corrosion of the bubbler tube cannot be determined.

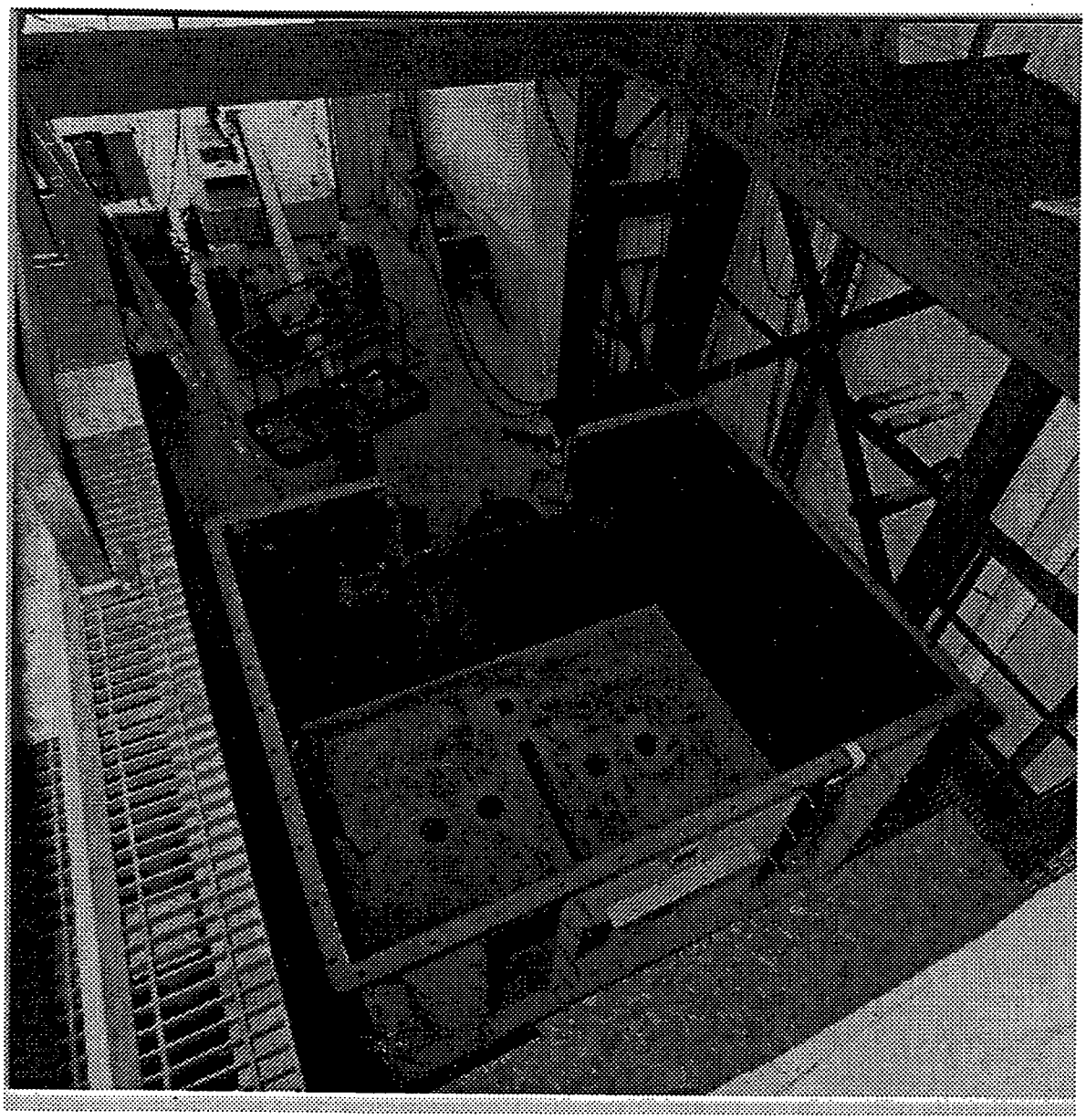
However, at some point, the bubbler corroded back to the stainless steel sheath surrounding the bubbler tube which was recessed 2-1/2" into the bottom of the Monofrax K-3 melter floor (Figure IV-2). This allowed the high-temperature glass melt to come into contact with the stainless steel sheath. At some point, which is not able to be determined, the molybdenum disilicide bubbler tube and the stainless steel sheath corroded back out of the Monofrax K-3 block (6" thick) into the underlying AZS brick (Figure IV-3). Corrosion increased after introduction of oxidized and lead bearing glass in Campaign 2 (August 26, 1996). It is documented that in an oxidized glass melt molybdenum disilicide will experience a significantly enhanced corrosion rate.

Once the bubbler tube assembly corroded below the Monofrax K-3 surface into the underlying bonded AZS refractory, several phenomena were already occurring:

- Upward drilling of the Monofrax K-3 surface blocks generally occurred. Upper drilling occurs when gas bubbles escape from the melt or the refractory material and attach to the underside of the refractory material. The bubbles cause an upward drilling effect on the refractory material. The severity of the attack depends on the solubility of the refractory material, the surface tension of the glass melt and the dissolved refractory material, and the density and viscosity of the glass melt.

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PHOTOGRAPH IV-4



PHOTOGRAPH IV-5



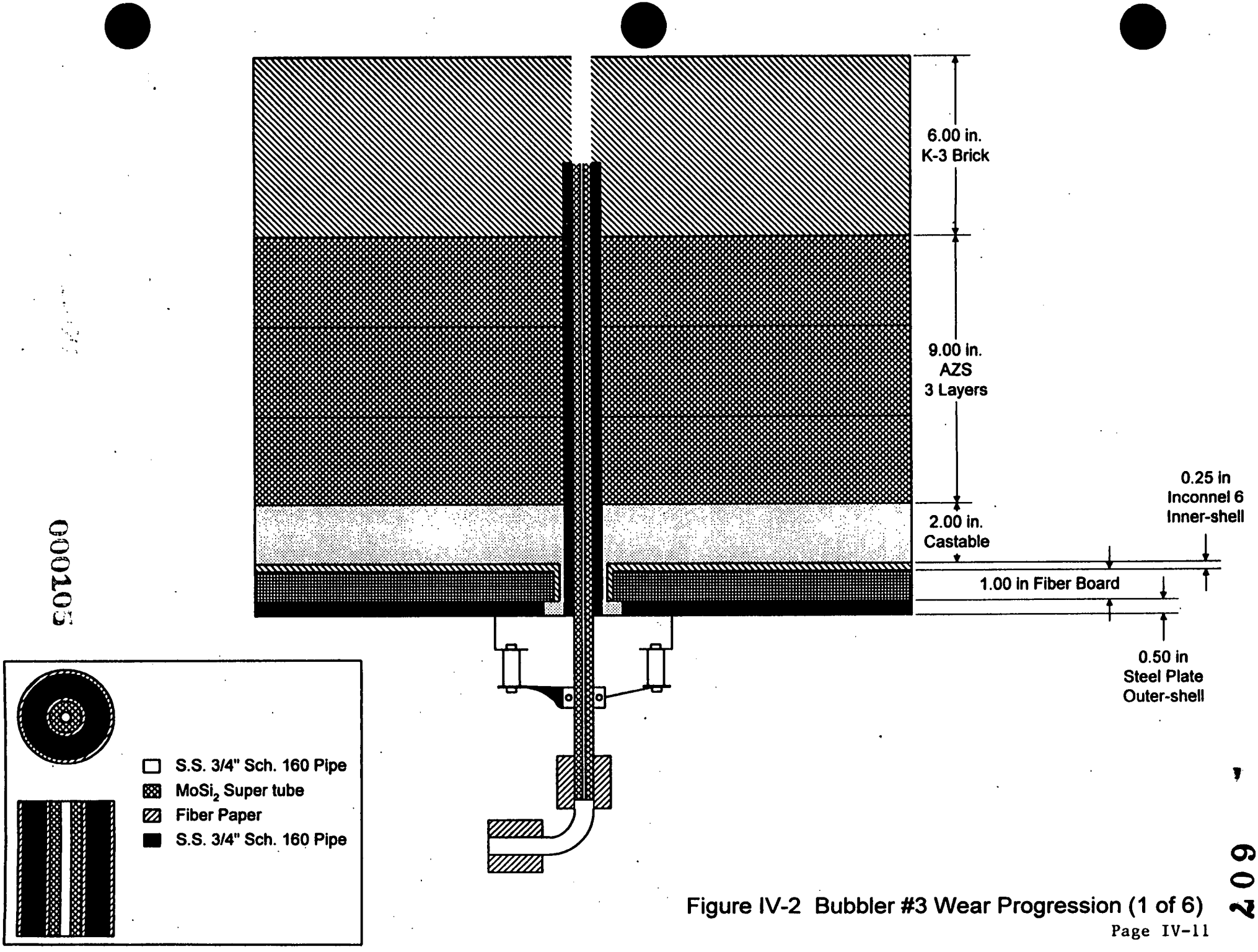


Figure IV-2 Bubbler #3 Wear Progression (1 of 6)

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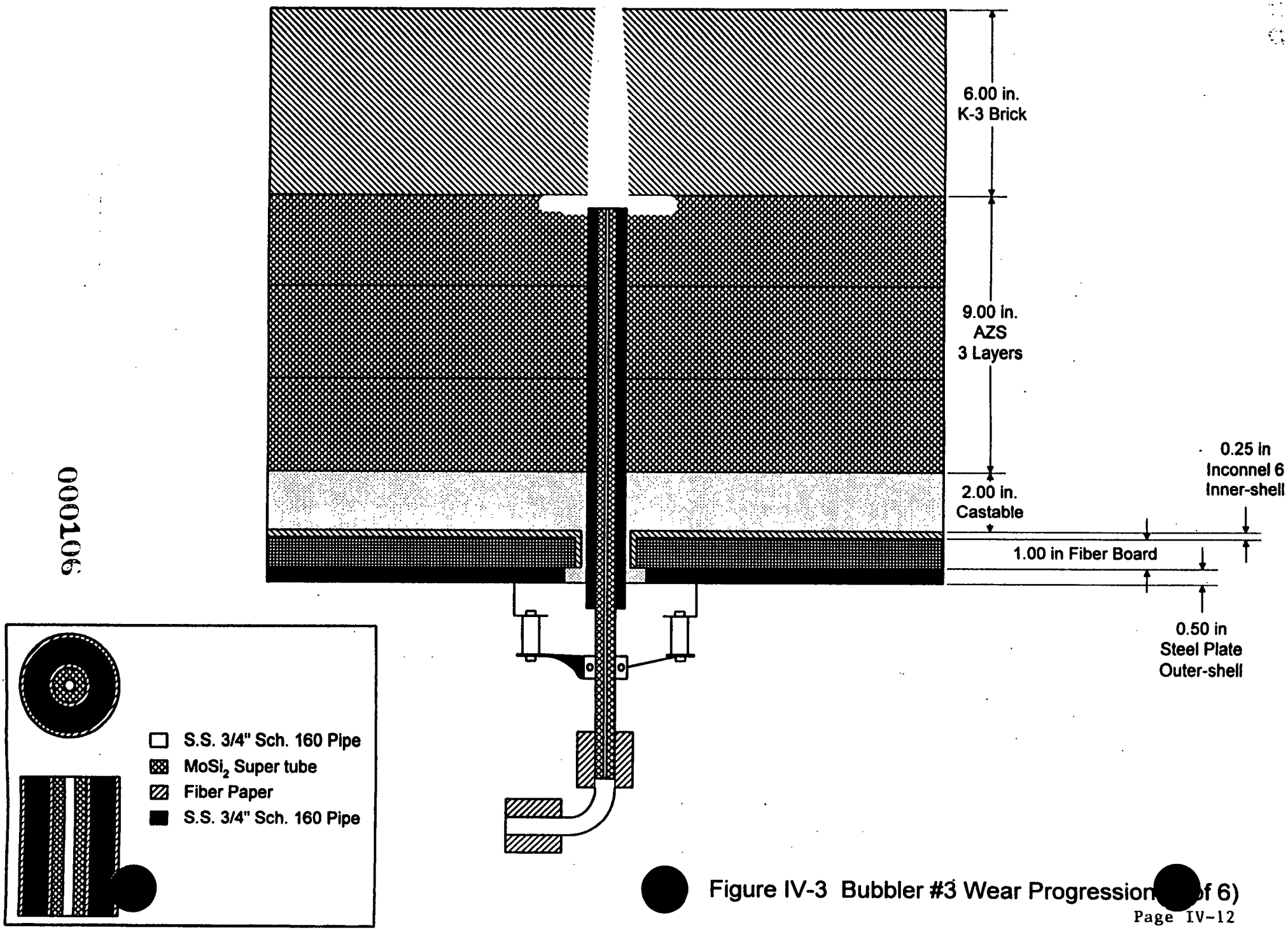


Figure IV-3 Bubbler #3 Wear Progression (1 of 6)

- The bubbler holes through the Monofrax K-3 through which the molybdenum disilicide bubbler tube are extended were widened from their original 5/8" diameter to approximately 1-1/4" (the diameter of the counter bored K-3 brick) (Photograph IV-6). In addition, the joint between the two Monofrax K-3 blocks was visibly widened. This process was exacerbated by the turbulent flow of the glass melt in these areas, resulting from the compressed air discharged through the molybdenum disilicide bubbler tube.

- Significant exudation and corrosion of the bonded AZS refractory occurred (Photographs IV-7 - IV-10). The bonded refractory is not very resistant to corrosion due to its high degree of porosity.

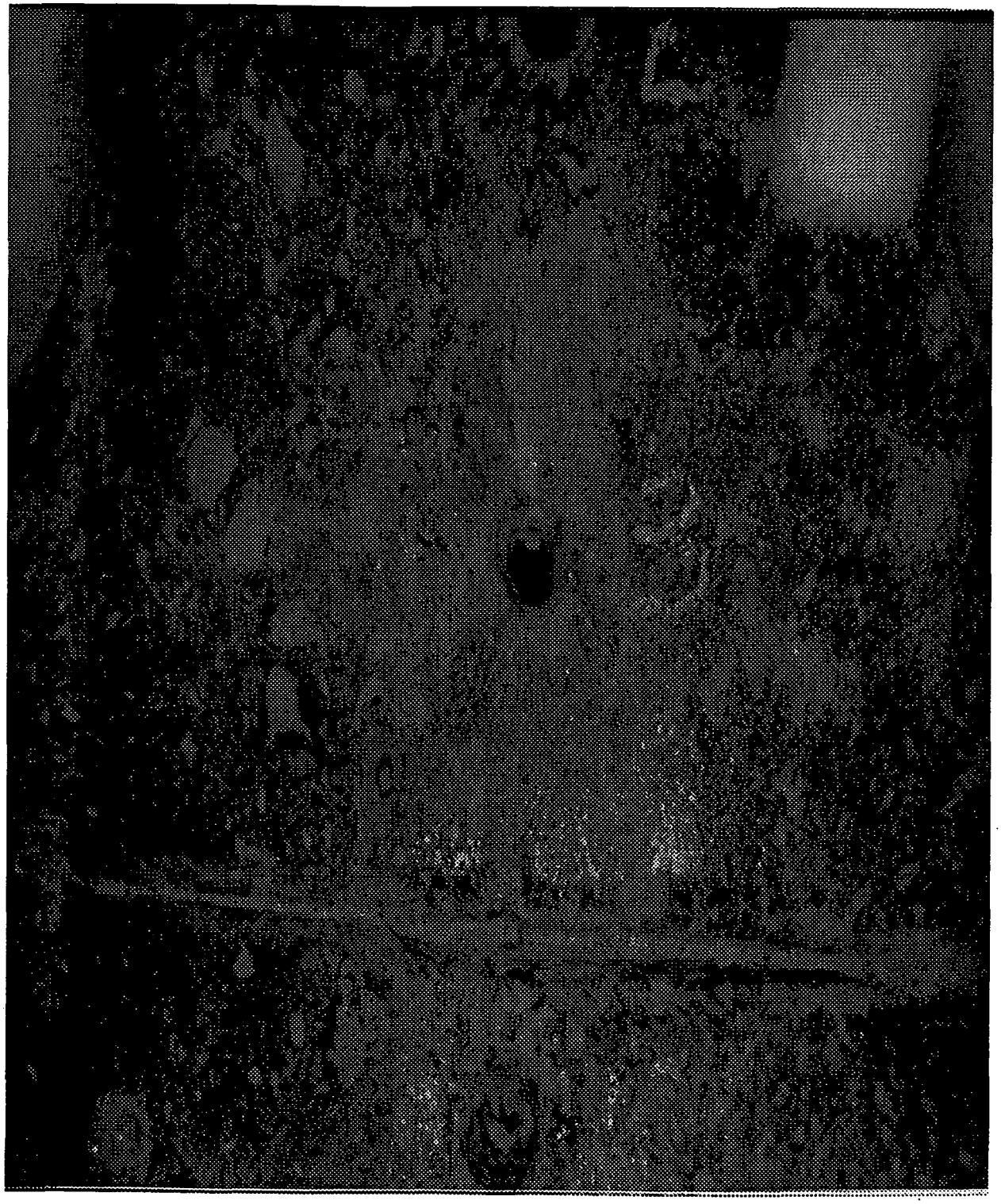
- Once the bubbler tube assemblies had deteriorated below the Monofrax K-3 surface block, the hot, turbulent conditions associated with the glass melt in these areas caused a rapid deterioration of the AZS refractory lining. The air cooling in the areas most likely retarded the corrosion process, but only moderately. Due to the high porosity (18%), the bonded AZS is vulnerable to rapid corrosion unlike the Monofrax K-3 fused cast refractory, which is very highly corrosion resistant. It is known that AZS are subject to attack by direct contact with components of the glass melt or through vapor phase transport of volatile species. Bonded AZS refractories are especially susceptible to exacerbated corrosion attack due to liquefied phase formation (exudation).

  The boroscope and core-drilling of the bubbler assembly areas revealed extensive cavitation and channeling of the underlying AZS brick (Figures IV-4, 5 and 6). It was obvious through viewing the hot face of the Monofrax K-3 surface paving (Photo IV-11) that the glass melt contents of the melter drained from the melter chamber through five (5) primary areas:

  - Bubbler 1
  - Bubbler 2
  - Bubbler 3
  - Bubbler 4
  - Bottom Drain 2,

  then exited from the bottom of the melter at the location of the Bubbler Assembly 3 (Figure IV-7).

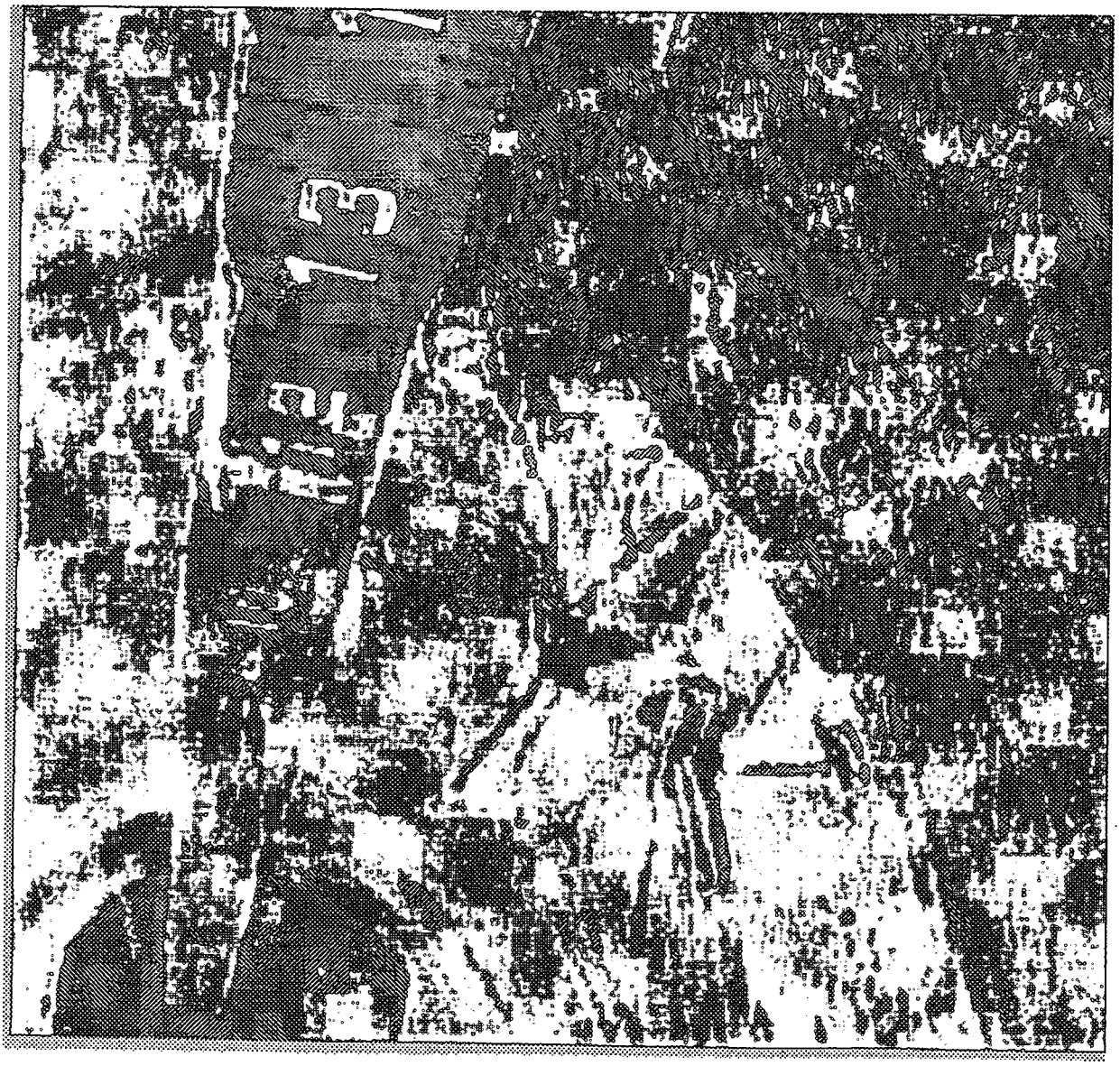
PHOTOGRAPH IV-6



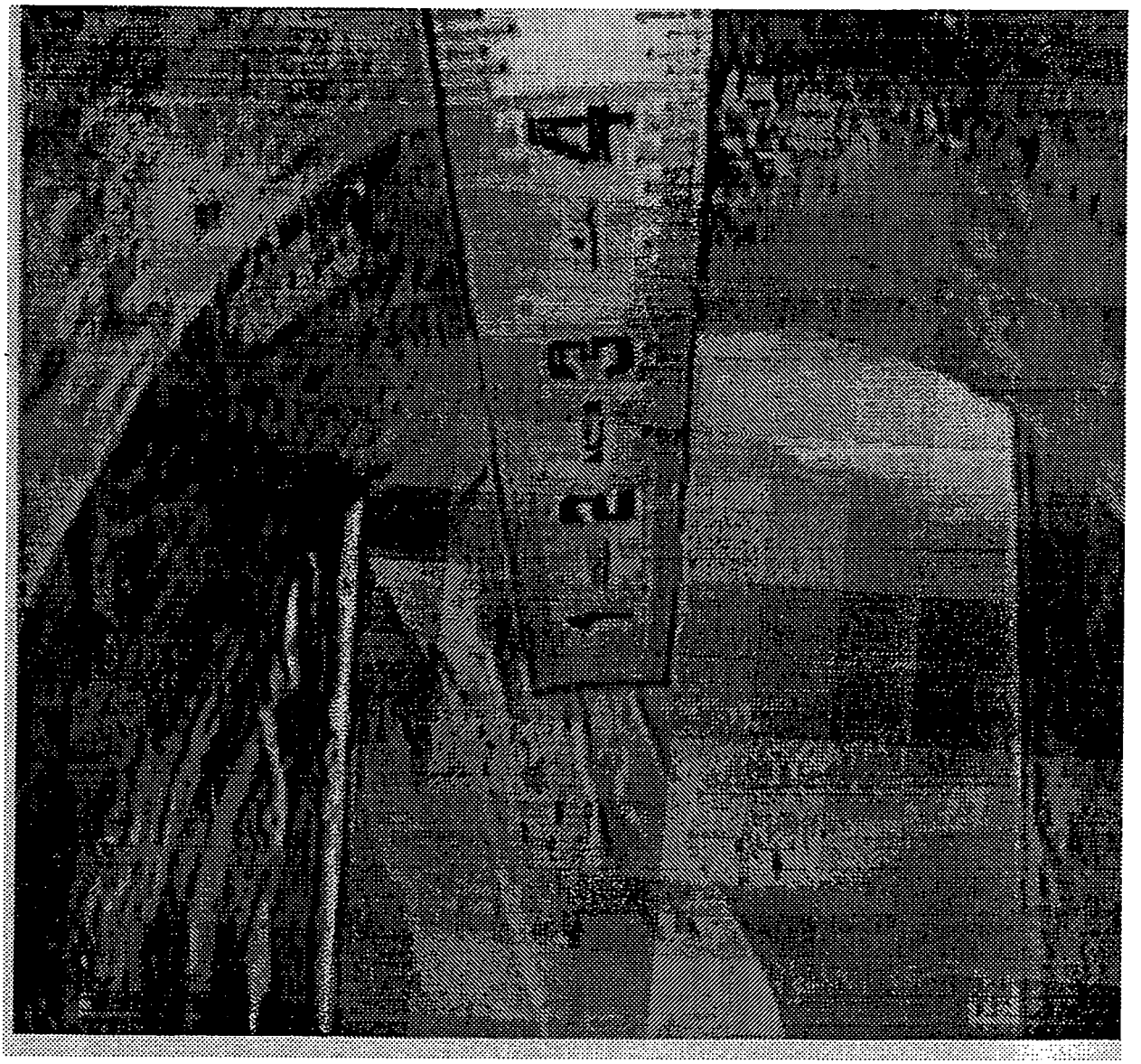
PHOTOGRAPH IV-7



PHOTOGRAPH IV-8

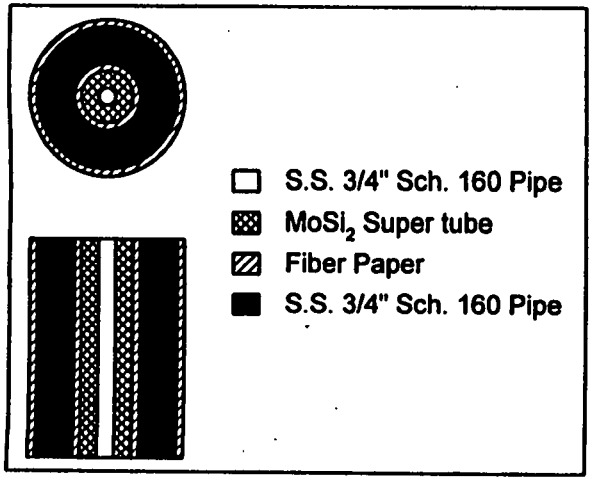


PHOTOGRAPH IV-9



PHOTOGRAPH IV-10





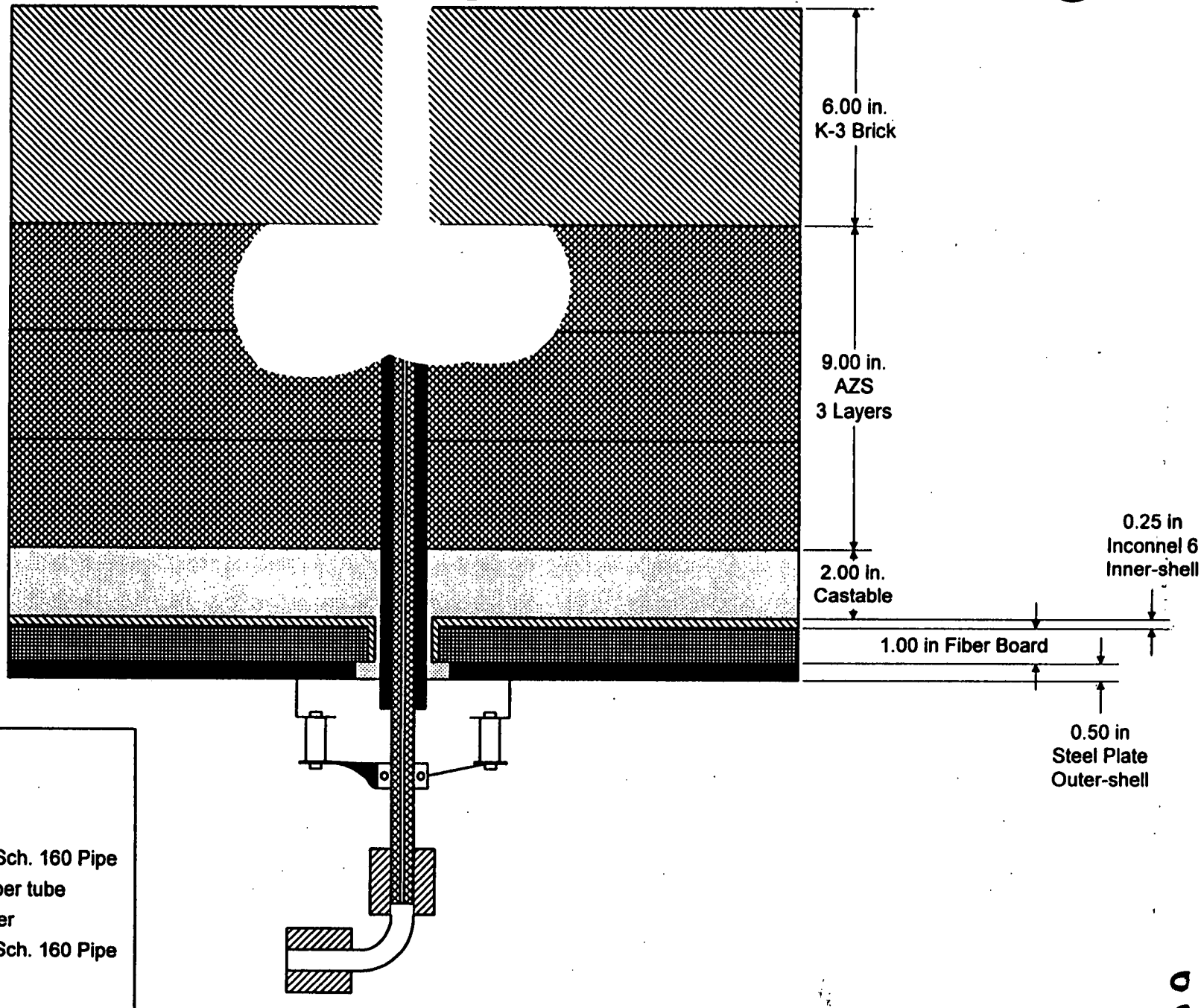


Figure IV-4 Bubbler #3 Wear Progression (3 of 6)

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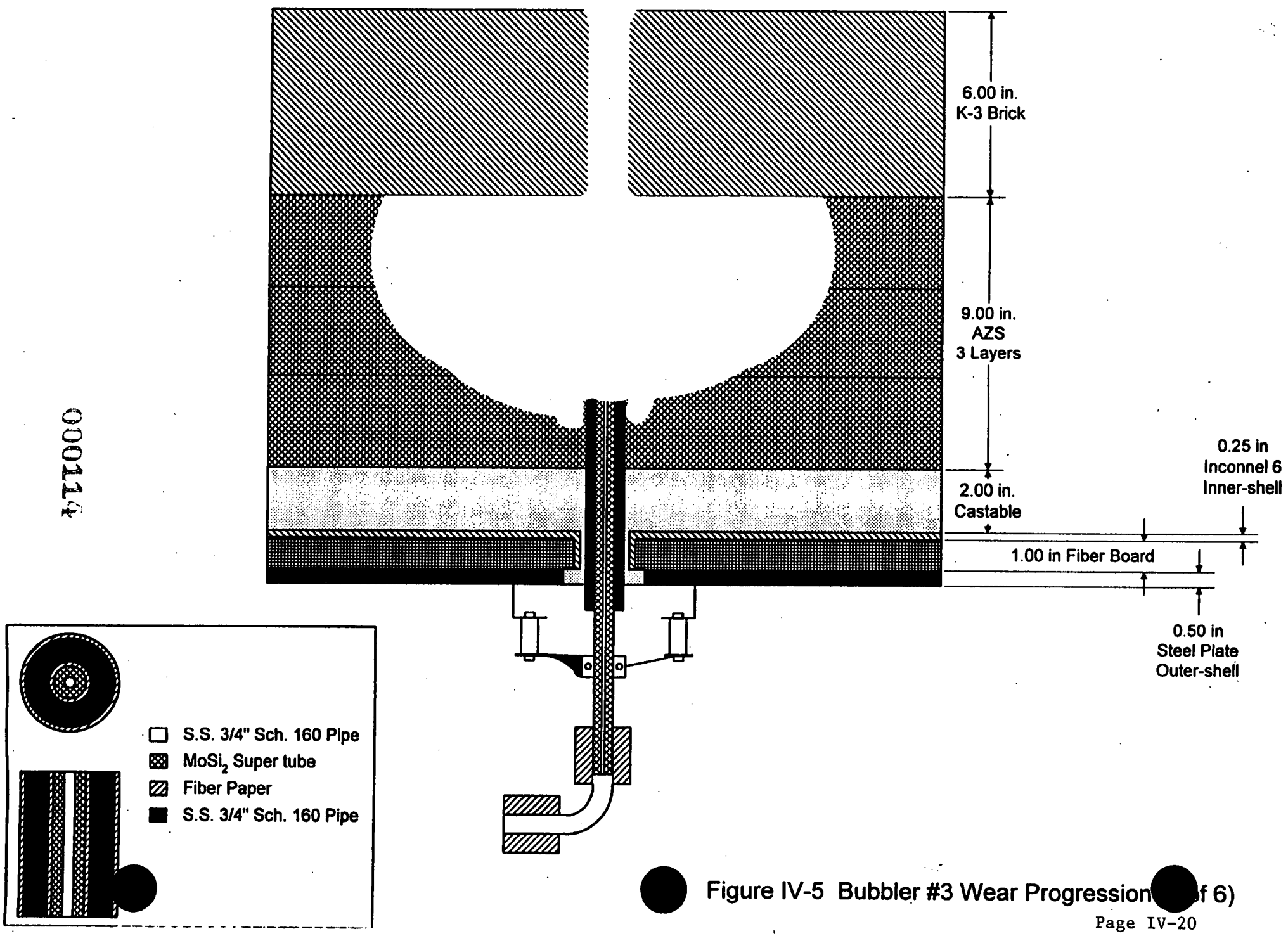
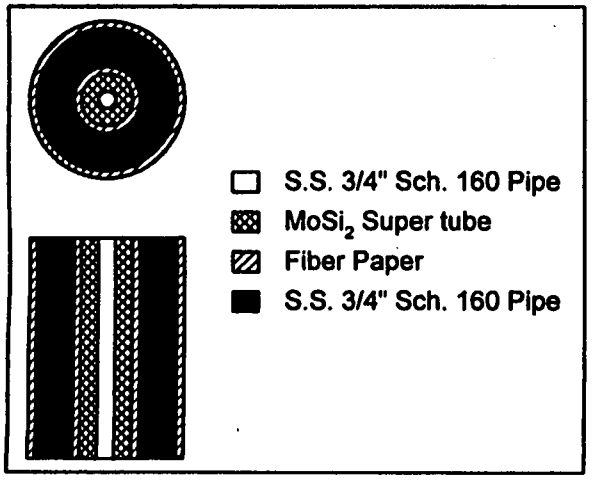
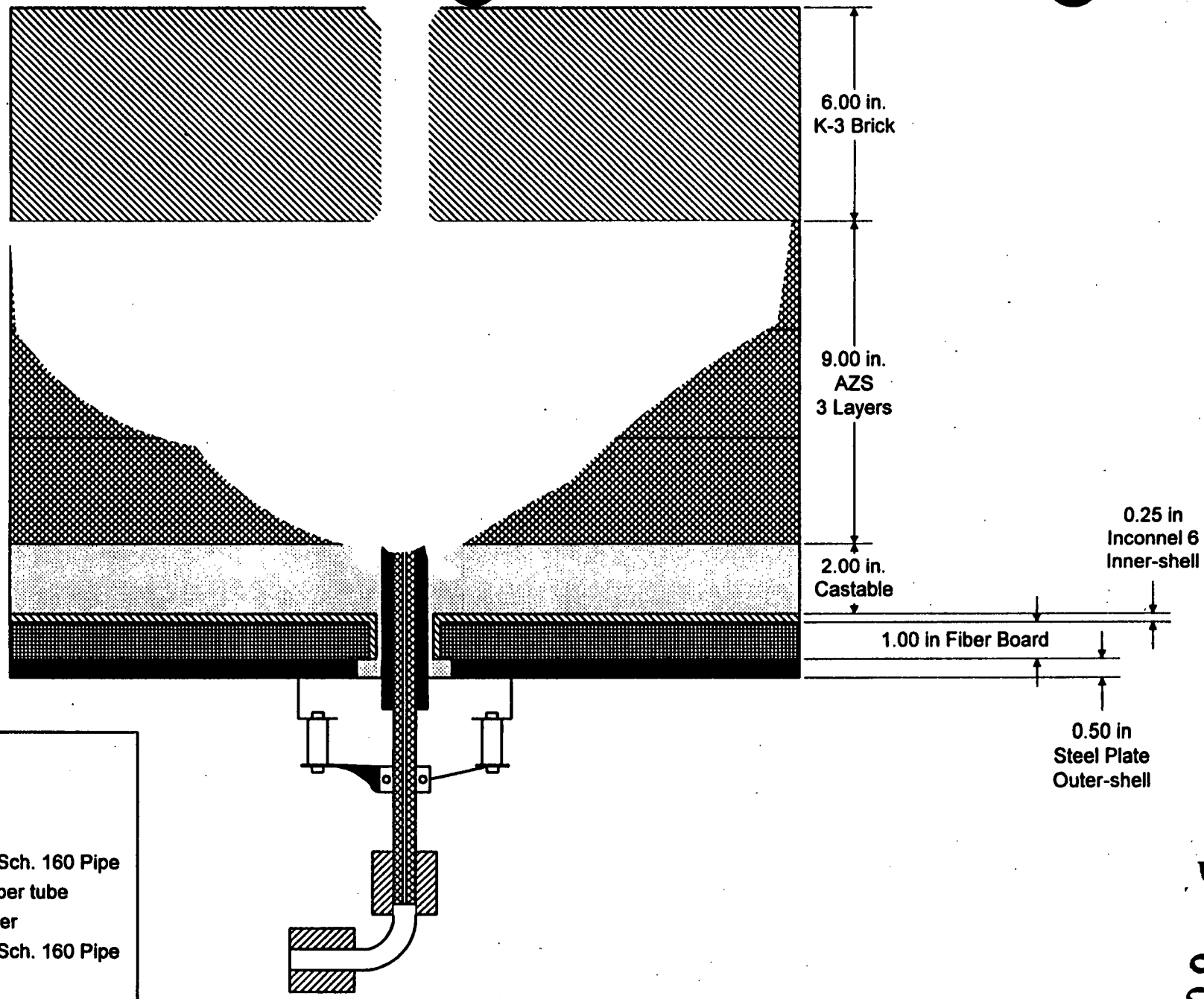


Figure IV-5 Bubbler #3 Wear Progression (1 of 6)



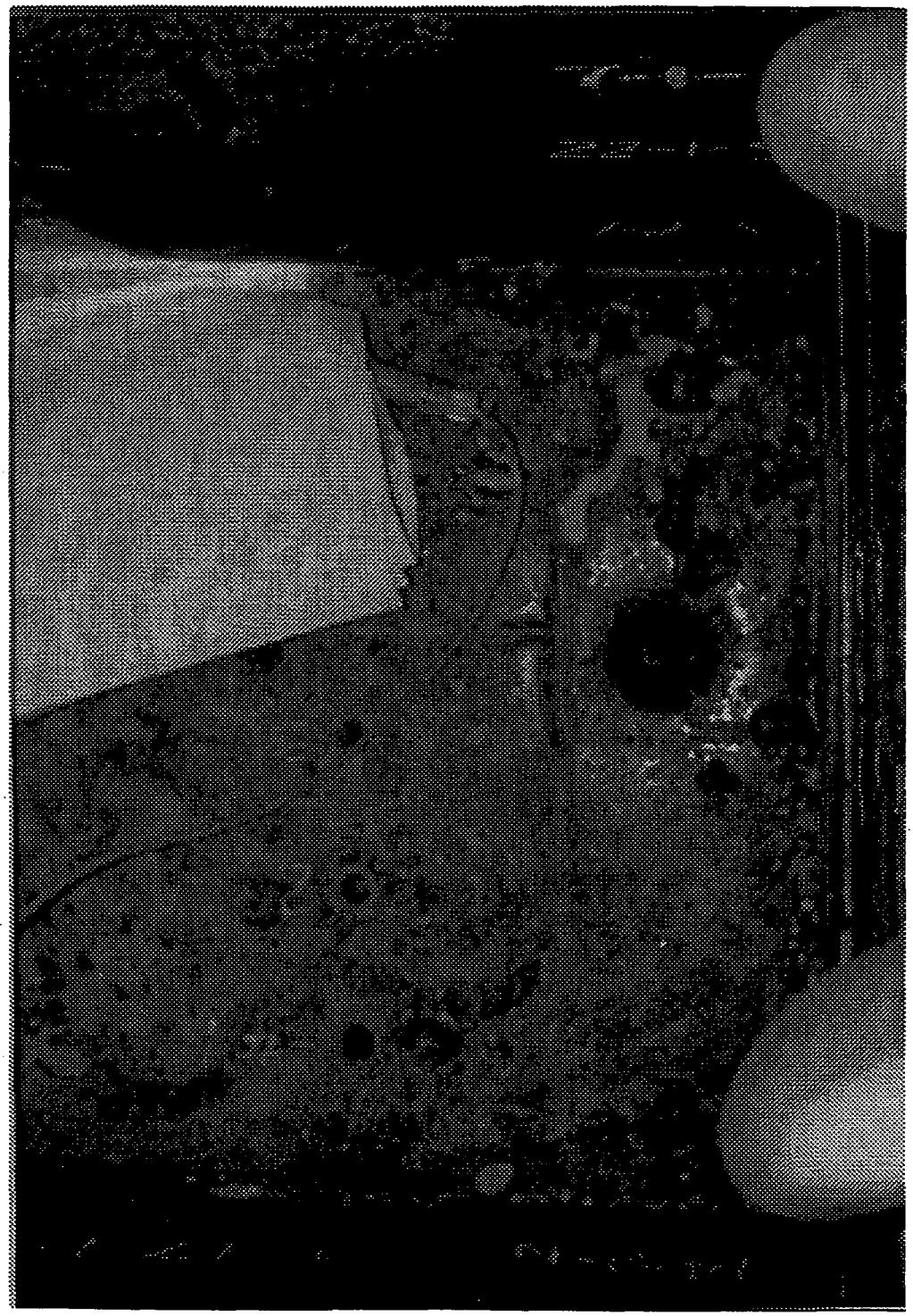


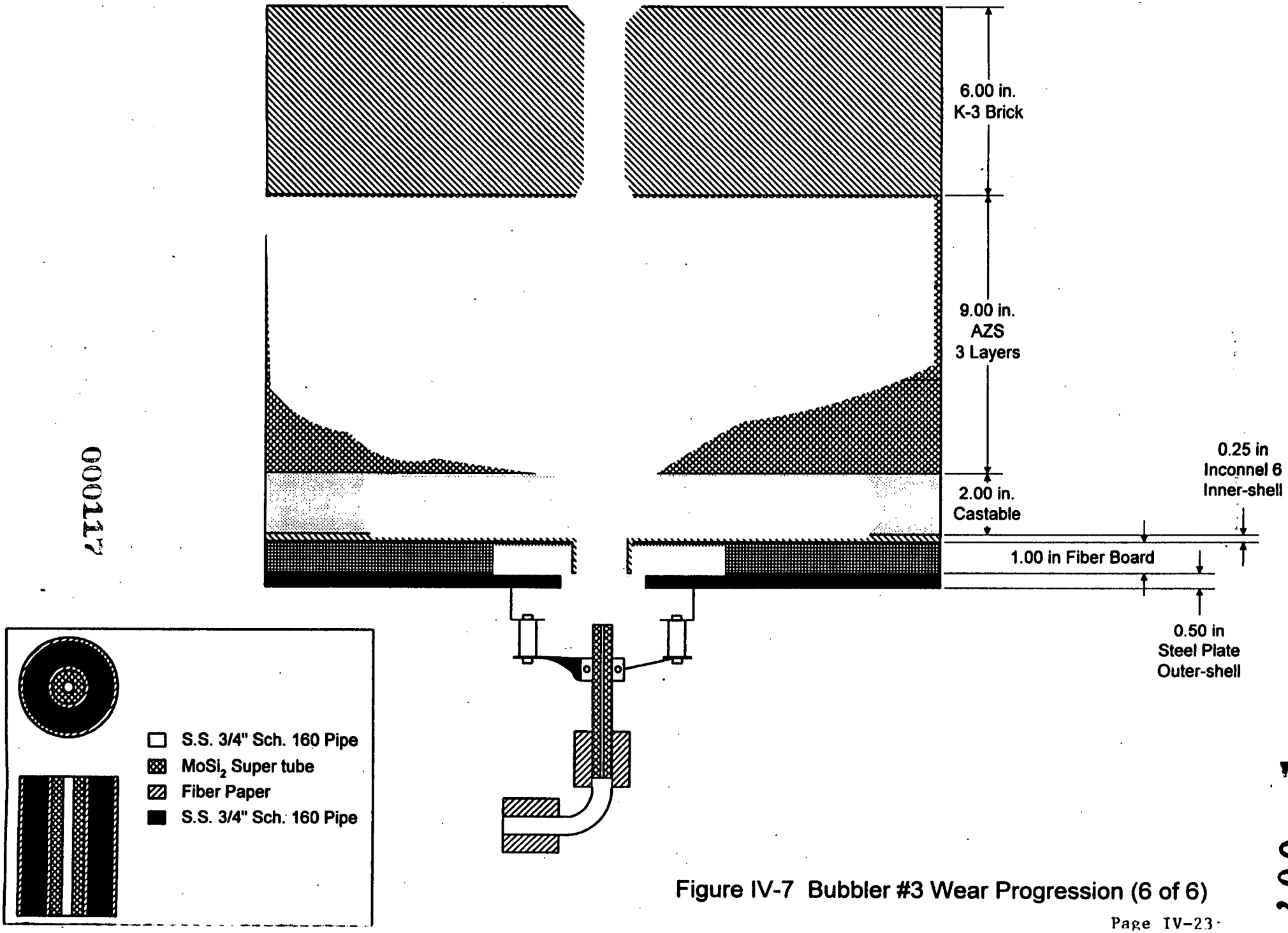
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Figure IV-6 Bubbler #3 Wear Progression (5 of 6)

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PHOTOGRAPH IV-11





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Figure IV-7 Bubbler #3 Wear Progression (6 of 6)

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Visual inspection by the boroscope of the cored areas revealed that the first layer of AZS brick directly underneath the Monofrax K-3 bubbler blocks had been dissolved and replaced with glass. There was extensive cavitation in this area that was caused by the glass flowing from this area when the melter emptied through Bubbler 3. Turbulence from the compressed air aided the erosion of the bonded AZS while the glass continued to corrode the AZS through its lower layers.

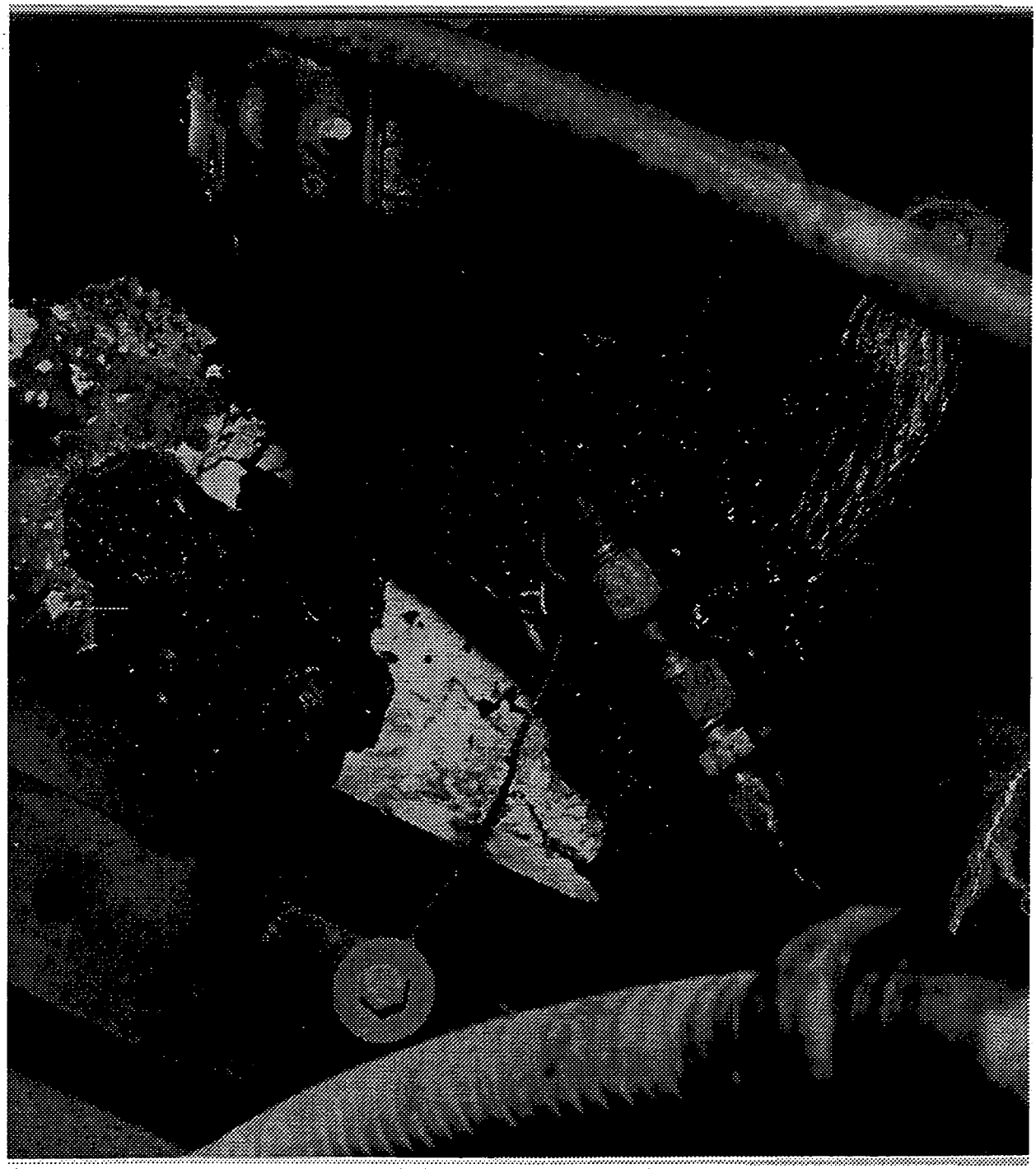
- Downward drilling occurred throughout the underlying refractory due to the formation of molten lead in the glass melt. Core borings of the bubbler tube areas revealed that molten lead and the mechanism of downward drilling were instrumental in the corrosion of the underlying refractories. Paths made from this drilling effect enhanced the migration of the glass melt through the AZS refractory, thus reducing the melter life expectancy. There were visible quantities of molten lead droplets throughout the glass melt matrix. A single lead plug was also noted attached to the Bubbler 3 anchoring assembly at the point of the melter breach (Photograph IV-12). Refractory materials within the areas of molten metal droplets are preferentially dissolved. Since the lead droplets are more dense than the glass, they drill downward through the refractory, providing pathways for accelerated migration of the glass melt downward and eventually outside the melter confinement.

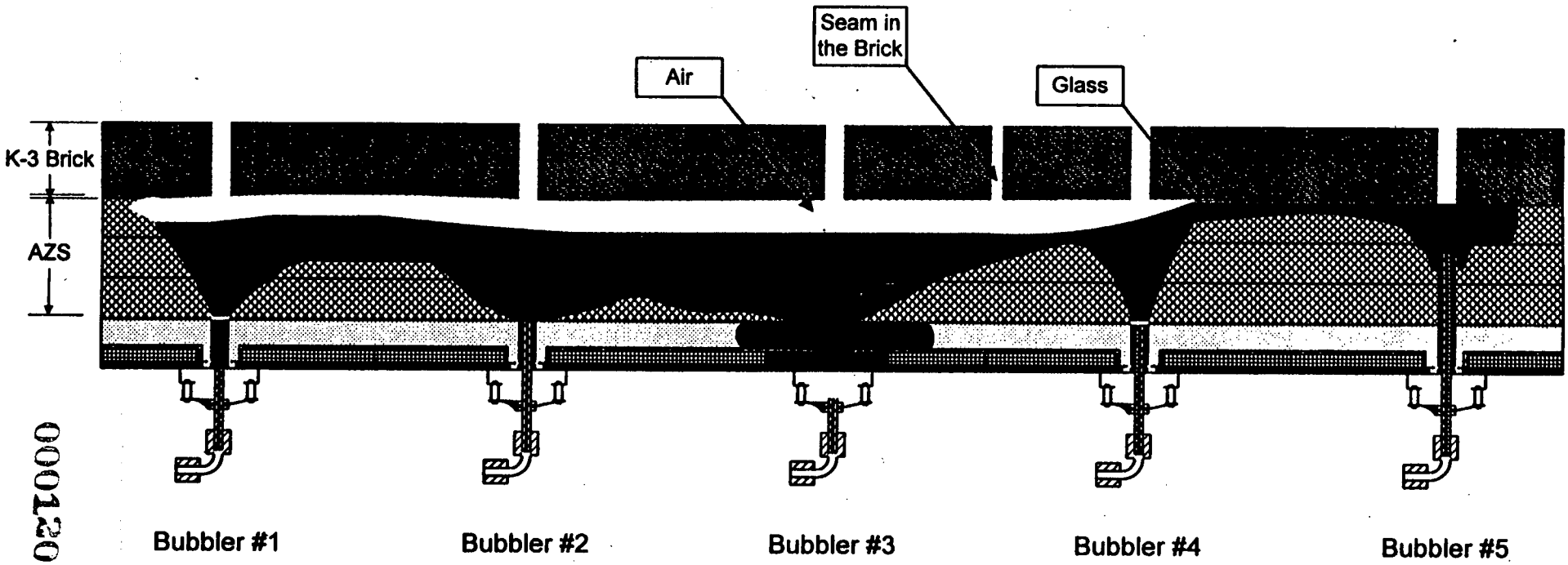
All the aforementioned mechanisms were providing continuous and highly corrosive processes which dissolved away the bubbler tube assemblies and the underlying AZS refractory, allowing the lead glass melt to reach the castable refractory barrier. The KAO-TAB95 castable insulating refractory has little corrosive resistivity. The breach of this barrier was certainly most rapid (Figure IV-8).

Summary

In summary, the visual observations from core boring through the bubbler tube assembly areas have verified that the bubbler tube assemblies corroded back into the castable insulating refractory (the final barrier before breach of the melter). The underlying AZS and castable refractories experienced extensive corrosion, resulting in significant cavitation and channeling of the glass melt throughout the refractory lining underneath the bubbler tube Monofrax K-3 blocks. In addition, it is apparent that significant channeling of the refractory and downward migration of the glass melt occurred underneath the surface paving (Monofrax K-3 hot face refractory), including the bottom drain areas as well. Downward drilling from molten lead exacerbated the corrosion of the underlying refractories and accelerated the migration of the glass melt downward and eventually out of the melter. The pathway of least resistance was the bubbler assembly areas. The bottom drain penetrations were also potential failure areas, but due to water cooling in these areas, the corrosion and subsequent migration of glass downward were most probably reduced relative to the air cooled bubbler assembly areas.

PHOTOGRAPH IV-12





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Figure IV-8 Glass Infiltration in the Air Bridge

Glass Chemistry

Tables in Appendix G, number 1 through 7, show the various compositions of glass used throughout the seven-month testing period. Tables 8 through 16, also in Appendix G, show the mass balance calculations associated with lead and barium.

Redox State of Side Chamber Glass Samples

On October 15, 1996, samples of the west and east side chambers of the Vitrification Pilot Plant (VITPP) Melter were collected and submitted for redox determination at a contract laboratory. Final sample results were received from this lab during the second week of December. The samples were collected during the processing of post-Campaign 2 rejected slurry batches. The samples identification numbers were S10W-961015-1530 and S10E-961015-1525. Laboratory personnel reported that the east side chamber sample was 74% reduced and the west side chamber sample was 50% reduced. In accordance with the Melter Inspection Plan (Appendix C), additional glass samples have been sent for analysis to verify this condition. Final analytical results will be presented in an addendum to this report.

Lead Mass Balance for Campaign 2

The results listed in the "REG." column in Table 9 (Appendix G) are results presented by the Analytical Laboratory Service (ALS) department. These results are the analyzed weight percent of the samples submitted. All quality control samples met the laboratory's acceptance criteria.

Due to low total weight percents, the results were normalized to bring the total weight percent to 100 wt% per individual sample. These results are listed in the "NORM." column in Table 10 (Appendix G).

Samples with unusually low disilicide weight percents (analyzed) cause the normalized value for other constituents to be increased, making the normalized lead result for these samples suspect.

The percent of lead retained in the glass is included in the tables mentioned above (Appendix G) for both the straight analysis and normalized results.

Lead Mass Balance for Campaign 4

During Campaign 4, a total of 1,926 lbs. of lead oxide was introduced into the VITPP feed preparation system. Approximately 1,836 lbs. of lead were retained in the product glass, which is 95% of the total amount according to the review and reduction of analytical data. Combining the amount of lead retained in the glass with the estimated amount of lead discharged in the off-gas system, approximately 96% of the lead oxide introduced to the VITPP feed preparation system was accounted

for. The variability surrounding these approximations is not known. Although lead is visible as minute beads suspended in the glass matrix in the understructure of the melter, the total amount of lead reduced cannot be accurately determined.

The lead mass balance essentially accounts for all the lead because the same mass balance was done for lead and barium and the results were very similar. Approximately 92% of the barium was accounted for in the mass balance as compared to 96% of the lead. Because barium is a very reactive metal, barium metal can only precipitate from the glass melt under very extreme redox conditions (> 99.9% reduced). Comparing the numbers implies that the accuracy of determining the mass balance is within approximately 5%. Therefore, the amount of lead in the bottom of the melter should be of the order of 10s of pounds, not 100s of pounds. This could have come from the corrosion reactions of the molybdenum electrodes and the molybdenum disilicide parts in the melter.

It is postulated that there were three primary mechanisms resulting in molten lead forming in the main melter chamber:

- Reduction of lead in the east and west side chambers occurred. The interconnections between the main and side chambers resulted in the main chamber glass mixing with the side chamber glass, where lead could easily become reduced due to lack of an oxidizing environment and interaction with the molybdenum electrodes. The molten lead could then either penetrate the sub-layer refractory through the bottom drain assemblies or possibly flow to the main chamber through crack penetrations in the E-walls.

- Corrosion of the molybdenum disilicide bubblers causes the precipitation of free lead in the bubbler tube assemblies.

- Localized hot spots within cracks of the E-wall (where electric current could have passed through the E-wall itself) may have resulted in increased temperatures causing an increase in the reduction of lead oxide. The lead could then have flowed to the main chamber and penetrated to the sub-layer refractory either through seams or bottom penetrations (bubblers or bottom drains).

Thermal and Electrical Conditions

Both temperature and grounding conditions impacted the corrosion of materials of construction which, in turn, facilitated the migration of the glass melt downward through the refractory lining. Following is a discussion of these parameters:

Temperature Profiles and Power Requirements

Prior to, and up through, the transition batch (B09) for low-temperature (1,150°C) Silos 1 and 2 surrogate testing in Campaign 4, the 1,150°C idle requirement was slightly increased from approximately 105 to 110 Watts per Degree C as indicated in Table17 of Appendix G. Please note that the accuracy of producing such a chart is questionable, because of the down times, at which the data for several instances were recorded are short. The melter may not have had enough time to come to equilibrium. The melter may be "burning" left-over sulfate, and this takes extra heat/power. However, it does appear that after batch B09 of Campaign 4, and just before the unexpected draining of the melter, the idle power requirement increased to 130 Watts per Degree Celsius (Figure IV-9).

There are two possible scenarios, not exclusively independent, which could explain this phenomenon and could be directly related to this event:

- Glass was permeating toward the melter outer shell (verified by core borings). This drew the heat from the melter close to the outer shell of the melter, increasing temperature at the surface and thereby increasing the heat loss. Batch B09 of Campaign 4 was a Series A low-temperature transition batch. This batch produced a thin (less viscous) conductive glass. This thinner glass allowed deeper penetration through the refractory lining, thereby increasing power requirements.

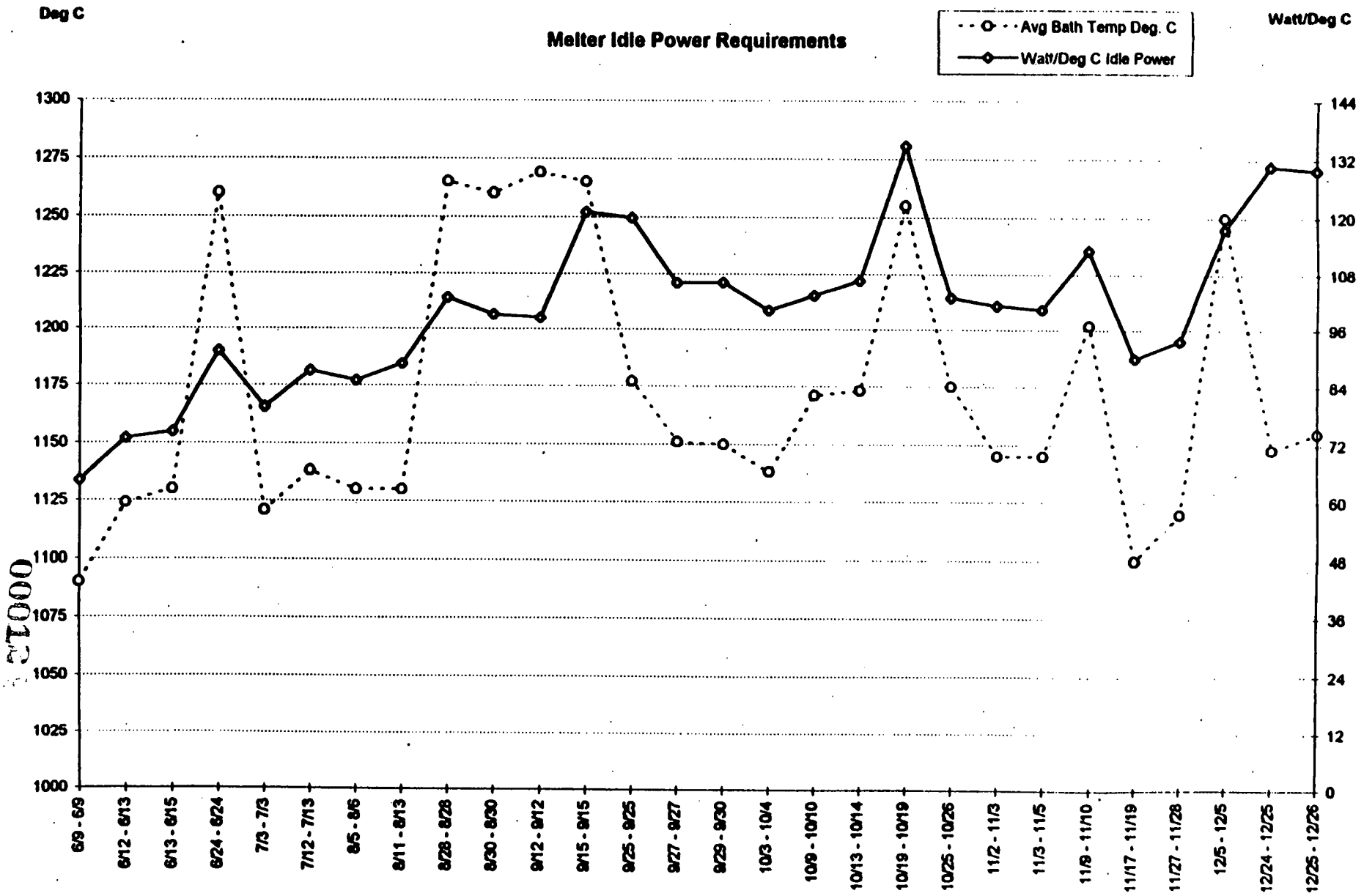
- A ground developed from electrode to electrode when molten glass or molten lead came in contact with the inner shell in two independent locations. This would allow the inner shell to complete a circuit between the electrodes. The heat generated by this circuit would be very close to the outer shell would increase the idle power requirements, would heat the glass more and finally cause self discharge from the melter.

Summary

In summary, there were no significant temperature or power excursions identified during idle conditions prior to Batch B09 which would have indicated abnormal operating conditions. Power increases seen after Batch B09 can be attributed to the glass melt penetrating close to the inner shell and grounding of the inner shell by either molten lead, or the glass melt, or a combination of the two.

Refractory degradation as a result of lead formation and glass migration into the refractory was a chronic event that increased exponentially with time. This exponential curve culminated in the melter breach at Bubbler 3.

FIGURE 2



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V. OBSERVATIONS & RECOMMENDATIONS

ROOT CAUSES

Three causal factors, identified during the root cause analysis, which were the primary contributors for the discharge of glass through the bottom of the melter. The root cause analysis was bounded by the parameters of the as-built melter at the VITPP. Contributing factors to the failure and other potential areas for improvement are included in the lessons learned section of this report. The primary causal factor was the corrosion of the molybdenum disilicide bubbler tubes. Other contributing factors were the generation of electrical current to ground paths inside the melter, and glass migration and/or metals precipitation to the bottom of the melter.

Observation R1

The bubbler tube assemblies were not resistant to corrosion by the glass, or metal phases in the glass melt, nor was the molybdenum disilicide tube resistant to an oxidation reduction reaction with lead oxide.

Recommendation R1

Molybdenum disilicide should be treated as a consumable material. Designs must consider the need to continuously replenish it. (Reference 13 and 14)

Observation R2

The bonded AZS refractory, although possessing excellent thermal shock properties, was not resistant to downward drilling by metal nor did it possess a high corrosion resistance to the glass melt. The failure of this barrier contributed to the glass migration to the bottom of the melter and also may have contributed to the formation of electrical current to ground and the inner shell.

Recommendation R2

Refractory design should consider use of material with exceptional corrosion resistance to glass melts and to metal attack. A graded layering of chrome-bearing fused cast refractories such as Monofrax K-3 or fused cast AZS refractory (instead of bonded) should be considered in future refractory designs. It is important that the selected material be continuous from the glass pool to the outer shell around any melter penetrations.

Observation R3

Penetrations through refractory containing the melt pool (i.e., bottom drains, bubbler assemblies and thermowells) resulted in extensive migration of the glass melt and metals to sub-layer refractory.

Recommendation R3

Penetrations through melt pool refractory should be minimized to the extent possible in future melter designs.

Observation R4

Materials of construction testing for melter components and refractory lining was not conducted.

Recommendation R4

Careful consideration should be given to materials of construction testing on melter components and refractory with both glass surrogate materials and actual silo waste. Chemical reactions including redox should be considered.

Observation R5

Glass migration and metals precipitation to the inner shell and to areas between the inner and outer shells resulted in the formation of a disruptive current path. The formation of the electrical current path was facilitated by the presence of inner and outer shells, because the two shells were constructed of electrically conductive material. The disruptive electrical current path may have contributed to the heating of the sub-surface glass pool and prevented the glass from solidifying below the surface refractory.

Recommendation R5

Future melter design(s) should consider the elimination of electrically conductive shells enclosing the refractory. The materials of construction should provide a temperature profile from melt pool to the outer melter surface that freezes migrating glass well in advance of the glass reaching the outer container.

Observation R6

Although the project was performing reduction/oxidation (redox) monitoring in accordance with project procedures, process experts stated that better redox monitoring could be accomplished via analytical analyses. Improved redox monitoring and controls may minimize metals precipitation at the bottom of the melter.

Recommendation R6

Project management should re-evaluate the monitoring system in place to control reduction/oxidation (redox) chemical reactions to determine if the monitoring system meets the needs of the project.

LESSONS LEARNED

The following lessons learned are the result of the Event and Causal Factor Chart developed for the operational analysis. These items were determined by other events and conditions which were not directly related to the failure of the melter, but were deemed as contributing causes and/or potential areas for improvement.

Observation L1

Project personnel identified a concern about bubbler tube erosion, and the concern was addressed in the initial Phase I Test Plan and Hazards and Operability Report. The concern was omitted from the Final Hazard Analysis Report without mitigating measures identified or implemented.

Recommendation L1

The project organizational structure should include an independent external technical review by industry experts. In addition, concerns that arise must be captured and maintained until formal resolution is reached through an approval process.

Observation L2

Project personnel's level of knowledge in melter design, components and operations was less than adequate. Project personnel were not trained to recognize the limitations of the unique melter design and the components used for construction of the melter.

Recommendation L2

Managers, engineers, operators, and maintenance personnel should be trained by an expert in melter design, operation, and unique phenomena associated with the melter to develop a basic understanding. Industry experts should be used as consultants in the beginning of the project. Research of industry publications of material suppliers must be conducted to verify the quality of contracted designs.

Observation L3

The invitation for bid did not satisfactorily address existing procedural requirements. The design change control process did not start until construction began.

Recommendation L3

Contracts must identify the site and functional area requirements in contract specifications prior to Invitation for Bid (IFB) and Request for Proposal (RFP) to assure that quality products are delivered (i.e., the design change control process must be formalized and documented).

Observation L4

The engineering, procurement and construction (EPC) process was compromised by the project's desire to recover from incurred schedule slippages. Concurrent design led to design changes, adaptations, additional schedule slippages, and cost over-runs.

Recommendation L4

Design integration is essential and should not be compromised to maintain schedules. Increased design controls are required when more than one design organization is responsible for deliverables. Design change control should be applied during the Title I Design to verify and justify deviations from the design functional requirements.

Observation L5

Numerous design deficiencies were identified during melter operations and during the post-incident melter inspection.

Recommendation L5

The following recommendations would enhance melter design performance:

- Materials of construction must be evaluated for form, fit, function, reactions to each other, and life expectancy (e.g., the interaction of molybdenum disilicide and lead, riser block, AZS, etc.)

- Consider alternate melter design (i.e., gas, low-temperature, electrical)
- Minimize or eliminate refractory penetrations
- Develop reliable temperature monitoring of the glass pool
- Install ground current monitoring of electric melters
- Improve reliability of the melter discharge chamber
- Improve reliability of the melt pool level indication
- Redundancy of support and backup systems need to be evaluated (e.g., lack of redundancy for SCR 1)
- Performance specifications should be developed with the aid of industry experts during the conceptual design phase, subsequent design reviews, and technology selection.

Observation L6

Due to a lack of timely analytical results, operational productivity was impacted, reaction to changing conditions was delayed, and assessment of the impact of operational adjustments could not be ascertained. The project had to rely on three remote laboratory facilities.

Recommendation L6

Provide adequate resources to improve the turn around time of chemical analytical results. Project personnel should inform vital support organizations in advance that increased services may be required, depending on variables experienced during operation. This advanced notice will enable the support organization to plan for increased demands for services by the project.

Observation L7

Critical components were not evaluated collectively for operational impact. Breakdown of level indicators and thermal wells were detected during operations, but other components made of the same material, such as air lifts and bubblers, were not evaluated for potential failure.

Recommendation L7

Develop a material failure and trending process to identify deficiencies that potentially can affect similar processes or materials prior to a failure. Develop a failure mode analysis for all systems and components prior to operations and develop casualty procedures to minimize the effects of an abnormal event or condition. Thoroughly develop data quality objectives that include the materials of construction, additives, products, by-products, lubricants and solutions to assure that all process materials and components are baselined before start-up and monitored during operations.

000130

APPENDICES

APPENDIX A

FLUOR DANIEL FERNALD

INTEROFFICE MEMORANDUM

To:	R. P. Heck, MS52-5	**Date:**	December 30, 1996
Location:	Fernald	**Reference:**	N/A
From:	R. P. Scholz, MS31	**FD Fernald #:**	M:OPI(ES/EP):96-0045
Location:	Fernald	**Client:**	DOE DE-AC24-92OR21972
Extension:	648-5562	**Subject:**	**NOTIFICATION REPORT FOR OH-FN-FDF-FEMP-1996-0075**

Attached is a copy of the Occurrence Notification Report, OH-FN-FDF-FEMP-1996-0075, generated on an event which occurred in your area of responsibility. The initial report was completed and issued to the Department of Energy (DOE) within the specified time requirement.

As required by DOE Order 232.1-1, which replaced DOE Order 5000.3B, a Final Occurrence Report is required for this event. Information for the Final Report is due to the Occurrence Reporting Team (ORT) within thirty-five (35) calendar days of event categorization. When the ORT receives complete information, the report will be generated and routed for approval before it is submitted to the Occurrence Reporting and Processing System (ORPS).

Effective October 1, 1996, a POC has been applied to Occurrence Reports for: 1) timeliness in submitting the Final Report, and 2) closure of corrective actions on or before the target completion date. Written notification must be sent to the ORT stating when each corrective action is completed. Supporting documentation (i.e., Procedure Change Notices, meeting minutes, attendance rosters, pictures, memos, copies of cc:mail messages, etc.) for the completion of each corrective action must accompany your written notification.

Your Final Report information is due to the ORT on January 31, 1997.

The ORT is available to assist you in the development and approvals of the Occurrence Report. Please feel free to call Laura Morrow (648-4175) or Ron Joseph (648-4313) if you have any questions.

RPS:llm
Attachment

ES-F-3934 (8 31/95)

FLUOR DANIEL FERNALD

INTEROFFICE MEMORANDUM

c: With Attachment
R. B. Allen, DOE-FEMP, MS45
P. M. Bishop, FDF, MS43
J. B. Bradburne, FDF, MS17
G. J. Brown, DOE-FEMP, MS45
R. C. Collison, DOE-FEMP, MS45
J. E. Curry, FDF, MS43
R. D. Daniels, FDF, MS30
P. A. Darnell, DOE-FEMP, MS45
P. L. Doherty, FDF, MS15
L. E. Doll, WISE Construction, MS16-2
R. E. Gist, DOE-FEMP, MS45
K. M. Graham, FDF, MS82-3
G. G. Griffiths, FDF, MS45
T. D. Hagen, FDF, MS65-2
R. J. Hiestand, FDF, MS52-4
R. P. Hennerd, IGUA, MS22
D. L. Howe, FDF, MS30
S. K. Kaster, DOE-FEMP, MS45
D. W. Kirby, FDF, MS31
D. R. Kozlowski, DOE-FEMP, MS45
L. L. Morrow, FDF, MS31
W. J. Neyer, DOE-FEMP, MS45
D. A. Nixon, FDF, MS52-4
J. O'Donahoe, DOE-FEMP, MS45
D. Paine, FDF, MS52-4
L. E. Parsons, DOE-FEMP, Contracts Specialist, MS45
D. A. Pfister, DOE-FEMP, MS45
S. M. Pope, IG, MS54
W. J. Quaider, DOE-FEMP, MS45
D. L. Riley, DOE-FEMP, MS45
T. L. Rutherford, FDF, MS3
R. M. Schwab, FDF, MS33
J. Shine, DOE-FEMP, MS45
K. A. Solomon, FDF, MS52-4
File Record Storage Copy 106.4.32.4.2

FS-F-3934 (8-31-95)

(Name of Facility)

Balance-of-Plant

(Facility Function)

Fernald Environ. Mngmnt. Project

(Name of Laboratory, Site, or Organization)

Name: Don Paine
Title: Silos Project Manager Telephone No.: (513)648-

(Facility Manager/Designee)

Name: JOSEPH, RONALD L
Title: HEALTH PHYSICS SUPERVISOR Telephone No.: (513)648-

(Originator)

Name: Date:

(Authorized Classifier (AC))

1. OCCURRENCE REPORT NUMBER: OH-FN-FDF-FEMP-1996-0075
   Vitrification Pilot Plant Melter Breach

2. REPORT TYPE AND DATE: Date Time
   [X] Notification Report 12/27/1996 1152
   [ ] Initial Update
   [ ] Latest Update
   [ ] Final Report

3. OCCURRENCE CATEGORY:
   [ ] Emergency [X] Unusual [ ] Off-Normal [ ] Cancelled

4. NUMBER OF OCCURRENCES: 01 ORIG. OR:

---

5. DIVISION OR PROJECT: FDF/FEMP

6. SECRETARIAL OFFICE: EM - Environmental Management

7. SYSTEM, BLDG., OR EQUIPMENT:
   Vitrification Pilot Plant Melter

8. UCNI?: No

9. PLANT AREA: Vitrification P

10. DATE AND TIME DISCOVERED:
    12/26/1996 2230

11. DATE AND TIME CATEGORIZED:
    12/27/1996 0905

12. DOE NOTIFICATION:

12/27/1996	1020	Rick Kahn	DOE-H

13. OTHER NOTIFICATIONS:

12/27/1996	0800	Tim Marcus	DOE-C
12/26/1996	2245	Gordon Brown	DOE-F
12/27/1996	0005	Joe Neyer	DOE-F

14. SUBJECT OR TITLE OF OCCURRENCE:
Vitrification Pilot Plant Melter Breach

15. NATURE OF OCCURRENCE:
10) Cross-Category Items
C. Potential Concerns/Issues

16. DESCRIPTION OF OCCURRENCE:
On December 26, 1996, at approximately 2222 hours, a Fluor Daniel Fernald (FDF) Vitrification Pilot Plant (VPP) Maintenance Supervisor was sent to the Melter Room by the System Engineer to inspect the West Camera for damage. The camera had recently been adjusted by an Instrument Mechanic and the Melter Operator, who was located on the second floor of the Melter Room, noticed a wisp of smoke in the vicinity of the camera. When the Maintenance Supervisor entered the Melter Room (on the ground floor), she noticed a small stream of molten glass flowing from the bottom VPP Melter into the Bottom Drain Container.

Prior to this discovery, the VPP was conducting normal operations, processing surrogate material for testing and development of vitrification technologies. The material in the melter contains some concentrations of barium and lead. The material is not radioactive. The compliment of shift personnel included 9 VPP Team Members, 6 Maintenance personnel, and 1 Radiological Control Technician.

NOTIFICATIONS MADE:

AEDO - Assistant Emergency Duty Officer
EDO - Emergency Duty Officer
DOE-FEMP Duty Officer
DOE-OH Duty Officer
DOE-HQ Duty Officer
Public Affairs

17. OPERATING CONDITIONS OF FACILITY AT TIME OF OCCURRENCE:
Does Not Apply

18. ACTIVITY CATEGORY:
Normal Operations

000136

19. IMMEDIATE ACTIONS TAKEN AND RESULTS:
The Maintenance Supervisor alerted the System Engineer, who directed the Control Room Operator to ramp-down power to Silicon Control Rectifier (SCR) #1, in order to decrease Melter temperature. The Maintenance Supervisor notified Fire & Safety personnel via radio that smoke was present in the melter room, but no fire was observed. At 2225 hours, the Communication Center received the first smoke alarm from the VPP. The Communication Center Operator immediately notified the Assistant Emergency Duty Officer (AEDO). The System Engineer directed all personnel in the Melter Room to stand by the exit to the room on the first floor, near the Personal Protective Equipment (PPE) Room door. The Shift Manager attempted to "freeze" the glass stream (which was about the diameter of a pencil) by applying water to the stream from the Bottom Drain water hose. The attempt was abandoned after it was determined to not be effective in reducing the flow of glass from the Melter. At 2234 hours, the Shift Supervisor directed the Control Room Operator to initiate an emergency shutdown of the Melter by depressing the Emergency Stop Button and ordered the evacuation of all personnel from the building. Two electricians were ordered to open the main electrical disconnect for SCR #1 and SCR #2, which resulted in the complete electrical isolation of the Melter.
As the glass continued to flow from the Melter, the hole widened to approximately 2 inches in diameter. Some of the flow was diverted and fell outside of the Bottom Drain Container and onto a concrete floor. The epoxy paint on the floor ignited and a small (about 2' X 2') fire resulted. Two portable fire extinguishers were unsuccessfully expended on the fire. Emergency Response personnel arrived on the scene by 2240 hours and put out the fire by coating the floor with water. Approximately one cubic yard of surrogate material leaked from the Melter. An accountability of the evacuated personnel was performed and some personnel were released to assist with the control and recovery actions.

The event was initially categorized as an Off-Normal Occurrence at 2350 hours by the AEDO. A post-event critique was conducted on December 27 at 0830 hours. Following the critique, the event was up-graded to an Unusual Occurrence at 0905 hours.

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24. EVALUATION: (By Facility Manager/Designee)
There was no significant impact to the safety and health of any off-site or on-site personnel as a result of this occurrence. Equipment damage estimates cannot be determined at this time, but will be included in the Final Report.

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25. IS FURTHER EVALUATION REQUIRED?: Yes [X] No []

IF YES - BEFORE FURTHER OPERATIONS?: Yes [X] No []

BY WHOM?: Silos Project Management

000137

BY WHEN?: 01/10/1997

---

36. SIGNATURES:

Approved by: _____ Date: _____
Facility Manager/Designee Telephone No.: _____

APPENDIX B

Fernald Environmental
FERMCO
Restoration Management Corporation
(now renamed Fluor Daniel Fernald)
P. O. Box 538704 Cincinnati, Ohio 45253-8704 (513) 648-3000

January 2, 1997

Fernald Environmental Management Project
Letter No. C:OOTP:97-0003

Mr. Jack R. Craig, Director
Department of Energy
Fernald Environmental Management Project
P. O. Box 538705
Cincinnati, Ohio 45253-8705

Dear Mr. Craig:

CONTRACT DE-AC24-92OR21972, VITRIFICATION PILOT PLANT MELTER INCIDENT

As you know, on December 26, 1996, at approximately 2222 hours a breach in the melter shell resulted in the non-radioactive molten glass in the furnace to drain to the bottom drain container. In response to this event, Fluor Daniel Fernald and DOE-Fernald have formed the following three (3) teams:

1. Data Analysis and Path Forward Team
2. Incident Analysis Team
3. Safety Review Team

Please find attached a copy of each team's leader(s), members, charter and deliverables. The teams will assemble on-site January 6, 1997 and will target to complete their analysis by the end of January. The individual teams will be working closely with the Silos Project Independent Technical Review Team to ensure that the March path forward decision date is maintained.

This letter, along with the attachment, will be distributed to the attached list upon receipt of your concurrence.

If there are any specific questions pertaining to the subject effort, please contact Nina Akgündüz at (513) 648-3110 or Don Paine at (513) 648-5310.

Sincerely,

John Bradburne
President

Concurrence: 0001/89 1/3/97
Jack R. Craig, Director Date

JCB:DAN:kdg
Attachment

Distribution List:

DOE

N. Akgündüz
G. Brown
P. Darnell
J. Desormeau
D. Maynor
J. Neyer
S. Peterman
J. Reising
D. Yockman

FDF

L. Bogar
J. Bradburne
R. Crawford
D. Daniels
R. Frost
R. Gimpel
T. Hagen
B. Heck
R. Joseph
D. Nixon
D. Nordquist
D. Paine
B. Previty
R. Reinhart
H. Robertson
J. Smets
K. Solomon
R. Vogel
R. Worsley

CONS

G. Bingham
F. Carlson
C. Chapman
J. Edmondson
Refractory Industry (TBD)
B. Rusche
D. Stritzke

SRS

J. Plodinec

Numatek

TBD

FATLC

C. Shoust
B. Tabor

WVNS

TBD

Duratek

B. Bowen
H. Hojaji
X. Mao

FRESH 000141

V. Dastillung

File Record Storage Copy 102.1
Project Number 40100

January 3, 1997

DATA ANALYSIS AND PATH FORWARD TEAM

Leader:	N. Akgündüz (DOE)
Co-Leaders:	D. Paine (FDF), D. Nixon (FDF)
Members:	D. Yockman (DOE), R. Gimpel (FDF), J. Smets (FDF), R. Frost (FDF), H. Robertson (FDF), R. Vogel (FDF), T. Hagen (FDF), J. Plodinec (SRS), B. Rusche (Cons.), C. Chapman (Cons.), TBD (Numatek), D. Stritzke (Cons.), J. Edmondson (Cons.), B. Tabor (FATLC), V. Dastillung (FRESH)
Charter:	Evaluate data and determine deficiencies for detailed design and operation. Determine need for low temperature melts and alternatives to VITPP restart. Determine melter "Post Incident" data needs. Determine "Path Forward" for the VITPP Phase I Test Program.

Deliverables:

1. Develop database of outstanding data requirements and operations experience that would have been gained from the balance of Campaign 4 to support full scale detailed design.
2. Provide recommendations for acquiring outstanding Phase I data and operating experience.
3. Provide options for the path forward for the VITPP Phase I Test Program.
4. Cost/benefit analysis for path forward options.

000142

Attachment 209

January 3, 1997

INCIDENT ANALYSIS TEAM

Leader: R. Reinhart (FDF)

DOE Oversight: J. Neyer, J. Desormeau

Members: G. Bingham (Cons.), X. Mao (Duratek), B. Bowen (Duratek), H. Hojaji (Duratek), C. Shoust (FATLC), J. Edmondson (Cons.), J. Bradburne (FDF), R. Worsley (FDF), K. Solomon (FDF), D. Paine (FDF), R. Joseph (FDF), WVNS (Representative), D. Maynor (DOE-OH), F. Carlson (Cons.), Refractory Industry (Representative)

Charter: To evaluate the root cause for the melter to empty its contents, and develop Lessons Learned from melter operation and disassembly.

Deliverable:

1. Prepare Melter Inspection Plan.
2. Perform Failure Modes Analysis for root cause determination.
3. Develop Lessons Learned.
4. Prepare Final Report.

0001 63

January 3, 1997

SAFETY REVIEW TEAM

Co-Leaders: L. Bogar and R. Crawford

DOE Oversight: P. Darnell

Members: D. Nordquist (FDF), B. Previty (FDF), D. Daniels (FDF), B. Tabor (FATLC)

Charter: To evaluate appropriateness of the response to the incident in terms of procedures and processes used. Determine adequacy of engineering controls with regard to promoting safety during the event.

Deliverables:

1. Final Report including Lessons Learned and recommendations for further safeguards.

2. Evaluate Environmental Safety and Health implications in regard to Lessons Learned resulting from the Incident Analysis.

000144

Attachment 6 0 7

APPENDIX C

INCIDENT ANALYSIS TEAM

TEAM MEMBERS:

Ray Reinhart, Team Leader	Fluor Daniel Fernald
Joe Neyer, Oversight	Department of Energy -FEMP
Joe Desormeau, Oversight	Department of Energy - FEMP
Gail Bingham, Team Member	Consultant
Brad Bowan, Team Member	GTS Duratek
Joel Bradburne, Team Member	Fluor Daniel Fernald
Hamid Hojaji, Team Member	GTS Duratek
Vijay Jain, Team Member	West Valley
Ron Joseph, Team Member	Fluor Daniel Fernald
Xing Mao, Team Member	GTS Duratek
Jill Oligee, Team Member	Fluor Daniel Fernald
Donald Paine, Team Member	Fluor Daniel Fernald
Kareld Solomon, Team Member	Fluor Daniel Fernald
Ron Worsley, Team Member	Fluor Daniel Fernald

CONSULTANT:

Jim Edmondson	Consultant

FOR INFORMATION ONLY

APPROVAL:

1-14-97
Raymond H. Reinhart — Date
Team Leader

1-17-97
Donald Paine — Date
Silos Project Manager

1.0 PURPOSE

The purpose of this plan is to provide the instructions and inspection requirements for the VITPP furnace to support comprehensive Melter Drain Incident Analysis and to obtain samples required to determine the root cause of the failure. Disassembly and destructive testing are not restricted but shall be minimized to only the extent necessary (by the least intrusive methods readily available) to perform failure mode analysis and root cause determination.

2.0 SCOPE

This plan will involve detailed disassembly and inspection work on the vitrification furnace system. Supporting sampling and data collection (identified in Table 1, Incident Analysis Sampling and Analytical Minimum Requirements) will also be performed to fulfill the purpose of this inspection.

3.0 PREREQUISITES

3.1 Operation Work Instructions (OWI) will need to be developed to implement the requirement of this plan. Additionally, all sampling identification in Table # 1 will be collected in accordance with site and project procedures and requirements.

3.2 Lockouts have been performed to isolate energy sources to the melter.

3.3 Video/Media services are required to support this activity. Narration shall be performed at the time the video and film data are gathered when practicable.

3.4 All personnel performing Hazardous Energy and Material Control (FERMCO lockout tagout) must be qualified to the requirements of OP-0004 (revised).

3.5 Complete and comprehensive work plans shall be reviewed and approved prior to commencing work activities in accordance with approved maintenance and operations procedures.

3.6 All activities performed in support of sampling, inspections, examinations, disassembly, and maintenance shall be documented in the form of detailed plans, laboratory logbooks, OWI's, etc. and retained by the project for future analysis.

3.7 All red-lines and component condition/damage mapping shall be performed on drawings and maintained as a comprehensive source of this information.

3.8 Copies of all video and film used in the purposes of documentation for this instruction shall be considered attachments of this instruction and referenced in the procedure section at the time of collection to facilitate retrieval of the information when it is required.

3.9 Coordinate with Data Retrieval and Analysis Team to ensure sampling opportunities are not missed.

4.0 PRECAUTIONS AND LIMITATIONS

4.1 Any work which has the potential to disturb fiber type insulation on the interior of the furnace will require additional controls not specified in this document.

4.2 An adequate source of negative HEPA filtered ventilation (as determined by the system engineer or applicable work permits) is required to be available to support this activity.

4.3 The plan may temporarily suspend due to unexpected conditions or to support the requirements of other activities. The plan may recommence once the prerequisites have been reverified.

4.4 Ensure that the data required in Table #1 (Incident Analysis Sampling and Analytical Minimum Requirements) is entered at the time the sample is collected and noted on a sample grid to maintain traceability of relevant data.

4.5 Access into the melter box may involve special precautions for confined space entry as directed by Industrial Hygiene.

4.6 This plan does not circumvent any existing safety precautions, existing procedures and/or requirements.

4.7 All components and materials removed from the furnace shall be bagged and tagged indicating the date and time of removal, location, orientation (if applicable), and stored in the staging area provided by operations personnel.

4.8 Sampling performed in support of the melter inspection shall include an archive (duplicate) sample where practicable to be retained in accordance with the sampling plan.

4.9 Physiological monitoring of the work force shall be conducted as directed by Industrial Hygiene to minimize the possibility and extent of heat stress and other environmental hazards.

4.10 Proper chain of custody for sampling should be followed during the entire process.

5.0 PROCEDURE

5.1 Perform video and visual inspection of the exterior of the furnace. Pay particular attention to areas with damaged components and heat affected areas especially the bottom drains, bubblers, airlift, and thermowells. Document observations of the affected areas on the drawings.

NOTE: ANY ITEMS DISTURBED DURING THE VIDEO INSPECTION SHALL BE RECORDED AND LABELED IN ACCORDANCE WITH SECTION 4.7.

5.2 Perform boroscope inspection with video records of all melter wall and floor penetrations. Document observations on the drawings and catalogue.

5.3 Verify that the melter lid and H-brick have been removed. Catalogue the removal of the three superstructure (H-Brick) bricks. Record brick ID numbers on drawings, dimensions, weight, and location of any damage or deformities for each block on Attachment 6.1.

NOTE: DURING LID REMOVAL COLLECT SAMPLES OF ANY MATERIAL (INCLUDING THE H-BRICK) WHICH MAY HAVE MIGRATED TO THE INTERIOR INSULATION LAYERS IN THE VICINITY OF THE LID PENETRATIONS. ENSURE ANY SAMPLES COLLECTED ARE CATALOGUED.

5.4 Remove debris from beneath the furnace (ie. dead wires, burned thermocouples, cooling air and cooling water). Keep materials segregated and tagged in accordance with section 4.7.

5.5 Perform video and visual inspection of the interior of the furnace. Perform detailed inspection of individual superstructure, glass contact, E-wall refractories, bubblers, and airlift. Document damage, cracks, unusual appearances, and as found dimensions on drawings. Copies of all video shall be retained in accordance with section 3.8.

5.6 Collect information and samples in accordance with the detailed plans and the following schedule and catalogue:

5.6.1 NDE (video, visual, boroscope, XRF, and radiography etc.).

5.6.2 Loose samples (material which can be collected by non-intrusive means).

5.6.3 Samples available by non-destructive means once the interior of the furnace is accessible.

5.6.4 Samples which require intrusive or destructive methods (ie. core drilling, plasma cutting, or advanced furnace disassembly).

5.7 Perform the NDE, ultrasound, photo/film, conductivity, and other data required by the data analysis and/or accident analysis teams. Attach copies of the results as an addendum to this instruction.

5.8 Verify that all teams have gathered samples, data, and measurements required prior to further disassembly.

5.9 Inspect the interior of the discharge chamber. Pay particular attention for signs of degradation of the upper fiber board. Record the condition of the trough and riser blocks. Record any material samples and material evaluations in Attachment 6.4. Inspect/video the air lift through the crack in the riser block.

NOTE: OWI (DISCHARGE CHAMBER LID REMOVAL) MUST BE COMPLETED TO PERFORM THIS STEP.

5.10 Excavate and core drill as applicable bottom drain number spare and bubbler #1 (excavation shall be performed on these components first to provide a learning process which will ensure the highest possible sample quality when this operation is performed on bottom drain #2 and bubbler #3). Disassemble in accordance with CRU4-M-M-028 ensuring that all pieces of the assemblies are removed. Catalogue and tag each assembly and record measurements on Attachment 6.4. Samples of the surrounding material, components, and unusual observations shall be collected in accordance with the sampling plan and catalogued.

5.11 Remove bottom drain #2 and bubblers #2 through 5, in accordance with process developed in Step 5.10. Pay particular attention to bubblers #3 and 4.

NOTE: DISSECTION AND SAMPLE PREPARATION OF CORE DRILL SAMPLES IS TO BE PERFORMED ONLY BY THE ANALYTICAL LAB SO THAT IMPORTANT INFORMATION IS PRESERVED.

5.12 Obtain sample of bottom drain bimetallic weld area for tests of chloride stress cracking. Catalogue all information.

5.13 Inspect the varying layers of the penetrations around the bubblers and bottom drains. Record results of the inspection and video tape observation.

5.14 Verify that all teams have gathered samples, data, and measurements required prior to further disassembly. Determine if further melter disassembly will be required.

NOTE: FURTHER DISASSEMBLY IS NOT REQUIRED FOR THE PURPOSES OF THE MELTER INCIDENT ANALYSIS INVESTIGATION. FURTHER DISASSEMBLY MAY BE PERFORMED TO FACILITATE THE REQUIREMENTS OF OTHER DATA ANALYSIS, BUT SHOULD NOT PROCEED WITHOUT THE APPROVAL FROM THE PROJECT MANAGER.

CAUTION: CERTAIN TYPES OF NDE MAY REQUIRE SPECIAL PERMITS OR SAFETY PRECAUTIONS WHICH ARE NOT COVERED UNDER THIS INSTRUCTION.

5.15 Catalogue the removal of the Glass Contact Brick (K-3 and E-Wall). Record brick ID number, dimensions, weight, and location of any damage or deformities for each block on Attachment 6.2.

5.16 Excavate and core drill bottom drains #1 and #3.

5.17 Verify that all teams have gathered samples, data, and measurements required prior to further disassembly.

5.18 Catalogue the removal of the back-up refractory (AZS-Brick). Video, photo, and record brick ID number, dimensions, weight, and location of any damage or deformities for each block on Attachment 6.3.

5.19 Video, photo, and document the appearance, depth, and condition of the castable lining.

5.20 Verify that all teams have gathered samples, data, and measurements required prior to further disassembly.

5.21 Remove and inspect the inner shell if required.

6.0 ATTACHMENTS

6.1 Attachment 6.1 Superstructure Inspection Report

6.2 Attachment 6.2 Glass Contact Brick Inspection Report

6.3 Attachment 6.3 Back up Refractory Inspection Report

6.4 Attachment 6.4 Melter Component Inspection Report

6.5 Video and Film Collection for Melter Incident Analysis (as performed)

6.6 Drawing Package With Red Line Comments (as performed)

6.7 Miscellaneous Examination Reports (as performed)

7.0 TABLES

Table 1

Incident Analysis Sampling and Analytical Minimum Requirements

Table 2

Glass Analysis

Attachment 6.1
SUPERSTRUCTURE INSPECTION REPORT

BLOCK #	MEASURED DIM.	APPEARANCE / CONDITION	WEIGHT	Sample Required? Y/N

000153

Attachment 6.2
GLASS CONTACT INSPECTION REPORT

BLOCK #	MEASURED DIM.	APPEARANCE / CONDITION	WEIGHT	Sample Required? Y/N

Attachment 6.3
BACK UP REFRACTORY INSPECTION REPORT

BLOCK #	MEASURED DIM.	APPEARANCE / CONDITION	WEIGHT	Sample Required? Y/N

000155

Attachment 6.4
MELTER COMPONENT INSPECTION REPORT

NOUN NAME OF COMPONENT	Reference Drawing	Appearance/Condition	Dimensions / Weight	Sample of Material or residue taken? Y / N
Bubbler #1				
Bubbler #2				
Bubbler #3				
Bubbler #4				
Bubbler #5				
Airlift				
Bottom Drain # 1				
Bottom Drain # 2				
Bottom Drain # spare				
Bottom Drain # 3				
Feed Tube				
West Side Frit Feeder				
East Side Frit Feeder				
Discharge Chamber				

Table 1
INCIDENT ANALYSIS
SAMPLING AND ANALYTICAL MINIMUM REQUIREMENTS

SAMPLE	ANALYSIS	PROPOSED LAB	QUANTITY	PURPOSE	PRIORITY
Glass attached to bottom exterior at Bubbler #3	SEM/EDX Bulk Chemical Analysis	Vitreous State Lab Fluor Daniel Fernald	Total Available	Evaluate element presence and dispersion	HIGH
Material located on Bottom Drains 1, 2 & 3	SEM/EDX Bulk Chemical Analysis	University of Cincinnati Fluor Daniel Fernald	Total Available	Evaluate element presence and dispersion	HIGH
MoSi, Level Indicators	SEM/EDX Interface	University of Cincinnati	Total Available	Evaluate erosion, corrosion, chemical attack	HIGH
Glass from Bottom Drain Container	SEM/EDX Bulk Chemical Analysis Container X-Ray	University of Cincinnati Fluor Daniel Fernald Fluor Daniel Fernald	4 Core Borings	Evaluate erosion, corrosion, chemical attack	HIGH
Air lift tube	Visual Inspection	Fluor Daniel Fernald	None	Verify erosion	MEDIUM
Migration materials located on the interstitial layer f the melt roof assembly	SEM/EDX Interface	University of Cincinnati		Evaluate Material Defect Evaluation	LOW
Feed Tube	SEM/EDX	University of Cincinnati	Small section of weld failure	Defect Identification	LOW
Scrape Top 3 West Electrodes; Scrape Top 2 East Electrodes	SEM/EDX Bulk	University of Cincinnati	6 grams each electrode	Evaluate element presence and dispersion	MEDIUM
Surface Drain	Visual SEM/EDX if visual is not sufficient	Fluor Daniel Fernald University of Cincinnati	Total unit small section	Defect identification	LOW
Melter Center Chamber Floor	XRF, Ultra Sound Conductivity	Fluor Daniel Fernald	None	Verify metal present in refractory and electrical current path	HIGH
Melter penetrations	Borescope	Fluor Daniel Fernald	None	Visual inspection of existing condition	HIGH
Bubbler Assemblies	SEM/EDX Interface SEM/EDX Bulk	University of Cincinnati University of Cincinnati	Total Assembly	Defect identification Material identification	HIGH
Bottom Drains 1, 2, Spare & 3	SEM/EDX Interface SEM/EDX Bulk	University of Cincinnati University of Cincinnati	Total Assembly	Defect Identification Material Identification	HIGH
Molten Metallic Substance located on the exterior of Melter around bubblers and bottom drains			Total Available		HIGH

TABLE 2
GLASS ANALYSIS

ADDITIONAL INFORMATION NEEDED BY MELTER INCIDENT ANALYSIS TEAM WHICH MAY AID IN THE DETERMINATION OF THE ROOT CAUSE. THIS ANALYIS WILL BE PERFORMED ON SAMPLES THAT WERE PREVIOUSLY COLLECTED DURING PHASE I TESTING.

Type of Sample	Campaign 1	Campaign 2				Campaign 4	
	Run 1	Run 1	T	Run 2	Post Campaign 2 Requested Slurry	Run 1	T
Redox	N/A	B,E	B	M,E	E	B,M,E	E
SEM/EDX	E	E		E	E	E	E
Chem. Anal	E	E		E	E	E	E
Samples from sides chambers when possible							

Explanation of abbreviations:

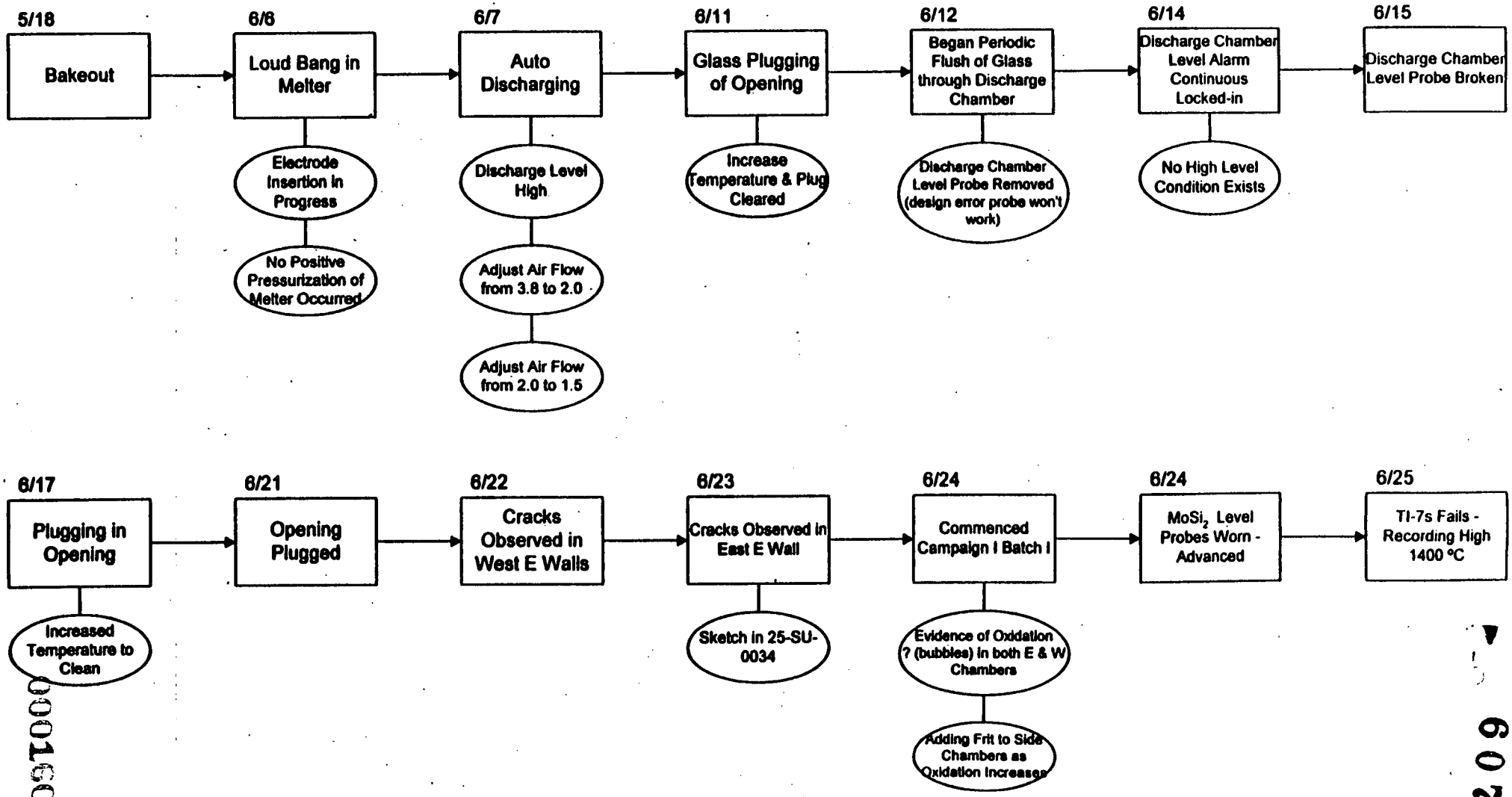
B = Beginning of run

M = Middle of run

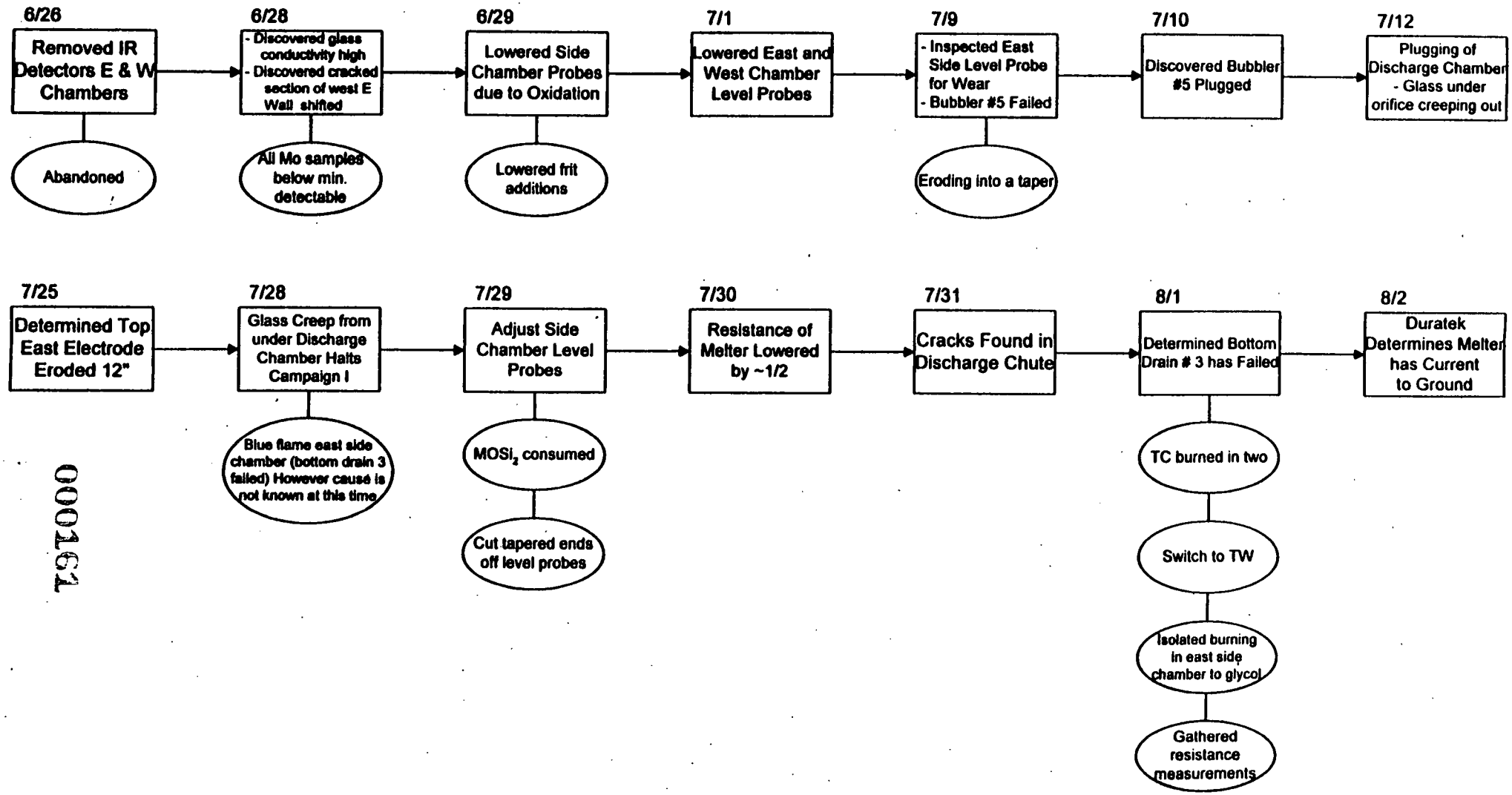
E = End of run

N/A = Not Applicable

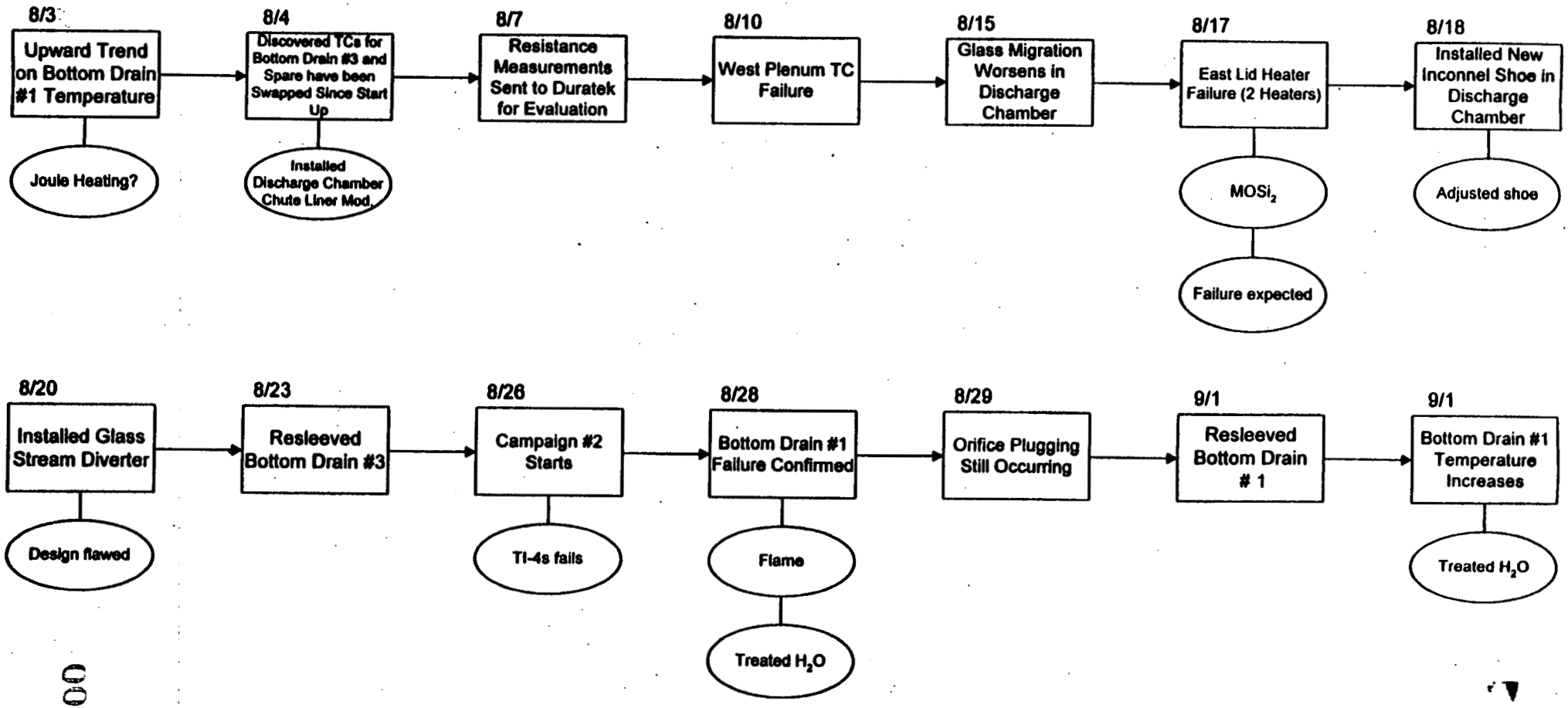
APPENDIX D


5/18
Bakeout
6/6
Loud Bang in Melter
Electrode Insertion in Progress
No Positive Pressurization of Melter Occurred
6/7
Auto Discharging
Discharge Level High
Adjust Air Flow from 3.8 to 2.0
Adjust Air Flow from 2.0 to 1.5
6/11
Glass Plugging of Opening
Increase Temperature & Plug Cleared
6/12
Began Periodic Flush of Glass through Discharge Chamber
Discharge Chamber Level Probe Removed (design error probe won't work)
6/14
Discharge Chamber Level Alarm Continuous Locked-in
No High Level Condition Exists
6/15
Discharge Chamber Level Probe Broken
6/17
Plugging in Opening
Increased Temperature to Clean
6/21
Opening Plugged
6/22
Cracks Observed in West E Walls
6/23
Cracks Observed in East E Wall
Sketch in 25-SU-0034
6/24
Commenced Campaign I Batch I
Evidence of Oxidation ? (bubbles) in both E & W Chambers
Adding Frit to Side Chambers as Oxidation Increases
6/24
MoSi2 Level Probes Worn - Advanced
6/25
TI-7s Fails - Recording High 1400 °C

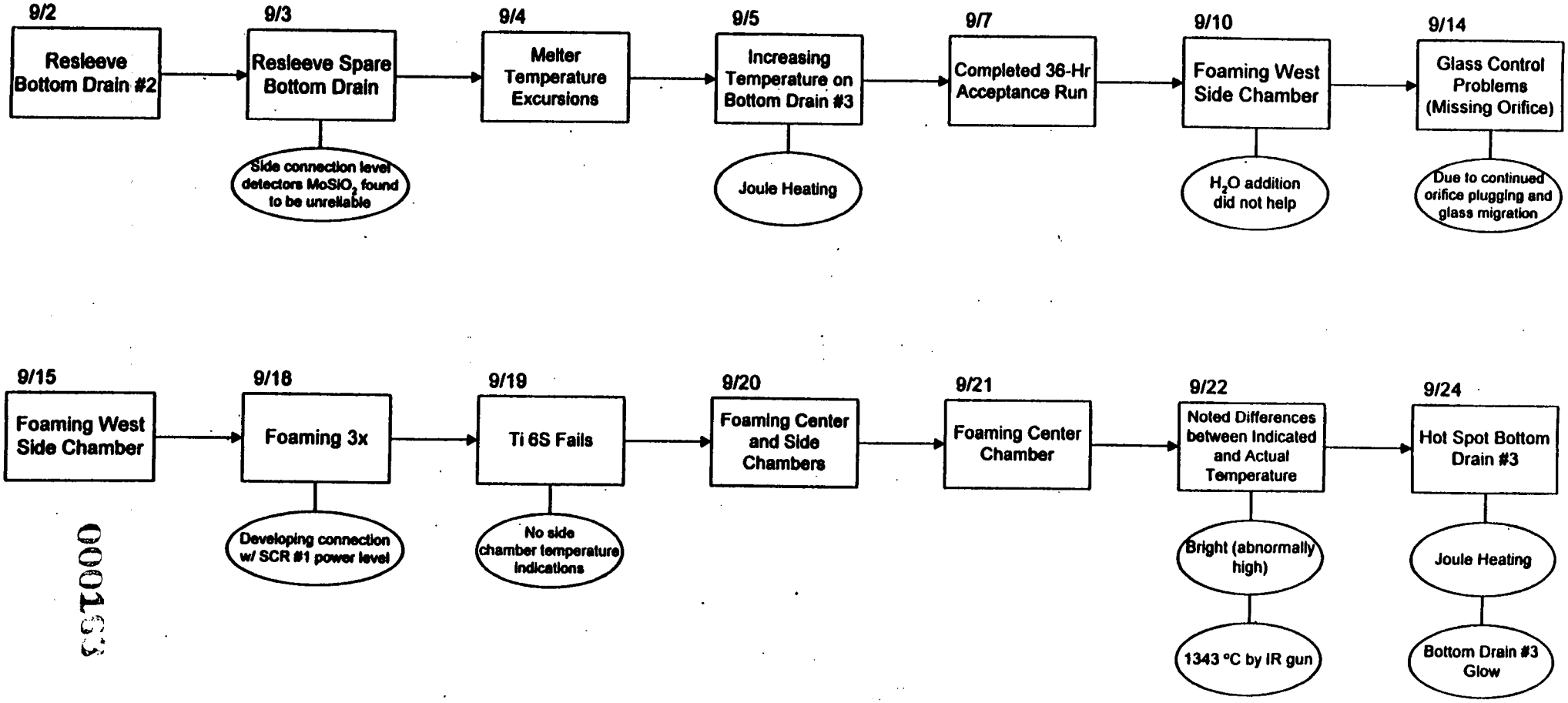
0001160


6/26
Removed IR Detectors E & W Chambers
Abandoned
6/28
- Discovered glass conductivity high
- Discovered cracked section of west E Wall shifted
All Mo samples below min. detectable
6/29
Lowered Side Chamber Probes due to Oxidation
Lowered frit additions
7/1
Lowered East and West Chamber Level Probes
7/9
- Inspected East Side Level Probe for Wear
- Bubbler #5 Failed
Eroding into a taper
7/10
Discovered Bubbler #5 Plugged
7/12
Plugging of Discharge Chamber - Glass under orifice creeping out
7/25
Determined Top East Electrode Eroded 12"
7/28
Glass Creep from under Discharge Chamber Halts Campaign I
Blue flame east side chamber (bottom drain 3 failed) However cause is not known at this time
7/29
Adjust Side Chamber Level Probes
MOSi2 consumed
Cut tapered ends off level probes
7/30
Resistance of Melter Lowered by ~1/2
7/31
Cracks Found in Discharge Chute
8/1
Determined Bottom Drain # 3 has Failed
TC burned in two
Switch to TW
Isolated burning in east side chamber to glycol
Gathered resistance measurements
8/2
Duratek Determines Melter has Current to Ground

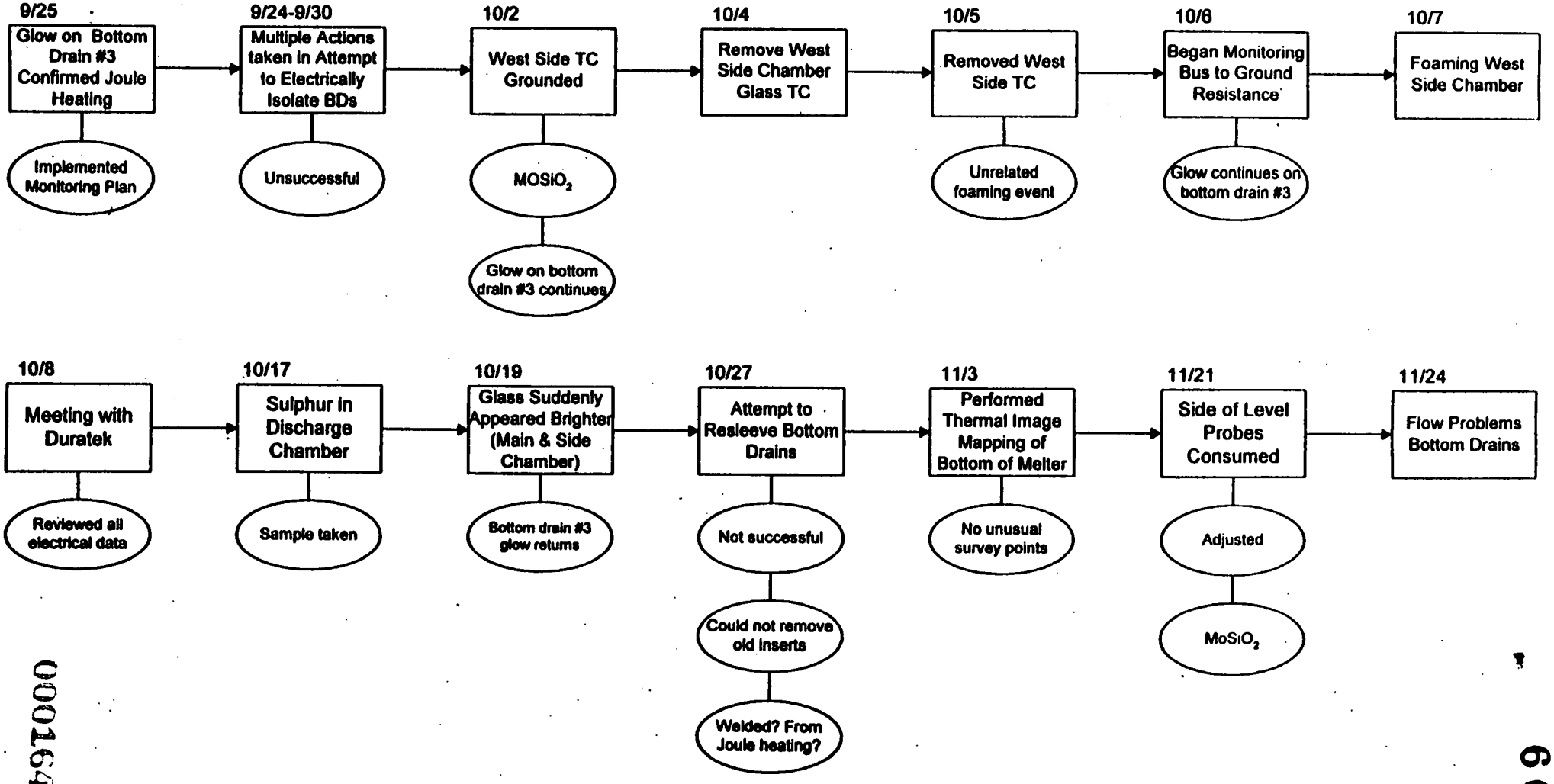
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8/3
Upward Trend on Bottom Drain #1 Temperature
Joule Heating?
8/4
Discovered TCs for Bottom Drain #3 and Spare have been Swapped Since Start Up
Installed Discharge Chamber Chute Liner Mod.
8/7
Resistance Measurements Sent to Duratek for Evaluation
8/10
West Plenum TC Failure
8/15
Glass Migration Worsens in Discharge Chamber
8/17
East Lid Heater Failure (2 Heaters)
MOSi2
Failure expected
8/18
Installed New Inconnel Shoe in Discharge Chamber
Adjusted shoe
8/20
Installed Glass Stream Diverter
Design flawed
8/23
Resleeved Bottom Drain #3
8/26
Campaign #2 Starts
TI-4s fails
8/28
Bottom Drain #1 Failure Confirmed
Flame
Treated H2O
8/29
Orifice Plugging Still Occurring
9/1
Resleeved Bottom Drain # 1
9/1
Bottom Drain #1 Temperature Increases
Treated H2O

000162


9/2
Resleeve Bottom Drain #2
9/3
Resleeve Spare Bottom Drain
Side connection level detectors $MoSiO_2$ found to be unreliable
9/4
Melter Temperature Excursions
9/5
Increasing Temperature on Bottom Drain #3
Joule Heating
9/7
Completed 36-Hr Acceptance Run
9/10
Foaming West Side Chamber
H_2O addition did not help
9/14
Glass Control Problems (Missing Orifice)
Due to continued orifice plugging and glass migration
9/15
Foaming West Side Chamber
9/18
Foaming 3x
Developing connection w/ SCR #1 power level
9/19
Ti 6S Fails
No side chamber temperature indications
9/20
Foaming Center and Side Chambers
9/21
Foaming Center Chamber
9/22
Noted Differences between Indicated and Actual Temperature
Bright (abnormally high)
1343 °C by IR gun
9/24
Hot Spot Bottom Drain #3
Joule Heating
Bottom Drain #3 Glow

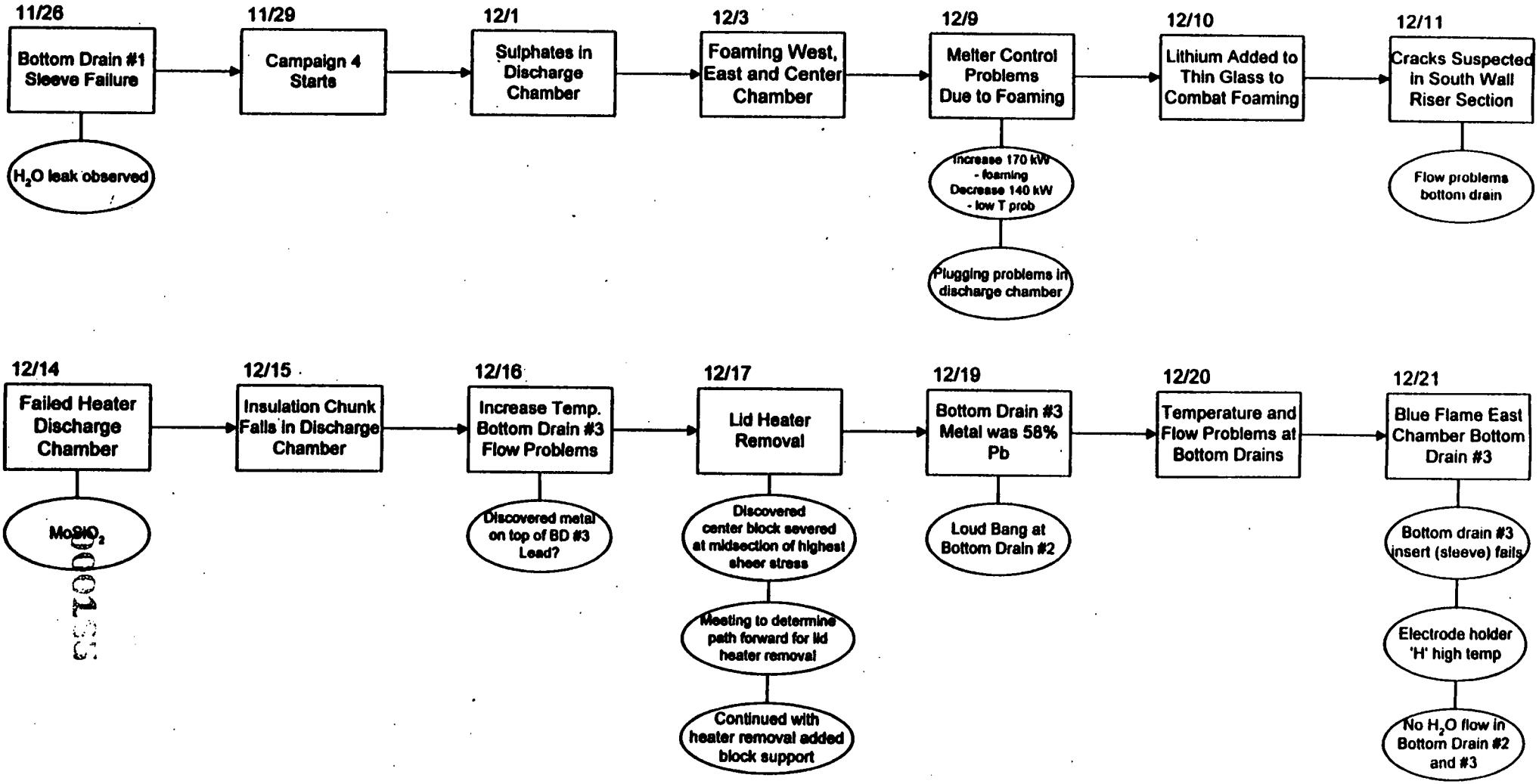
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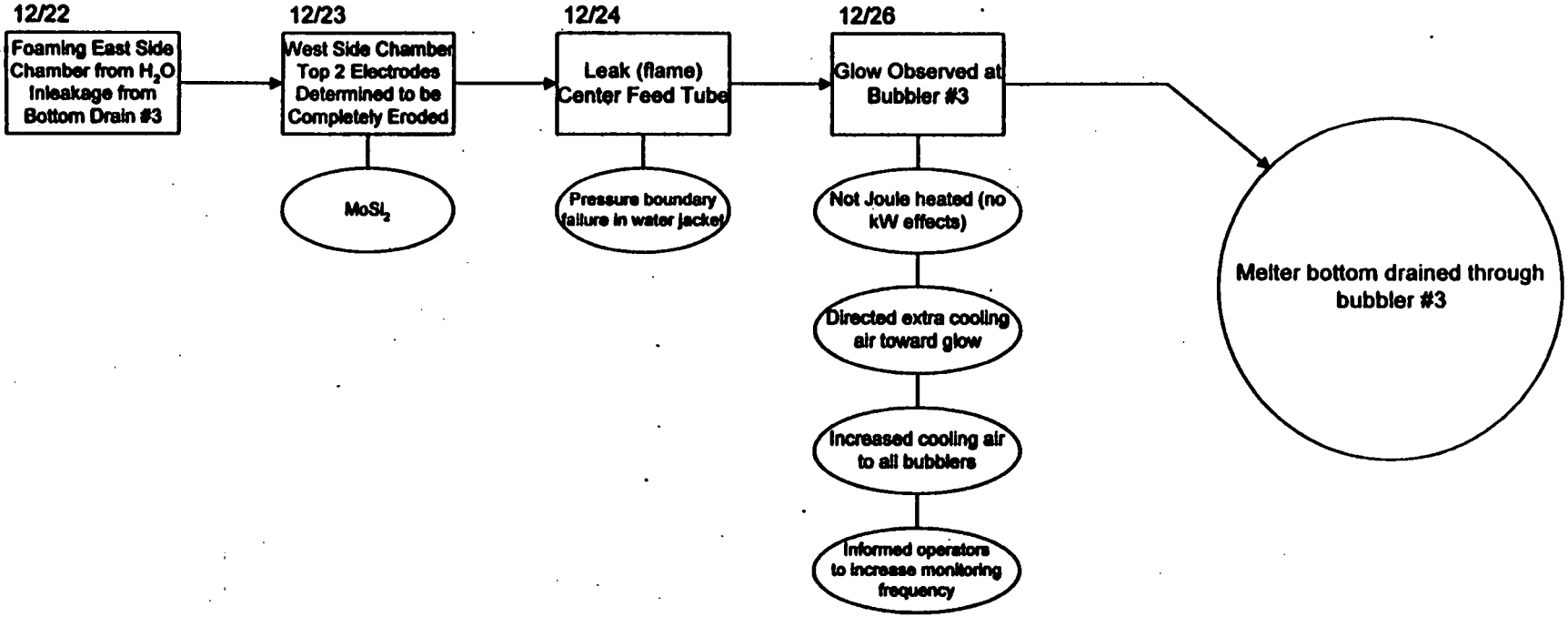
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Appendix D-5

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11/26
Bottom Drain #1 Sleeve Failure
H_2O leak observed
11/29
Campaign 4 Starts
12/1
Sulphates in Discharge Chamber
12/3
Foaming West, East and Center Chamber
12/9
Melter Control Problems Due to Foaming
Increase 170 kW - foaming Decrease 140 kW - low T prob
Plugging problems in discharge chamber
12/10
Lithium Added to Thin Glass to Combat Foaming
12/11
Cracks Suspected in South Wall Riser Section
Flow problems bottom drain
12/14
Failed Heater Discharge Chamber
$MoSiO_2$
12/15
Insulation Chunk Falls in Discharge Chamber
12/16
Increase Temp. Bottom Drain #3 Flow Problems
Discovered metal on top of BD #3 Lead?
12/17
Lid Heater Removal
Discovered center block severed at midsection of highest sheer stress
Meeting to determine path forward for lid heater removal
Continued with heater removal added block support
12/19
Bottom Drain #3 Metal was 58% Pb
Loud Bang at Bottom Drain #2
12/20
Temperature and Flow Problems at Bottom Drains
12/21
Blue Flame East Chamber Bottom Drain #3
Bottom drain #3 insert (sleeve) fails
Electrode holder 'H' high temp
No H_2O flow in Bottom Drain #2 and #3

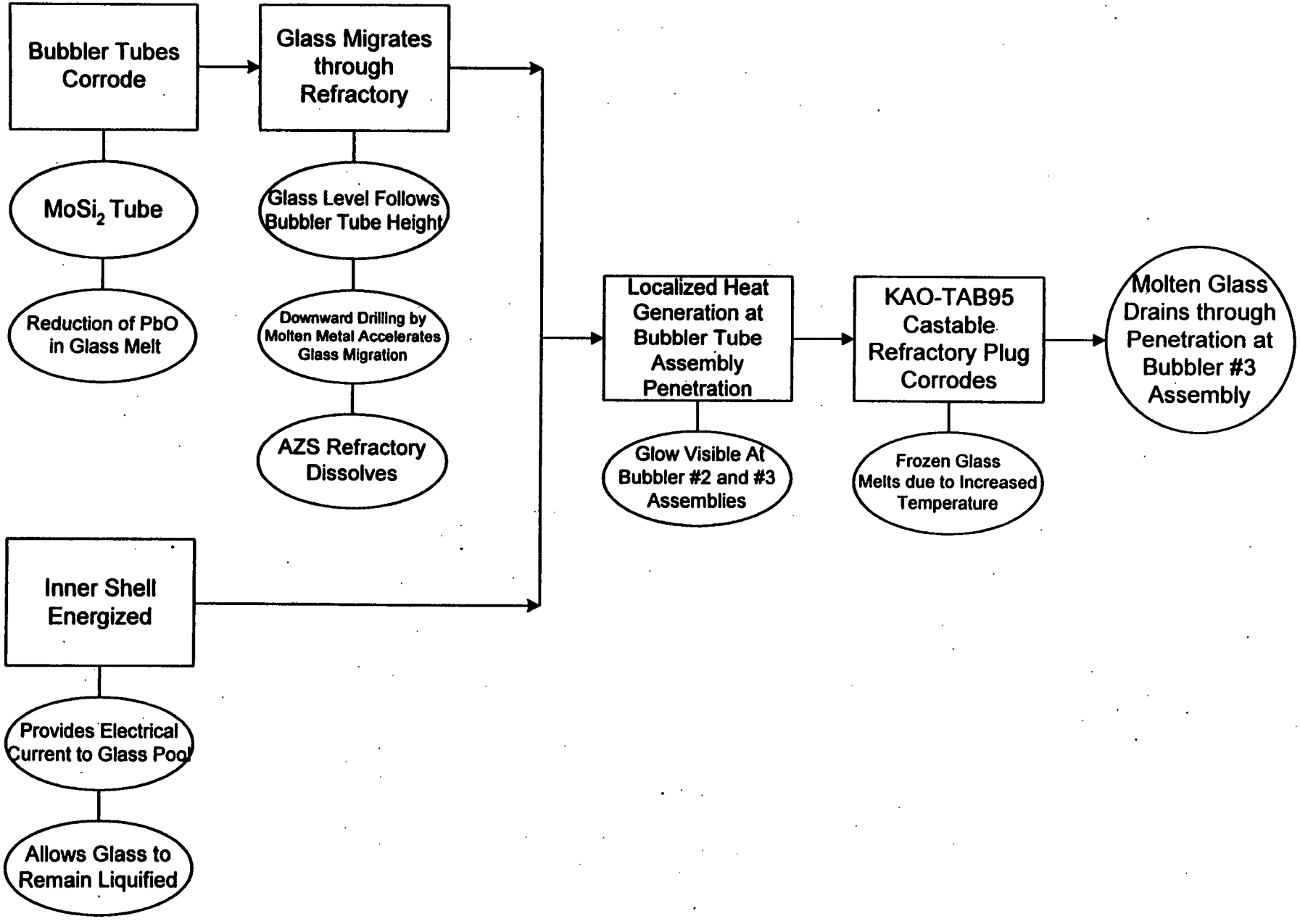
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12/22
Foaming East Side Chamber from H_2O Inleakage from Bottom Drain #3
12/23
West Side Chamber Top 2 Electrodes Determined to be Completely Eroded
$MoSi_2$
12/24
Leak (flame) Center Feed Tube
Pressure boundary failure in water jacket
12/26
Glow Observed at Bubbler #3
Not Joule heated (no kW effects)
Directed extra cooling air toward glow
Increased cooling air to all bubblers
Informed operators to increase monitoring frequency
Melter bottom drained through bubbler #3

000166

APPENDIX E

Appendix E
Event and Causal Factor Chart for Melter Failure



89008

APPENDIX F

North American Refractories Co.

EXECUTIVE OFFICES
500 Halle Building
1228 Euclid Avenue
Cleveland, Ohio 44115-1901
(216) 621-5200

Brand: ZIRMUL
Class: ZIRCON BEARIN BRICK/

TYPICAL TEST DATA

PHYSICAL PROPERTIES

Bulk Density, lb/ft3 - g/cm3	194 - 3.08
Apparent Porosity, %	18
Modulus of Rupture, lb/in² - MPa @ 70°F - 21°C	2000 - 14
Reheat Change, % @ 3092°F - 1700°C	+ 0.6
Reversible Linear Thermal Expansion, % in/in/°F @ 80° - 2700° cm/cm/°C @ 27° - 1482°	3.89 x 10-6
Hot Load Deformation, % @ 2900°F - 1593°C	0.12
Thermal Conductivity Btu in/ft2hr °F (W/m.K)	13 - 1.88

CHEMICAL ANALYSIS, WT. %

Al2O3	70.0	ZrO2	19.5
SiO2	10.2	Others	0.3

The data given above are based on averages of test results on samples selected from routine plant production by standard A.S.T.M. procedures where applicable. Variation from the above data may occur in individual tests. These results cannot be taken as minima or maxima for specification purposes.

11/94

NORTH AMERICAN REFRACTORIES COMPANY
8361 Broadwell Rd., Cincinnati, OH 45244
FAX: 513-388-2112 - PHONE: 513-388-2100

DATE: 31 January, 1997

TO: Dennis Nixon Fax: 513-648-4850

ATTN: Ford Fluor Daniel

FROM: Jackie Camden

TOTAL NUMBER OF PAGES INCLUDING THIS COVER SHEET 2

Per your telephone request, attached please find our technical data sheet on ZIRMUL.

Should you have any questions or require any additional information, please don't hesitate to contact us.

Regards,

Jackie Camden

Jackie Camden
Marketing Assistant

Att.

Unique Monofrax® refractory properties can handle your most demanding applications

TABLE OF MONOFRAX® REFRACTORY PROPERTIES

Property	Units	Type Designation	Fused-Cast Aluminas A-1	M	H
Max. Hot Face Temperature[1]	°C °F		1930 3500	1870 3400	1930 3500
Modulus of Rupture ASTM C133	MPa @ 20°C (psi @ 70°F) MPa @ 1350°C (psi @ 2460°F)		416/5800 —	251/3500 —	108/1500 —
Bulk Density ASTM C134	g/cm³ lb/ft³		3.77 235	3.40 212	3.13 195
Porosity ASTM C20	%		2.0	1.9	4.0
Hot-Load Contraction 50 hrs at 50 psi (3.5 kg/cm²) @ 1500°C (2730°F) ASTM C546	% of length		0.0 @ 3400°F	0.0 @ 3400°F	0.0 @ 3400°F
Thermal Expansion Coefficient (mean)	1/°C x 10^{-6} (20°–1400°C) 1/°F x 10^{-6} (68°–2552°F)		7.9 4.4	7.9 4.4	6.4 3.6
Thermal Conductivity ASTM C202 at mean temperature shown	W/m°K @ 1477°K BTU in./hr ft² °F @ 2200°F		7.2 50	4.8 33	3.5 24
Specific Heat 0–1400°C (2550°F)	cal/gm°C		0.28	0.28	0.28
Reheat Change ASTM C113 5 hrs @ 1664°C (3030°F)	%		-0.1	0.0	-0.06
Abrasion Resistance Index[2]			0.2	0.5	2.0
Compressive Strength ASTM C133	MPa psi		1434 20,000	1291 18,000	574 8000
Permeability @ 25°C (70°F)	cm³/min of air flow for: 1 in. H_2O press., 1 in.² area, 1 in. thkns.		0.0	0.0	0.0
Chemical Analysis Core Chemistry	SiO_2		0.7	0.8	0.3
	Al_2O_3		98.2	94.5	93.3
	Fe_2O_3		0.1	0.1	0.1
	CaO		0.1	0.2	0.1
	TiO_2		—	—	—
	Na_2O		0.7	3.8	5.9
	MgO		—	—	—
	Cr_2O_3		—	—	—
	ZrO_2		—	—	—
	Other		0.2	0.6	0.3

Notes to tables

It is our policy to be conservative in publishing all properties, such as recommended service temperature limits for refractory materials. In part, this reflects the following facts:

1. Maximum hot face temperature is seldom an isolated property for which a refractory is selected. Maximum hot face temperatures listed are based on these installation conditions:
   One hot face—the material is heated on one side only.
   Dry heat—no contact with corrosive fumes, slags or molten metals.

Magnesia-Alumina Spinels / Chrome-Alumina Spinels				Alumina-Zirconia Silica				
L	K-3	E	B	CS-3	CS-4	CS-5	R	Z
1950 3542	1870 3400	1930 3500	1930 3500	1590 2900	1590 2900	1590 2900	1590 2900	1870 3400
— —	574/8000 —	402/5600 —	394/5600 —	703/9800 —	624/8700 —	570/7950 —	703/9800 —	— —
— —	3.83 239	4.25 265	4.57 285	3.70 231	3.78 236	3.94 246	3.70 231	5.29 330
—	4.2	5.4	5	1.0	0.6	1.0	1.0	0.7
—	0.0 @ 3000°F	0.0 @ 3000°F for 5 hours	—	0.0 @ 25 psi @ 2600°F	0.0 @ 25 psi @ 2600°F	0.0 @ 25 psi @ 2600°F	0.0 @ 25 psi @ 2600°F	-2.0 @ 28 psi @ 3400°F
— —	7.9 4.4	7.4 4.1	2.4 4.4	7.3 4.1	8.0 4.5	7.9 4.4	7.3 4.1	— —
— —	2.9 20	4.6 32	— —	2.9 20	3.0 21	3.5 24	2.9 20	— —
—	0.25	0.25	—	0.24	0.24	0.24	0.24	—
—	0.2	-0.2	—	0.0 @ 2550°F	0.0 @ 2550°F	0.0 @ 2550°F	0.0 @ 2550°F	-3.3 @ 2732°F
—	0.3	0.3	—	0.3	0.3	0.3	0.3	—
— —	1434 20,000	1434 20,000	— —	1434 20,000	1434 20,000	1434 20,000	1434 20,000	— —
—	0.0	0.0	—	0.0	0.0	0.0	0.0	—
0.4	1.6	1.8	—	15.3	14.0	12.9	15.3	3.6
53.6	58.6	6.5	13.7	49.7	47.8	45.8	49.7	0.6
—	6.1	5.9	—	0.1	0.1	0.1	0.1	0.2
0.3	—	—	—	0.3	0.2	0.2	0.3	—
—	—	—	—	0.1	0.1	0.1	0.1	0.4
0.2	0.3	0.2	—	1.1	1.1	1.1	1.1	—
44.9	6.1	9.4	1.5	—	—	—	—	—
—	27.1	77.7	82.2	—	—	—	—	—
—	—	—	—	33.0	36.4	39.5	33.0	94.0
—	6.3	7.9	4.1	0.4	0.3	0.3	0.4	1.2

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An atmosphere—or an equivalent gas that is essentially neutral to slightly oxidizing

Low pressure—minimal pressure to drive corrosive material into the pores of the refractory

Normal shock and unusual amounts of vibration either thick or thin

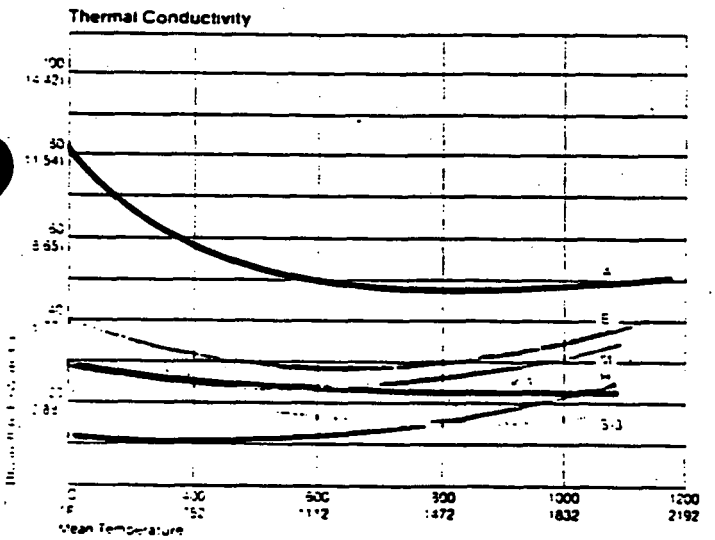
Light load—material that supports only its own weight

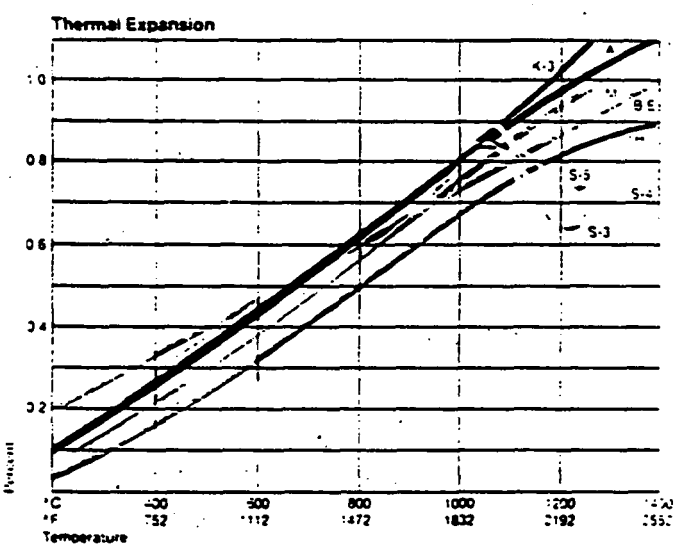
Continuous duty—mass specified limits can be exceeded for short bursts if other operating conditions are not severe

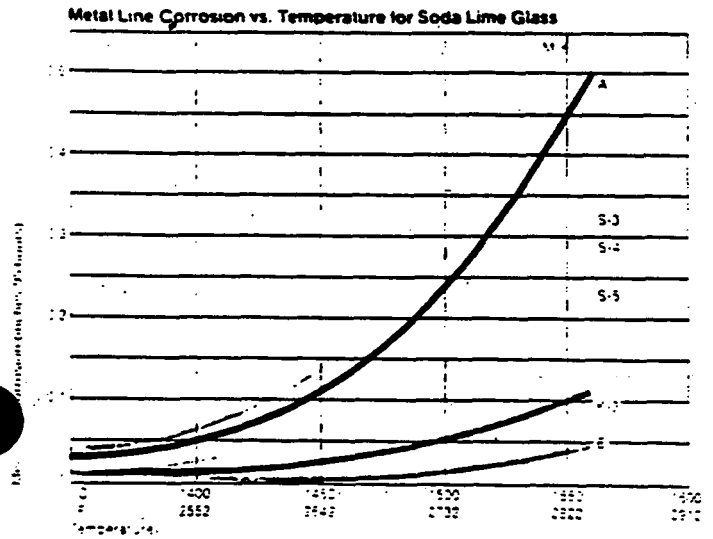
Moderate heat shock—freedom from repeated, rapid temperature changes

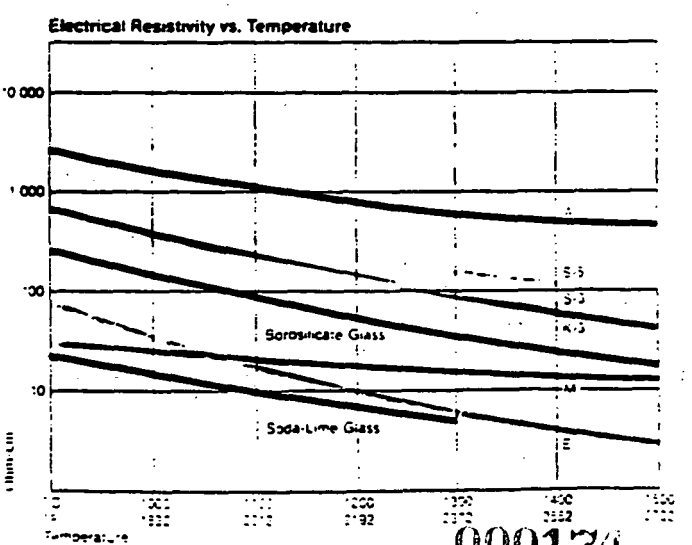
2. Brick samples positioned radially on 20" diameter turntable. A new standard 1" diameter sandblast nozzle is positioned 1" away from surface of brick samples and set to impinge at 90° angle. Electrite fused alumina #30 grain is fed at air pressure of 60 psi. Turntable is rotated at 12 rpm for 200 revolutions. Weight loss of samples due to abrasion is converted by calculation to volume loss and reported as an abrasion index in cubic inches of loss. Carborundum #80. Other types of abrasion are often encountered; consultation with our engineers is advised in abrasion applications.

Monofrax Refractory Type	Typical Chemical Composition (%) Al_2O_3	ZrO_2	Cr_2O_3	SiO_2	Alkali and Accessory Oxides	Typical Physical Properties Bulk Density lb/ft³(g/cm³)	Apparent Porosity (%)
S-3. Conditioned	49.7	33.0	—	15.3	2.0	231 (3.70)	1.0
S-4. Conditioned	47.8	36.4	—	14.0	1.8	236 (3.78)	1.0
S-5. Conditioned	45.8	39.5	—	12.9	1.8	246 (3.94)	1.0
A	98.2	—	—	0.7	1.1	235 (3.76)	2.0
M	94.5	—	—	0.8	4.7	212 (3.40)	2.0
H	93.3	—	—	0.3	6.4	195 (3.12)	4.0
K-3	58.6	—	27.1	1.6	12.7	239 (3.83)	4.0
E	6.5	—	77.7	1.7	14.1	260 (4.17)	5.0
B	13.7	—	82.2	0.8	3.3	285 (4.56)	5.0









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Appendix F-5

THERMAL CERAMICS
MATERIAL SAFETY DATA SHEET

MSDS GROUP: 101

PRODUCT IDENTIFICATION

GROUP A:

→ **KAO-TAB 93**
KAO-TAB 95
KAO-TAB 95G
KAO-TAB SR
KAOLITE 3300
KAOLITE 3300 RFT
KAO-TAB 93 RFT
KAO-TAB 95 RFT
KAO-TAB 95G RFT
KAOCRETE HDHS 98
KAOCRETE HDHS 98 RFT

GROUP B:

INSUL-MIX RFT
INSUL-MIX
SR-99 DRY MORTAR

SECTION I

Manufacturer's Name:
THERMAL CERAMICS
P.O. BOX 923
AUGUSTA, GEORGIA 30903

Product Stewardship Program:
(800) 722-5681

Chemical Name and Synonyms:
N/A Mixture

Chemical Family:
Refractory Concrete
[CAS # 66402-68-4]

SECTION II - HAZARDOUS INGREDIENTS

A. As Manufactured:	WT. %	PEL
Aluminum Oxide [CAS #1344-28-1]	**75 to 98**	**10 mg/m³ Total Dust**
Nuisance Dust	**2 to 25**	**5 mg/m³ Respirable Dust 15 mg/m³ Total Dust**

B. After Normal Use: **See Section IX**

SECTION III - PHYSICAL DATA

Boiling Point: **N/A**
Vapor Pressure (mm Hg.): **N/A**
Vapor Density (Air = 1): **N/A**
Solubility in Water: **Insoluble**
Specific Gravity Range (H20 = 1): **0.9 - 3.1**
Percent Volatile by Volume (%): **N/A**
Evaporation Rate: **N/A**
Appearance and Odor: **Coarse aggregate with fine powdered matrix. No odor.**

SECTION IV - FIRE AND EXPLOSION HAZARD DATA

Extinguishing Media: **N/A**
Flammable Limit: **N/A**
Unusual Fire and Explosive Hazards: **N/A**
Special Fire Fighting Procedures: **N/A**
Flash Point (Method Used): **N/A**
LEL: **N/A**
UEL: **N/A**

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MSDS GROUP: 101

THERMAL CERAMICS
MATERIAL SAFETY DATA SHEET

SECTION V - HEALTH HAZARD DATA

Primary Route of Entry: **Inhalation, ingestion, skin contact**
Effects of Overexposure: **Exposure to nuisance dust may cause temporary irritation or discomfort to the skin, eyes, nose, throat or lungs and may aggravate bronchial disorders. These products do not appear on any NTP or IARC lists/reports of carcinogens.**

Emergency and First Aid Procedures:
Inhalation: **Remove to fresh air.** Ingestion: **Drink plenty of water.**
Skin: **Wash with mild soap and water.** Eyes: **Flush with plenty of water. If irritation persists, call a physician.**

SECTION VI - REACTIVITY DATA

Stability: **Stable** Hazardous Polymerization: **Will not occur.**
Materials/Conditions to Avoid: **None**

SECTION VII - SPILL OR LEAK PROCEDURES

Spill or Leak: **Follow routine housekeeping procedures. Collect spilled product in closed container.**
Waste Disposal: **Wastes are not hazardous wastes as defined by RCRA (40 CFR 261). Comply with Federal, State and local regulations.** Method of Disposal: **Landfill. RQ - N/A.**

SECTION VIII - SPECIAL PROTECTION INFORMATION

Respiratory Protection: **Dust respirator in compliance with OSHA Standard 29 CFR 1910.134 (MSHA/NIOSH-Approved, air purifying, half mask or full facepiece respirator with appropriate filter pad or cartridge).**

Ventilation:	**Local exhaust**	**Follow OSHA Standard 29 CFR 1910.94**
	Mechanical (General)	**Follow OSHA Standard 29 CFR 1910.94**
Protective Gloves:	**Recommended**	
Eye Protection:	**Goggles/safety glasses recommended**	

Other Protective Equipment: **As required to meet applicable OSHA Standards.**

SECTION IX - SPECIAL PRECAUTIONS

Precautions To Be Taken After Service and Upon Removal:
Group A: **Removal of htese products after service may generate respirable nuisance dust.**

Group B: **As manufactured, these products are aluminosilicates which may transform upon heating to mullite and cristobalite (a form of crystalline silica). Removal of these products after service may generate dust. Repeated inhalation of respirable free crystalline silica dust may cause delayed lung injury (silicosis). IARC has placed crystalline silica in Category 2A (IARC believes there is sufficient evidence of carcinogenicity in animals but evidence for the carcinogenicity to humans is limited). The recommended PEL and TLV for respirable cristobalite is 0.05 mg/m³.**

Groups A & B: **Appropriate ventilation and respiratory protection should be provided in compliance with OSHA Standards 29 CFR 1910.94 and 1910.134, respectively.**

Revision: Deleted Kaolite Fill. Now on MSDS #104.
Label: No. 3-1191

PRODUCT SAFETY INFORMATION

RESPIRABLE NUISANCE DUST

CAUTION: Handling this product may generate respirable nuisance dust which can cause eye and respiratory tract irritation.

Avoid breathing particulates and dust.

RISKS:

- May cause temporary irritation to eyes, skin and respiratory tract.

PRECAUTIONARY MEASURES:

- Minimize airborne dust with engineering controls.
- Use NIOSH/MSHA approved respiratory protection when airborne dusts exceed recommended exposure limits.
- Wear long sleeved loose-fitting clothing, eye protection, gloves.

FIRST AID MEASURES:

Eyes: flush with water. **Skin:** wash with soap and warm water. **Ingestion:** do not induce vomiting. Get medical attention if gastrointestinal symptoms develop. **Inhalation:** remove to fresh clean air.

If any of the above irritations persists seek medical attention immediately.

FOR ADDITIONAL PRODUCT INFORMATION AND WORK PRACTICES REFER TO THE MATERIAL SAFETY DATA SHEET (MSDS).

THERMAL CERAMICS
P. O. BOX 923 MS 300
AUGUSTA, GA 30903 USA
(800) 722-5681

No. 3-1191

11/20/91

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(216) 621-5200

Boiling Point (°F): Greater than 1000 Deg.F.
Solubility in Water: Nil.
% Volatile (by weight): Not applicable.
pH: 5 to 9 range
Density: 2.5 to 3.4 gm/cc range
Coefficient of Water/Oil Distribution: Not available.

********** SECTION IV - FIRE AND EXPLOSION HAZARD **********

Flammability: This product is non-flammable and will not support combustion.

********** SECTION V - HEALTH HAZARD **********

Threshold Limit Value:

For dust containing Zirconium compounds:
OSHA PEL.....5mg/m3
OSHA STEL.....10mg/m3

For respirable dust containing crystalline silica:
OSHA: For Respirable Dust:
10 divided by (% Quartz + 2)
expressed in mg/m3.

ACGIH: Quartz.....0.1mg/m3
Cristobalite....0.05mg/m3
Tridymite.....0.05mg/m3

For all ingredients not listed above:
OSHA:.....10mg/m3 total dust
ACGIH:.....10mg/m3 total dust

Effects of Overexposure: Chronic exposure to dust could cause pulmonary problems.

CRYSTALLINE SILICA:
Chronic overexposure to dust containing respirabl sized crystalline silica (quartz, cristobalite, and tridymite) can cause delayed lung injury (silicosis).
Points of attack: Respiratory system and lungs.
Route of entry: Inhalation.
Inhalation of dust containing crystalline silica may contribute to pre-existing pulmonary diseases such as asthma and lung disorders associated with the smoking of tobacco.

Some recent animal studies have caused the International Agency for Research on Cancer (IARC) to categorize crystalline silica as a 2A carcinogen. A 2A carcinogen is one which:

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(1) there is sufficient evidence for the carcinogenicity to experimental animals.
(2) there is limited evidence of the carcinogenicity to humans.

Crystalline silica has been listed in the most recent NTP Report on Carcinogens.

For more information on crystalline silica refer to:
(1) IARC Monograph, Volume 42
(2) NIOSH Document No. 75-120
(3) NTP Report on Carcinogens

Toxicity data:

Quartz:	LCLo:	300ug/m3 / 10Y-I inhalation human
Cristobalite:	TCLo:	16 mppcf/8H/17.9Y-I inhalation human
Tridymite:	TCLo:	16 mppcf/8H/17.9Y-I inhalation human

Note: LD50 and LC50 not available.

ZIRCONIUN SILICATES (zircon sands)
Zircon sands contain trace amounts (106-120 pCi/g of naturally occurring radioactive uranium and thorium. Overexposure by inhalation to respirable dust containing uranium and thorium may cause lung cancer. Eye contact with the dust may cause eye irritation.
Measurments made by Dupont during the use of a similar mineral sand indicated that the observance of the 5mg/m3 OSHA PEL for respirable dust and/or the PEL for quartz insure that the user is below the limits established for uranium and thorium.
No LD50 or LC50 can be found for zircon sand.

CARCINOGENICITY INFORMATION
With the exception of:
Crystalline silica
No ingredient in this product is found on either the Federal OSHA, NTP, or IARC list of carcinogens.

Emergency and First Aid Data:
Eyes: Flush with water and get medical help.
Skin: Wash thoroughly with soap and water.
Inhalation: Remove to fresh air.

********** SECTION VI - REACTIVITY DATA **********

Stability and Reactivity: This product is stable and non-reactive.
Hazardous Decomposition: Crystalline silica levels in used refractories may be higher or lower than as-shipped depend

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on service conditions. Hygiene monitoring must be done to insure the proper employee protection during tearout.

********** SECTION VII - SPILL AND LEAK PROCEDURES **********

Waste Disposal Method: Dispose of material according to local, state or federal regulations and as final used condition of the product dictates.

********** SECTION VIII - INDUSTRIAL HYGIENE INFORMATION *****

Ventilation: Local exhaust recommended to maintain exposures below TLV.
Respiratory Protection: NIOSH approved dust type for exposures above TLV.
Eye Protection: Recommended.

********** SECTION IX - SPECIAL PRECAUTIONS **********

Special Precautions: Dust created during demolition of used product may contain crystalline silica.

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NORTH AMERICAN REFRACTORIES
PRODUCT SAFETY DATA SHEET

MANUFACTURER

NORTH AMERICAN REFRACTORIES

MSDS # HT02-00

Vendor:

Emergency Phone Number
East (814) 236-3890
West (415) 432-4741
Canada (416) 765-4404

MSDS prepared by:
NORTH AMERICAN REFRACTORIES
Technical Center (814) 234-7981

Date Issued: 03/26/95 Date Revised: 01/27/94
Product Type: Refractory Plastic Or Ram Material
Trade Name: ZIRMUL 361 CEMENT
Product SARA Hazard Class: (2) Delayed or Chronic
WHMIS Hazard Class (CANADA): Class D - Division 2: Toxic Material (CPR 6

SECTION I - PRODUCT IDENTIFICATION

Chemical Name: Al2O3,ZrO2.SiO2
Chemical Family: Alumina Zircon Refractory

SECTION II - CHEMICAL COMPOSITION

Hazardous Ingredients:	CAS Number:	PCT:
Crystalline Silica including:	N/A	0.1 - 1.0
Quartz	14808-60-7	
Cristobalite	14464-46-1	
Tridymite	15468-32-3	

Other Ingredients:	CAS Number:	PCT:
Hydrous Alumina Silicate	12141-46-7	1.0 - 5.0%
Sugar binder	8061-52-7	1.0 - 5.0%
Zirconium Silicate	10101-52-7	10.0 - 30.0%
Alumina (non-fibrous)	1344-28-1	40.0 - 70.0%

SECTION III - PHYSICAL DATA

Appearance and Odor: Orange, fine grained dry mixture with a sweet odor.
Odor threshold (p.p.m.): Not available.

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Specific Gravity: Not available.
Vapor Pressure (mm): Not applicable.
Vapor Density (air=1): Not applicable.
Boiling Point (°F): Not available.
Solubility in Water: Not available.
% Volatile (by weight): 1 to 3% at 1800 Deg.F.
pH: 5 to 9 range
Density: Not available.
Coefficient of Water/Oil Distribution: Not available.

********** SECTION IV - FIRE AND EXPLOSION HAZARD **********

Flammability: This product is non-flammable and will not support combustion.

********** SECTION V - HEALTH HAZARD **********

Threshold Limit Value:

For dust containing Zirconium compounds:
OSHA PEL:.....5mg/m3
OSHA STEL.....10mg/m3

For respirable dust containing crystalline silica:
OSHA: For Respirable Dust:
10 divided by (% Quartz + 2)
expressed in mg/m3.

ACGIH: Quartz.....0.1mg/m3
Cristobalite....0.05mg/m3
Tridymite.....0.05mg/m3

For all ingredients not listed above:
OSHA:.....10mg/m3 total dust
ACGIH:.....10mg/m3 total dust

Effects of Overexposure: Chronic exposure to dust could cause pulmonary problems.

CRYSTALLINE SILICA:
Chronic overexposure to dust containing respirable sized crystalline silica (quartz, cristobalite, and tridymite) can cause delayed lung injury (silicosis).
Points of attack: Respiratory system and lungs.
Route of entry: Inhalation.
Inhalation of dust containing crystalline silica may contribute to pre-existing pulmonary diseases such as asthma and lung disorders associated with the smoking of tobacco.

Some recent animal studies have caused the Inter-

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national Agency for Research on Cancer (IARC) to categorize crystalline silica as a 2A carcinogen. A 2A carcinogen is one which:

(1) there is sufficient evidence for the carcinogenicity to experimental animals.
(2) there is limited evidence of the carcinogenicity to humans.

Crystalline silica has been listed in the most recent NTP Report on Carcinogens.

For more information on crystalline silica refer to:

(1) IARC Monograph, Volume 42
(2) NIOSH Document No. 75-120
(3) NTP Report on Carcinogens

Toxicity data:

Quartz:	LCLo:	300ug/m3 / 10Y-I inhalation human
Cristobalite:	TCLo:	16 mppcf/8H/17.9Y-I inhalation human
Tridymite:	TCLo:	16 mppcf/8H/17.9Y-I inhalation human

Note: LD50 and LC50 not available.

ZIRCONIUN SILICATES (zircon sands)
Zircon sands contain trace amounts (106-120 pCi/g) of naturally occurring radioactive uranium and thorium. Overexposure by inhalation to respirable dust containing uranium and thorium may cause lung cancer. Eye contact with the dust may cause eye irritation.
Measurments made by Dupont during the use of a similar mineral sand indicated that the observance of the 5mg/m3 OSHA PEL for respirable dust and/or the PEL for quartz insure that the user is below the limits established for uranium and thorium.
No LD50 or LC50 can be found for zircon sand

CARCINOGENICITY INFORMATION
With the exception of:
Crystalline silica
No ingredient in this product is found on either the Federal OSHA, NTP, or IARC list of carcinogens.

Emergency and First Aid Data:
Eyes: Flush with water and get medical help.
Skin: Wash thoroughly with soap and water.
Inhalation: Remove to fresh air.

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********** SECTION VI - REACTIVITY DATA **********

Stability and Reactivity: This product is stable and non-reactive. Organic binder will decompose at 350 deg. F.
Hazardous Decomposition: May generate SO2 fumes. Crystalline silica levels in used refractories may be higher or lower than as-shipped depending on service conditions. Hygiene monitoring must be done to insure the proper employee protection during tearout.

********** SECTION VII - SPILL AND LEAK PROCEDURES **********

Waste Disposal Method: Dispose of material according to local, state or federal regulations and as final used condition of the product dictates.

********** SECTION VIII - INDUSTRIAL HYGIENE INFORMATION ****

Ventilation: Local exhaust recommended to maintain exposures below TLV.
Respiratory Protection: NIOSH approved dust type for exposures above TLV.
Eye Protection: Recommended.

********** SECTION IX - SPECIAL PRECAUTIONS **********

Special Precautions: Dust created during demolition of used product may contain crystalline silica.

000184

APPENDIX G

Campaign 1

Benign Glass Formula

Batch No: Startup Glass — v2.0 rfg — 03/28/00

Sample No: HHH

Oxide	mol wt	Sample data, wt%	Sample wt%, a	Sample - gases	Left Chem wt%	ght Chem wt%	Waste in	Left Chem Leakage	ght Chem Leakage	Additives oxides, wt%	Glass Result	Glass Target	Delta	Additives dry, wt%	Moisture wt%	Additives hydra, wt%	Additives Batch, lbs
Al2O3	101.96	6.41	6.75	6.75	12.70	12.70	4.91			6.00	10.61	10.62	-0.01	6.00		6.00	133
B2O3	69.62									10.66	10.66	10.66					
H3BO3	61.83																
BaO	153.34																
CO2	44.01	10.35	10.87				7.03										
CaO	56.08	5.06	6.20	6.20	6.40	6.40	4.66			10.00	14.66	14.66	0.00				
CaCO3	100.09													17.86		17.86	398
Cr2O3	151.99																
Fe2O3	159.69																
K2O	94.20																
Li2O	29.88				10.10	10.10											
Li2CO3	73.89																
MgO	40.31				4.10	4.10											
MnO2	86.94																
N2O5	108.01																
Na2O	61.98	7.09	6.30	6.30	6.30	6.30	6.12			6.40	14.62	14.63	-0.01				
Na2B4O7	201.22													15.29	30.60	22.13	497
Na2CO3	105.99													6.31		6.31	139
NiO	74.71																
P2O5	141.94																
PbO	223.19																
SrO	90.06																
SO3 in glass	80.06	na	na														
SiO2	60.08	59.31	62.27	62.27	56.30	56.30	46.43				45.43	45.41	0.02				
V2O5	181.88																
ZrO2	123.22	6.22	6.48	6.48	6.10	6.10	4.00				4.00	4.00	-0.00				
Urea	60.06																
Others	na																
a	1.00																
b	1.00																
H2O																	1,180
Total	n/a	99.34	100.00	99.13	100.00	100.00	72.66	0.00	0.00	34.06	100.00	100.00	0.00	45.45	na	52.29	2,368

	Input		Output		Output		Input						Output
SO3 in glass	0.50	wt%		est. factor							Mass in	Additives, hydrated	1,180
Urea addition	50.00	% stoich.										Waste, dry	1,004
Particulate carryover	0.00	wt%										Chamber inleakage	
Suspended solid	40.00	wt%										Water	2,784
Dissolved solids	10.00	wt%										Total feed	5,067
Total solids			50.00	wt%	1,004	lbs solids							
Feed moisture			50.00	wt%	1,004	lbs water					Mass out	Glass	2,180
Slurry density	1.40	g/ml										Particulate carryover	11
Feed density	1.40	g/ml										Gases	494
Slurry in mix tank							Use One:					Water	2,383
weight wet	3,200	lbs	3,200	lbs			1	Enter "1" to use					5,067
volume wet	200	gal	3,005	lbs	3.04%	diff		Enter "0" not to use					
after additives			440	gal approx.									

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Table V
Appendix G-1

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Campaign 2

Transitions Formula from Benign to Acceptance Glass

First LAB Component	mol. wt.	Initial Glass	Initial Glass normalized	Portions norm.	Portions in glass	Additives as oxides	Glass Result	Target C4D-15	Delta	Additives mol%	Glass mol%
Al_2O_3	101.96	10.92	10.92	10.92	9.83	1.94	11.77	18.13	-3.36	1.26	7.65
As_2O_3	197.84										
B_2O_3	69.62	10.69	10.69	10.69	9.62	0.02	10.44	10.08	0.36	0.00	0.04
BaO	153.34					0.49	0.49	2.48	-1.99	0.21	0.21
BeO	25.01										
CO_2	44.01							released			
CaO	56.08	14.66	14.66	14.66	13.19		13.19	7.79	5.40		16.46
Ce_2O_3	328.24										
CoO	74.93										
Cr_2O_3	151.99										
CuO	79.54										
Fe_2O_3	159.69										
K_2O	94.20					1.16	1.16	5.59	-4.43	0.48	0.48
La_2O_3	325.82					0.23	0.23	1.10	-0.87	0.16	0.16
Li_2O	29.88					0.05	0.05	0.16	-0.16	0.07	0.07
MgO	40.31					1.04	1.04	0.69	-3.99	1.71	1.71
MnO_2	86.94										
MoO_2	127.94										
N_2O_5	108.01										
Na_2O	61.98	14.63	14.63	14.63	13.09			released			
Nd_2O_3	336.48						13.09	7.01	6.07		13.09
NiO	74.71										
P_2O_5	141.94					0.07	0.07	0.40	-0.33	0.06	0.06
PbO	223.19					0.79	0.79	3.87	-3.08	0.37	0.37
SO_3	80.06					1.02	1.02	4.86	-3.83	0.30	0.30
SeO_2	110.96										
SiO_2	60.08	46.41	46.41	46.41	41.67	2.31	43.18	36.56	6.62	2.64	47.83
SrO	103.62										
ThO_2	264.04										
TiO_2	79.89										
UO_2	270.03										
V_2O_3	149.88										
Y_2O_3	225.81										
ZnO	81.37										
ZrO_2	123.22	4.00	4.00	4.00	3.60		3.60	0.06	3.54		1.94
Cl	35.45										
F	19.00										
Ignition loss								released			
SO_3 in glass	80.06										
Other A	1.00										
Other B	1.00										
Total	nb	100.00	100.00	100.00	90.00	10.00	100.00	98.00	0.02	6.00	100.00

0001827

Table
Appendix G-2

Compe[illegible]2

Acceptance Glass Recipe

Batch Quantities for: Acceptance Glass: C4D-45 or RB-01 — Rod F. Gimpel V2.6 — 08/19/96

Compound	Common Name	%Moisture	Silo 1 Surrogate	Silo 2 Surrogate	Silo 3 Surrogate	Accept. G. Adjust.	Surrogate Total	Additives Mix Tank	Batch Total	As Oxides	Glass Makeup	Glass Makeup, %	Off Gas Makeup	
Al2O3	Alumina	5.71	15	15	35		65	275	339	339	339	15.20		
										226	226	10.14		
B2O3														
H3BO3	Boric acid										86	2.60		
BaO														
BaCO3	Barium carbonate		22				22		22	16				
BaSO4	X-ray millshield	1.17	39	28			90		90	97				145
CO2														
CaO	Lime			3			3		3		163	7.30		
CaCO3	Chalk, calcite	0.72		19			19	212	231	129				
CaSO4	Gypsum	30.83		2	66	25	66		66	54				
Fe2O3	Rust		16	36	31	34	113		113	110	121	5.62		
Fe2(SO4)3	Iron sulfate, III	1.09			13		13		13	6				
K2O										1	24	1.09		
K2CO3	Potassium carbonate	6.66	7	6			12	6	18	11				
KNO3	Salt peter	2.19			18	8	26		26	12				
Li2O											2	0.10		
Li2CO3	Lithium carbonate	6.66			6		6		6	2				
MgO						17	17		17	21	108	4.66		
MgCO3	Magnesium carbonate		6	15			21		21	16				
MgSO4	Epsom salt	61.16			66		66		66	0				
Mg3(PO4)2	Magnesium phosphate		7	7	78	52	144		144	66				
MnO2					3		3		3	3	3	0.14		35
N2O5														
Na2O										3	163	6.94		
Na2B4O7	Borax	36.66						476	476	161				
Na2SO4	Sodium sulfate				20		20		20	0				
Na2CO3	Washing soda	3.63	20	6	14		38	11	49	28				
NaNO3	Soda niter	0.71		6	28		34		34	12				
NiO	Nickel oxide				2		2	0	0	0	0	0.40		
P2O5										79	79	3.66		
PbO	Lead oxide		74	34			107		107	107	107	4.66		106
SO3														
SiO2	Silica, sand, quartz		267	270	94	113	760		760	611	611	30.66		
TiO2										0	0	0.01		
V2O5	Vanadium pentoxide				3		3		3	3	3	0.11		
Bentonite		14.29					80		80	–	–	–	–	–
CO(NH2)2	Urea							76	76					76
SO3 in glass	Sulfate									0	0	0.00		
Other A														
Other B														
H2O	Water								–	–				223
			616	446	488	286	1,736	1,000	2,766	2,366	2,366	100.00		585

Batch, lbs glass: 2,366

** K2CO3 and NiO adjusted per VSL's request in the Additives Column

Table V
Appendix G-3

0000188

Campaign 2

Series D Formula

Batch Quantities for: Series D per C4D-01 (VSL) or AG-10 (FERMCO) — Rod F. Gimpel V2.6 — 03/22/96

Compound	Common Name	%Moisture	Silo 1 Surrogate	Silo 2 Surrogate	Silo 3 Surrogate	Surrogate Total	Additives Mix Tank	Batch Total	As Oxides	Glass Makeup	Glass Makeup, %	Off Gas Makeup
Al2O3	Alumina		17	20	26	64	224	288	302	302	13.71	
B2O3									224	224	10.19	
H3BO3	Boric acid									60	2.73	
BaO									19			
BaCO3	Barium carbonate		25			25		25				
BaSO4	X-ray milkshake		35	27		62		62	41			287
CO2												
CaO	Lime			3		3		3	5	298	13.53	
CaCO3	Chalk, calcite			20		20	460	480	269			
CaSO4	Gypsum	6.20		1	60	62		62	24			
Fe2O3	Rust		17	38	34	87		87	60	96	4.35	
Fe2(SO4)3	Iron sulfate, III				14	14		14	6			
K2O									1	18	0.83	
K2CO3	Potassium carbonate		7	6		13		13	9			
KNO3	Salt peter				19	19		19	9			
Li2O										25	1.13	
Li2CO3	Lithium carbonate				6	6	55	61	25			
MgO									5	73	3.32	
MgCO3	Magnesium carbonate		9	14		23		23	11			
MgSO4	Epsom salt				31	31		31	10			
Mg3(PO4)2	Magnesium phosphate		8	7	86	101		101	46			
MnO2					3	3		3	3	3	0.15	
N2O5												34
Na2O									3	150	6.84	
Na2B4O7	Borax	30.80					469	469	100			
Na2SO4	Sodium sulfate				21	21		21	9			
Na2CO3	Washing soda		21	6	14	40	2	42	26			
NaNO3	Soda niter			6	32	37		37	14			
NiO	Nickel oxide				3	3		3	3	3	0.12	
P2O5									54	54	2.47	
PbO	Lead oxide		81	37		117		117	117	117	5.33	
SO3												86
SiO2	Silica, sand, quartz		338	302	70	707		707	763	763	34.66	
TiO2									0	0	0.01	
V2O5	Vanadium pentoxide				3	3		3	3	3	0.13	
Bentonite		14.23				87		87	--	--	--	--
CO(NH2)2	Urea						28	28				28
SO3 in glass	Sulfate								11	11	0.51	
Other A												
Other B												
H2O	Water								--	--		192
			555	484	422	1,660	1,239	2,798	2,200	2,200	100.00	626
[illegible] glass			2,200									

0001839

Campaign 4

Transition Formula from Benign to Series A

Formula Description:		Change Benign to Series A – 12-Lift Approach (12th Lift)								Rod F. Gimpel V2.6		11/27/96
Component	mol. wt.	Startup	Startup n	Portions norm.	Portions in glass	Additives as oxides	Glass Result	Target C4A-03	Delta	Additives mol%	Glass mol%	Additives Ratio
Al2O3	101.96	6.27	6.27	6.27	5.65	0.35	5.99	5.89	0.10	0.22	3.78	2.20
As2O3	197.84											
B2O3	69.62	12.85	12.85	12.85	11.56	1.50	13.06	12.89	0.17	1.36	12.06	9.50
BaO	153.34	4.06	4.06	4.06	3.66	0.67	4.22	4.47	-0.25	0.24	1.77	3.60
BeO	25.01											
CO2	44.01							released				
CaO	56.08	5.52	5.52	5.52	4.97	0.20	5.17	5.08	0.09	0.23	5.92	1.25
CdO	128.40											
Ce2O3	328.24											
CoO	74.93											
Cr2O3	151.99	0.03	0.03	0.03	0.02		0.02		0.02		0.01	
CuO	79.54											
Fe2O3	159.69	3.87	3.87	3.87	3.48	0.51	3.99	3.98	0.01	0.21	1.61	3.25
K2O	94.20	0.75	0.75	0.75	0.68	0.06	0.74	0.61	0.13	0.04	0.50	0.40
La2O3	325.82											
Li2O	29.88	1.28	1.28	1.28	1.16	0.09	1.25	1.52	-0.27	0.20	2.69	0.60
MgO	40.31	1.32	1.32	1.32	1.19	0.14	1.33	1.32	0.01	0.23	2.12	0.90
MnO2	86.94	0.01	0.01	0.01	0.01		0.01		0.01		0.00	
MoO2	127.94	0.03	0.03	0.03	0.03		0.03		0.03		0.02	
N2O5	108.01							released				
Na2O	61.98	5.94	5.94	5.94	5.35	0.41	5.76	5.68	0.08	0.42	5.97	2.00
Nd2O3	336.48											
NiO	74.71	0.45	0.45	0.45	0.40	0.06	0.47	0.38	0.09	0.05	0.40	0.40
P2O5	141.94	0.65	0.65	0.65	0.58	0.09	0.66	0.63	0.06	0.04	0.31	0.60
PbO	223.19	8.35	8.35	8.35	7.52	1.18	8.70	8.63	0.07	0.34	2.50	7.50
SO3	80.06	0.76	0.76	0.76	0.68	0.11	0.79	1.62	-0.83		0.63	0.70
SeO2	110.96											
SiO2	60.08	47.66	47.66	47.66	43.06	4.72	47.79	47.21	0.58	5.06	51.11	30.00
SrO	103.62											
ThO2	264.04											
TiO2	79.90											
UO2	270.03											
V2O3	149.88	0.01	0.01	0.01	0.01		0.01		0.01		0.00	
Y2O3	225.81											
ZnO	81.37											
ZrO2	123.22							0.09	-0.09			
Cl	35.45											
F	19.00											
Ignition loss								released				
SO3 in glass	80.06											
Other A	1.00											
Other B	1.00											
Total	n/a	100.00	100.00	99.24	88.32	10.89	99.21	100.00	-0.80	8.66	90.78	63.89

Table V
Appendix G-5

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Campaign 4

Series A Formula

Batch Quantities for: Series A per C4A-03 (w/boric acid) Rod F. Gimpel V2.7 12/02/96

Compound	Common Name	%Moisture	Silo 1 Surrogate	Silo 2 Surrogate	Silo 3 Surrogate	Surrogate Total	Additives Mix Tank	Batch Total	As Oxides	Glass Makeup	Glass Makeup, %	Off Gas Makeup
Al2O3	Alumina		29	33		62	71	133	133	133	6.05	
B2O3										290	13.20	
H3BO3	Boric acid						516	516	290			
BaO										99	4.52	
BaCO3	Barium carbonate		41			41		41	32			
BaSO4	X-ray milkshake		58	45		103		103	68			
CO2												255
CaO	Lime			6		6		6	6	110	5.00	
CaCO3	Chalk, calcite			33		33	153	186	104			
CaSO4	Gypsum	6.20		2		2		2	1			
Fe2O3	Rust		28	60		88		88	88	88	3.99	
Fe2(SO4)3	Iron sulfate, III											
K2O										14	0.65	
K2CO3	Potassium carbonate		11	10		21		21	14			
KNO3	Salt peter											
Li2O										34	1.53	
Li2CO3	Lithium carbonate						83	83	34			
MgO										30	1.36	
MgCO3	Magnesium carbonate		16	23		39		39	16			
MgSO4	Epson salt											
Mg3(PO4)2	Magnesium phosphate		13	12		26		26	12			
MnO2												
N2O5												6
Na2O										128	5.80	
Na2B4O7	Borax	30.80										
Na2SO4	Sodium sulfate											
Na2CO3	Washing soda		34	9		43	169	212	124			
NaNO3	Soda niter			10		10		16	3			
NiO	Nickel oxide											
P2O5									14	14	0.62	
PbO	Lead oxide		133	61		194		194	194	194	8.82	
SO3												26
SiO2	Silica, sand, quartz		558	498		1,056		1,056	1,056	1,056	47.96	
TiO2												
V2O5	Vanadium pentoxide											
Bentonite		14.23										
CO(NH2)2	Urea								–	–	–	–
SO3 in glass	Sulfate						18	18				18
Other A									11	11	0.51	
Other B												
H2O	Water							–	–			243
			910	802		1,720	1,010	2,730	2,200	2,200	100.00	548
Ba[redacted] glass:		2,200			[redacted]							

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Table V
Appendix C.6

Campaign 4

Modified Series A Formula

Batch Quantities for: Series A per C4A-03 modified or S12-04 — Rod F Gimpel V2.6

Compound	Common Name	%Moisture	Silo 1 Surrogate	Silo 2 Surrogate	Silo 3 Surrogate	Surrogate Total	Additives Mix Tank	Batch Total	As Oxides	Glass Makeup	Glass Makeup, %	04/06/98 Off Gas Makeup
Al2O3	Alumina		26	30		56	79	135	135	135	6.15	
B2O3									231	231	10.61	
H3BO3	Boric acid											
BaO										90	4.09	
BaCO3	Barium carbonate		37			37		37	29			
BaSO4	X-ray milkshake		63	40		63		93	61			
CO2												330
CaO	Lime			6		6		6	6	293	13.31	
CaCO3	Chalk, calcite			30		30	483	513	287			
CaSO4	Gypsum	6.20		2		2		2	1			
Fe2O3	Rust		26	54		78		78	78	78	3.60	
Fe2(SO4)3	Iron sulfate, III											
K2O										13	0.58	
K2CO3	Potassium carbonate		10	9		19		19	13			
KNO3	Salt peter											
Li2O										28	1.28	
Li2CO3	Lithium carbonate						70	70	28			
MgO										27	1.23	
MgCO3	Magnesium carbonate		14	21		35		35	17			
MgSO4	Epson salt											
Mg3(PO4)2	Magnesium phosphate		12	11		23		23	10			
MnO2												
N2O5												
Na2O												6
Na2B4O7	Borax	30.80								161	7.33	
Na2SO4	Sodium sulfate						484	484	103			
Na2CO3	Washing soda		31	8		39	55	94	55			
NaNO3	Soda niter			8		8		8	3			
NiO	Nickel oxide											
P2O5									12	12	0.56	
PbO	Lead oxide		121	55		176		176	176	176	7.98	
SO3												33
SiO2	Silica, sand, quartz		603	482		984		984	984	954	43.38	
TiO2												
V2O5	Vanadium pentoxide											
Bentonite		14.23										
CO(NH2)2	Urea								--	--	--	--
SO3 in glass	Sulfate						7	7				7
Other A												
Other B												
H2O	Water								--	--		186
			831	725		1,556	1,178	2,734	2,200	2,200	100.00	540
Batch lbs glass			2,200									

Table V
Appendix G-7

0001192

Campaign 2
Lead & Barium Tracking

Table V
Pb & Ba Introduced into the VitPP System

BATCH	Lead (lbs) as PbO	Barium (lbs) as $BaSO_4$	Barium (lbs) as $BaCO_3$	Lbs Frit Added	Lbs Lead Introduced	Lbs Barium Introduced
					0.0	0.0
					0.0	0.0
					0.0	0.0
					0.0	0.0
					0.0	0.0
					0.0	0.0
					0.0	0.0
					0.0	0.0
					0.0	0.0
					0.0	0.0
					0.0	0.0
					0.0	0.0
					0.0	0.0
					0.0	0.0
					0.0	0.0
					0.0	0.0
					0.0	0.0
					0.0	0.0
					0.0	0.0
					0.0	0.0
					0.0	0.0
					0.0	0.0

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Total Lead introduced into system: 0.0

Total Barium introduced into system: 0.0

Campaign 2
Lead & Barium Tracking: Glass - Straight Analysis

Table V
p1 of 2
Pb & Ba Retained in Glass

User Sample I.D.	DRUM ID	FACTS I.D.	Concentration (wt%)		Lbs. of Glass Produced	Total Weight (lbs)	
			Pb as PbO	Ba as BaO		Lead	Barium
S10-960827-0030	C2B01D1		1.23		781	8.9	0.0
S10-960827-1149	C2B01D2		3.62		751	25.2	0.0
S10-960827-1205	C2B01D3		3.36		841	26.2	0.0
S10-960827-1241	C2B01D4		3.36		<20	0.0	0.0
S10-960906-1303	C2B02D1		3.10		869	25.0	0.0
S10-960906-1307	C2B02D2		3.20		303	9.0	0.0
S10-960906-1312	C2B02D3		2.20		293	6.0	0.0
S10C-960907-0300	C2B03D1		3.41		457	14.5	0.0
S10-960906-1030	C2B03D2		3.60		150	5.0	0.0
S10C-960907-0305	C2B03D3		3.34		144	4.5	0.0
S10-960906-0602	C2B04D3		3.40		1036	32.8	0.0
Not Sampled	C2B04D4		3.50		723	23.5	0.0
S10-960912-0833	C2B05D1		3.72		339	11.7	0.0
S10-960912-1010	C2B05D2		3.81		677	23.9	0.0
S10-960912-1023	C2B05D3		3.80		1134	40.0	0.0
S10-960914-2345	C2B06D1		3.70		352	12.1	0.0
S10-960915-0010	C2B06D2		3.47		988	31.2	0.0
S10-960914-2350	C2B06D3		4.01		282	9.8	0.0
S10-960915-0001	C2B06D4		3.77		750	26.2	0.0
S10-960920-0009	C2B07D1		3.52		756	24.7	0.0
S10-960916-0230	C2B07D2		3.41		801	25.4	0.0
S10-960920-0006	C2B07D5		2.96		437	12.0	0.0
S10-960920-0004	C2B08D1		3.52		501	16.4	0.0
S10-960920-0019	C2B08D2		3.65		513	17.4	0.0
S10-960920-0020	C2B08D3		3.65		862	28.2	0.0
S10-960920-0009	C2B08D4		3.52		723	23.6	0.0
S10-960920-0007	C2B08D5		3.56		95	3.1	0.0
S10-960921-2244	C2B09D1		2.77		644	16.8	0.0
S10-960921-2241b	C2B09D2		1.95		255	4.6	0.0
S10-960921-2241a	C2B09D3		2.47		458	10.5	0.0
S10-960920-2221	C2B09D4		1.91		598	10.8	0.0
S10-960920-2220	C2B09D5		1.92		559	10.0	0.0
S10-960920-2035	C2B09D6		1.80		909	15.2	0.0
S10-960920-2222	C2B09D7		2.39		283	6.5	0.0
S10-960921-2238	C2B09D8		2.19		24	0.5	0.0
S10-960921-2037	C2B09D9		1.67		140	2.4	0.0
S10-092296-1203	C2B10D1		2.01		852	15.9	0.0
S10-960923-2030	C2B10D2		2.89		236	5.9	0.0
S10-960923-2020	C2B10D3		2.70		908	22.8	0.0
S10-960923-1230	C2B10D4		2.58		942	22.6	0.0
S10-960924-1701	C2B11D3		3.18		814	24.0	0.0
S10-960925-1705	C2B11D4		3.14		878	25.6	0.0

Campaign 2
Lead & Barium Tracking: Glass - Straight Analysis

Table V
p2 of 2
Pb & Ba Retained in Glass

User Sample I.D.	DRUM ID	FACTS I.D.	Concentration (wt%)		Lbs. of Glass Produced	Total Weight (lbs)	
			Pb as PbO	Ba as BaO		Lead	Barium
S10-961010-1801	C2B12D3		0.53		231	1.1	0.0
S10-961010-1803	C2B12D4		2.37		446	9.8	0.0
S10-961010-2000	C2B12D5		2.70		254	6.4	0.0
S10-961013-1814	C2B13D1		2.93		382	10.4	0.0
S10-961013-1822	C2B13D2		2.96		575	15.8	0.0
S10-961026-0805	C2B14D1		2.89		670	23.3	0.0
S10-961019-0200	C2B15D2		2.89		198	5.3	0.0
S10-961019-0100	C2B15D3		2.66		93	2.3	0.0
S10-961019-0130	C2B15D4		2.72		325	8.2	0.0
S10-961021-2032	C2B16D1		2.46		820	18.7	0.0
S10-961026-0815	C2B16D3		2.41		93	2.1	0.0
S10-961026-0825	C2B16D4		2.18		769	15.6	0.0
S10-961026-0835	C2B16D5		2.46		338	7.7	0.0
S10-961024-0810	C2B17D1		1.58		1062	15.6	0.0
S10-961024-0815	C2B17D4		1.33		789	9.7	0.0
S10-961101-1230	C2B18D4		1.10		754	7.7	0.0
S10-961101-1240	C2B18D5		0.87		799	6.5	0.0

Total Pb introduced into system:
Total Pb discharged as glass: 847.1
Total Ba introduced into system:
Total Ba discharged as glass: 0.0

Campaign 2
Lead & Barium Tracking: Glass - Normalized Analysis

Table V
p 1 of 2
Pb & Ba Retained in Glass

User Sample I.D.	DRUM ID	FACTS I.D.	Concentration (wt%)		Lbs. of Glass	Total Weight (lbs)	
			Pb as PbO	Ba as BaO	Produced	Lead	Barium
S10-960827-0030	C2B01D1		[illegible]		781	8.9	0.0
S10-960827-1149	C2B01D2		4.11		751	28.7	0.0
S10-960827-1205	C2B01D3		3.83		841	29.9	0.0
S10-960827-1241	C2B01D4		3.76		<20	0.0	0.0
S10-960906-1303	C2B02D1		3.41		869	27.5	0.0
S10-960906-1307	C2B02D2		4.27		303	12.0	0.0
S10-960906-1312	C2BO2D3		3.61		293	9.8	0.0
S10C-960907-0300	C2B03D1		3.51		457	14.9	0.0
S10-960906-1030	C2B03D2		4.19		150	5.8	0.0
S10C-960907-0305	C2B03D3		3.51		144	4.7	0.0
S10-960906-0602	C2B04D3		3.58		1038	34.5	0.0
Not Sampled	C2B04D4		[illegible]		723	24.2	0.0
S10-960912-0933	C2B05D1		4.89		339	15.4	0.0
S10-960912-1010	C2B05D2		4.42		677	27.8	0.0
S10-960912-1023	C2B05D3		4.52		1134	47.6	0.0
S10-960914-2345	C2B06D1		4.22		352	13.8	0.0
S10-960915-0010	C2B06D2		4.36		968	38.2	0.0
S10-960914-2350	C2B06D3		4.60		262	11.2	0.0
S10-960915-0001	C2B06D4		5.28		750	36.8	0.0
S10-960920-0009	C2B07D1		3.92		758	27.5	0.0
S10-960916-0230	C2B07D2		4.27		801	31.8	0.0
S10-960920-0006	C2B07D5		3.85		437	15.8	0.0
S10-960920-0004	C2B08D1		3.67		501	16.9	0.0
S10-960920-0019	C2B08D2		4.08		513	19.4	0.0
S10-960920-0020	C2B08D3		3.99		862	31.9	0.0
S10-960920-0009	C2B08D4		3.96		723	26.6	0.0
S10-960920-0007	C2B08D5		3.67		95	3.4	0.0
S10-960921-2244	C2B09D1		3.04		644	18.2	0.0
S10-960921-2241b	C2B09D2		2.27		255	5.4	0.0
S10-960921-2241a	C2B09D3		2.87		458	12.2	0.0
S10-960920-2221	C2B09D4		2.58		596	14.3	0.0
S10-960920-2220	C2B09D5		2.36		559	12.2	0.0
S10-960920-2035	C2B09D6		2.39		909	20.2	0.0
S10-960920-2222	C2B09D7		2.66		293	7.2	0.0
S10-960921-2238	C2B09D8		2.30		24	0.5	0.0
S10-960921-2037	C2B09D9		2.09		140	2.7	0.0
S10-092296-1203	C2B10D1		2.16		852	17.1	0.0
S10-960923-2030	C2B10D2		2.93		236	6.4	0.0
S10-960923-2020	C2B10D3		2.97		908	25.0	0.0
S10-960923-1230	C2B10D4		2.75		942	24.0	0.0
S10-960924-1701	C2B11D3		3.26		814	24.6	0.0
S10-960925-1705	C2B11D4		[illegible]		878	25.8	0.0

Campaign 2
Lead & Barium Tracking: Glass - Normalized Analysis

Table V
p 2 of 2
Pb & Ba Retained in Glass

User Sample I.D.	DRUM ID	FACTS I.D.	Concentration (wt%)		Lbs. of Glass Produced	Total Weight (lbs)	
			Pb as PbO	Ba as BaO		Lead	Barium
S10-961010-1801	C2B12D3		0.65		231	1.4	0.0
S10-961010-1803	C2B12D4		2.68		446	11.1	0.0
S10-961010-2000	C2B12D5		3.14		254	7.4	0.0
S10-961013-1814	C2B13D1		5.13		382	18.2	0.0
S10-961013-1822	C2B13D2		3.43		575	18.3	0.0
S10-961026-0805	C2B14D1		3.42		870	27.6	0.0
S10-961019-0200	C2B15D2		3.59		196	6.6	0.0
S10-961019-0100	C2B15D3		3.36		93	2.9	0.0
S10-961019-0130	C2B15D4		3.51		325	10.6	0.0
S10-961021-2032	C2B16D1		3.19		820	24.3	0.0
S10-961026-0815	C2B16D3		3.06		93	2.6	0.0
S10-961026-0825	C2B16D4		2.70		769	18.3	0.0
S10-961026-0835	C2B16D5		3.09		338	9.7	0.0
S10-961024-0810	C2B17D1		1.84		1062	18.1	0.0
S10-961024-0815	C2B17D4		1.62		789	11.9	0.0
S10-961101-1230	C2B18D4		1.21		754	8.5	0.0
S10-961101-1240	C2B18D5		0.94		799	7.0	0.0

Total Pb introduced into system:	**231.0**
Total Pb discharged as glass:	**988.0**
Total Ba introduced into system:	**446.0**
Total Ba discharged as glass:	**0.0**

Campaign 2
Lead & Barium Tracking: Filters

Table V
Pb & Ba Accumulated on Filters

Customer Sample I. D.	FACTS Sample I.D.	Sample Concentration (mg/Kg)		Dry Filter Weight (Kg)	Total Lead on Filter (lbs)	Total Barium on Filter (lbs)
		Lead	Barium			
					0.000	0.000
					0.000	0.000
					0.000	0.000

Campaign 4
Lead & Barium Tracking

Table V
Pb & Ba Introduced into the VitPP System

BATCH	Lead (lbs) as PbO	Barium (lbs) as $BaSO_4$	Barium (lbs) as $BaCO_3$	Lbs Frit Added	Lbs Lead Introduced	Lbs Barium Introduced
C4B01	300	94	120	125	278.5	156.5
C4B02	300	80	100	725	278.5	222.4
C4B03	200	100	50.0	175	185.7	122.7
C4B04	200	100	0.00	375	185.7	116.1
C4B05	200	100	0.00	175	185.7	87.9
C4B06	200	100	0.00	125	185.7	80.8
C4B07	200	100	0.00	0	185.7	63.2
C4B07 1/2	100	50	0.00	125	92.8	49.2
C4B08	175	100	0.00	375	162.5	116.1
C4B09	200	100	25	50	185.7	87.5
Post C4B09	0.00	0.00	0.00	225	0.0	31.7

000199

Total Lead introduced into system: **1926.3**
Total Barium introduced into system: **1134.1**

Campaign 4
Lead & Barium Tracking: Glass - Straight Analysis

Table V
Pb & Ba Retained in Glass

[redacted] = Approximation

User Sample I.D.	DRUM ID	FACTS I.D.	Concentration (wt%)		Lbs. of Glass Produced	Total Weight (lbs)	
			Pb as PbO	Ba as BaO		Lead	Barium
S10-961202-1200	C4B01D1	200269566	2.89	1.51	783	21.0	10.6
S10-961201-1055	C4B01D3	200269365	4.14	2.38	879	33.8	18.7
S10-961201-1100	C4B01D4	200269366	2.38	1.44	855	18.9	11.0
S10-961203-1040	C4B02D1	200269601	5.13	2.82	698	33.2	17.6
S10-961203-2315	C4B02D2	200270231	5.73	3.21	539	28.7	16.5
S10-961203-2326	C4B02D3	200270230	5.21	3.51	886	42.9	27.9
S10-961204-1800	C4B02D5	200271183	6.13	3.62	118	6.7	3.8
S10-961204-2100	C4B02D6	200271184	6.13	3.31	395	22.5	11.7
S10-961211-2145	C4B03D1	200271764	7.13	3.85	782	51.8	27.0
S10-961207-1315	C4B03D2	200270497	4.83	3.32	619	27.8	18.4
S10-961207-1328	C4B03D4	200271680	4.17	2.86	739	28.8	18.9
S10-961208-0200	C4B04D1	200270280	4.92	3.63	776	35.4	25.2
S10-961209-1245	C4B04D3	200271330	6.59	4.46	730	44.7	29.2
S10-961208-1250	C4B04D4	200270520	6.14	4.52	941	53.8	38.1
S10-961209-1250	C4B04D5	200271331	4.89	3.21	759	34.5	21.8
S10-961209-2035	C4B05D1	200270471	5.15	3.06	842	40.3	23.1
S10-961210-1200	C4B05D2	200268027	7.93	3.80	969	72.8	33.7
S10-961210-0600	C4B05D5	200270472	7.99	4.16	124	8.2	4.6
S10-961211-1200	C4B06D1	200271736	7.86	3.42	815	59.5	25.0
S10-961211-2015	C4B06D4	200271765	8.97	3.38	934	77.8	28.1
S10-961212-2220	C4B07D1	200271801	5.15	2.21	821	39.3	16.3
S10-961215-1015	C4B07D2	200272136	6.40	2.90	283	15.6	6.8
S10-961212-1745	C4B07D4	200272634	6.12	2.91	1063	61.5	28.2
S10-961214-1000	C4B07D5	200272012	5.93	2.40	915	50.4	18.7
S10-961215-0540	C4B07D6	200271800	6.90	3.00	790	50.8	21.2
S10-961215-1000	C4B07D7	200272135	7.06	2.67	348	22.8	8.3
S10-961213-0200	C4B07D3	200272633	6.61	2.75	<5.0	0.0	0.0
S10-961217-2000	C4B08D1	200272754	6.90	3.10	856	54.8	23.8
S10-961217-2005	C4B08D4	200272755	6.80	2.90	645	40.7	16.8
S10-961217-2030	C4B08D5	200272780	6.70	3.00	450	28.0	12.1
S10-961223-1310	C4B09D1	200273902	6.54	3.49	995	60.4	31.1
S10-961224-0835	C4B09D4	200273040	6.17	3.09	1051	60.2	28.1
S10-961225-2142	C4B09D5	200273986	6.26	2.99	751	43.8	20.1
S10-961226-0203	C4B08AD1	200273987	5.41	2.36	368	18.5	7.8
c4b10d1			[redacted]	[redacted]	340	18.9	9.1
catc basin			5.53	3.07	3632	196.7	106.4
floor			5.20	2.85	1358	65.8	36.9

Total Pb introduced into system: 1926.3
Total Pb discharged as glass: 1571.1
Total Ba introduced into system: 1134.1
Total Ba discharged as glass: 801.5

Campaign 4
Lead & Barium Tracking: Glass - Normalized Analysis

Table V
Pb & Ba Retained in Glass

[redacted] = approximation

User Sample I.D.	DRUM ID	FACTS I.D.	Concentration (wt%)		Lbs. of Glass Produced	Total Weight (lbs)	
			Pb as PbO	Ba as BaO		Lead	Barium
S10-961202-1200	C4B01D1	200269566	2.89	1.50	783	16.7	10.8
S10-961201-1055	C4B01D3	200269365	4.22	2.42	879	30.7	19.5
S10-961201-1100	C4B01D4	200269366	2.54	1.54	855	19.5	12.9
S10-961203-1040	C4B02D1	200269601	5.23	2.87	866	30.2	18.4
S10-961203-2315	C4B02D2	200270231	6.48	3.63	539	31.6	18.8
S10-961203-2326	C4B02D3	200270230	6.09	4.11	866	48.8	35.1
S10-961204-1600	C4B02D5	200271183	6.60	3.90	118	7.8	5.2
S10-961204-2100	C4B02D6	200271184	6.78	3.65	395	28.2	18.4
S10-961211-2145	C4B03D1	200271764	7.38	3.98	782	51.2	35.5
S10-961207-1315	C4B03D2	200270497	5.51	3.79	619	30.8	20.8
S10-961207-1328	C4B03D4	200271680	5.37	3.68	739	35.8	24.1
S10-961208-0200	C4B04D1	200270260	5.85	4.17	776	38.8	28.7
S10-961209-1245	C4B04D3	200271330	7.88	5.33	730	53.4	34.8
S10-961208-1250	C4B04D4	200270520	6.75	4.97	941	57.3	41.5
S10-961209-1250	C4B04D5	200271331	5.42	3.67	759	37.1	24.7
S10-961209-2035	C4B05D1	200270471	10.34	6.15	842	78.6	45.9
S10-961210-1200	C4B05D2	200268027	8.38	4.02	989	73.5	45.4
S10-961210-0600	C4B05D5	200270472	8.22	4.28	124	8.0	6.1
S10-961211-1200	C4B06D1	200271736	8.31	3.62	815	60.1	33.7
S10-961211-2015	C4B06D4	200271765	9.05	3.39	934	75.9	36.1
S10-961212-2220	C4B07D1	200271801	7.47	3.51	821	55.3	25.6
S10-961215-1015	C4B07D2	200272136	7.85	3.47	283	20.9	9.3
S10-961212-1745	C4B07D4	200272634	7.90	3.76	1083	77.2	36.1
S10-961214-1000	C4B07D5	200272012	7.84	3.09	915	63.1	25.1
S10-961215-0540	C4B07D6	200271800	7.78	3.38	790	63.8	27.2
S10-961215-1000	C4B07D7	200272135	7.06	5.15	348	25.5	18.3
S10-961213-0200	C4B07D3	200272633	6.61	2.75	<5.0	0.0	0.0
S10-961217-2000	C4B08D1	200272754	7.58	3.41	856	67.3	29.8
S10-961217-2005	C4B08D4	200272755	7.83	3.25	645	51.9	21.4
S10-961217-2030	C4B08D5	200272780	7.26	3.25	450	33.9	14.9
S10-961223-1310	C4B09D1	200273902	8.57	4.57	995	87.3	47.9
S10-961224-0835	C4B09D4	200273040	11.74	6.17	1051	128.3	67.1
S10-961225-2142	C4B09D5	200273986	6.26	6.47	751	48.1	50.3
S10-961226-0203	C4B08AD1	200273987	5.41	2.36	368	20.4	9.0
c4b10d1			[redacted]	[redacted]	340	18.9	8.1
calc basin			5.53	3.07	3832	198.7	105.4
floor			5.20	2.95	1358	66.6	35.9

Total Pb introduced into system: 1926.3
Total Pb discharged as glass: 1835.9
Total Ba introduced into system: 1134.1
Total Ba discharged as glass: 1045.0

Campaign 4
Lead & Barium Tracking: Water **

Table V
p1 of 3
Pb & Ba Discharged from Buiding Sump Tank

Customer Sample I.D.	FACTS Sample I.D.	Concentration of S-17 Sample (ug/L)		Run time of BST Pump (min)	Total Pb Discharged (lbs)	Total Ba Discharged (lbs)
		Lead	Barium			
S17-961130-0040	200270203			11.40	0.00000	0.00000
S17-963011-1100	200270206	904.0	503.9	18.00	0.00204	0.00114
S17-961130-1832	200269372	699.8	266.0	126.34	0.01124	0.00427
S17-961130-0158	200270205	1895.0	983.0	35.28	0.00837	0.00434
S17-961201-0146	200270448	239.0	BDL	24.00	0.00072	0.00000
S17-961202-0610	200270727	130.7	BDL	39.00	0.00064	0.00000
S17-961202-0715	200270728	3669.0	BDL	39.00	0.01791	0.00000
S17-961202-0830	200270729	4136.0	BDL	40.00	0.02070	0.00000
S17-961202-0930	200270730	187.0	BDL	0.00	0.00000	0.00000
S17-961202-1030	200270731	3163.0	BDL	32.00	0.01267	0.00000
S17-961202-1130	200270732	507.2	BDL	27.00	0.00171	0.00000
S17961202-0945	200269573	4270.0	BDL	39.00	0.02084	0.00000
S17-961202-1230	200270746	1833.0	310.0	33.00	0.00757	0.00128
S17-961202-1334	200270747	971.4	218.3		0.00000	0.00000
S17-961203-0140	200270745	310.0	305.0	29.00	0.00113	0.00111
S17-961203-0243	200270940	1360.0	BDL	42.00	0.00715	0.00000
S17-961203-0400	200270819	3217.0	BDL	24.00	0.00966	0.00000
S17-961203-0425	200269606	4191.0	693.0	8.00	0.00420	0.00069
S17-961203-0500	200270820	615.3	BDL	39.00	0.00300	0.00000
S17-961203-0600	200270821	150.7	BDL	70.00	0.00132	0.00000
S17-961203-0800	200270822	108.0	BDL	32.00	0.00043	0.00000
S17-961203-0905	200270823	76.0	BDL	11.00	0.00010	0.00000
S17-961204-0915	200270237			163.02	0.00000	0.00000
S17-961204-2253	200271201	8330.0	709.0	39.00	0.04066	0.00346
S17-961205-0003	200271202	4131.0	BDL	49.00	0.02533	0.00000
S17-961205-0107	200271658	5851.0	148.6	64.00	0.04526	0.00119
S17-961205-0315	200271660	4426.0	54.3	178.00	0.09660	0.00121
S17-961205-0800	200270265			63.00	0.00000	0.00000
S17-961205-1200	200271296			28.00	0.00000	0.00000
S17-961205-1610	200271437	5259.0	535.0	15.00	0.00697	0.00100
S17-961206-0940	200270478	1008.0	BDL		0.00000	0.00000
S17-961207-1030	200270502	583.3	52.7		0.00000	0.00000
S17-961208-0410	200270527	29650.0	10010.0		0.00000	0.00000
S17961208-1200	200271337	2860.0	601.0		0.00000	0.00000
S17961208-1202	200271338	2933.0	608.5		0.00000	0.00000
S17-961210-0005	200272513				0.00000	0.00000
S17-961210-0935	200272497	1122	BDL		0.00000	0.00000

Total Pb discharged from Sump Tank (lbs):	**0.351**	**Estimated total:**	**0.866**
Total Ba discharged from Sump Tank (lbs):	**0.020**		**0.069**

** The results for the first third of the samples listed in this table were evaluated for total lead and barium detected. The analysis for the first third of the samples accounted for only 0.351 pounds of lead and 0.020 pounds of barium. It was determined that the amount of lead and barium detected in the building sump tank was negligible, and the remaining sample calculations be estimated by using the average pump run time.

Campaign 4
Lead & Barium Tracking: Water

Table V
p2 of 3
Pb & Ba Discharged from Buiding Sump Tank

Customer Sample I.D.	FACTS Sample I.D.	Concentration of S-17 Sample (ug/L)		Run time of BST Pump (min)	Total Pb Discharged (lbs)	Total Ba Discharged (lbs)
		Lead	Barium			
S17-961212-0105	200271773	16740.0	2882.0		0.0000	0.0000
S17-961212-0948	200271772	2309.0	562.2		0.0000	0.0000
S17-961214-1035	200272020	1719.0	BDL		0.0000	0.0000
S17-961214-1040	200272021	1636.0	BDL		0.0000	0.0000
S17-961215-1812	200272146				0.0000	0.0000
S17-961215-1910	200272143	16930.0	2295.0		0.0000	0.0000
S17-961215-2010	200273343	4345.0	529.0		0.0000	0.0000
S17-961215-2110	200273345	458.0	140.4		0.0000	0.0000
S17-961215-2210	200273597	785.0	238.0		0.0000	0.0000
S17-961215-2310	200273600	411.0	161.0		0.0000	0.0000
S17-961216-1200	200273311				0.0000	0.0000
S17-961216-0100	200273312	366.0	BDL		0.0000	0.0000
S17-961216-0200	200273313	851.2	BDL		0.0000	0.0000
S17-961216-0300	200273314	336.0	BDL		0.0000	0.0000
S17-961216-0400	200273315	273.0	BDL		0.0000	0.0000
S17-961216-0500	200273316				0.0000	0.0000
S17-961216-1030	200272664	1714.0	143.3		0.0000	0.0000
S17-961216-1032	200272665	1888.0	183.0		0.0000	0.0000
S17-961216-1600	200273269				0.0000	0.0000
S17-961216-1830	200273270	2096.0	79.0		0.0000	0.0000
S17-961216-1930	200273271	1186.0	216.0		0.0000	0.0000
S17-961216-2230	200273278	769.4	205.9		0.0000	0.0000
S17-961217-0006	200273281	344.7	76.6		0.0000	0.0000
S17-961217-0215	200273283				0.0000	0.0000
S17-961217-0410	200273286	220.2	73.0		0.0000	0.0000
S17-961217-1240	200272762	2197.0	1061.0		0.0000	0.0000
S17-961217-1242	200272761	2310.0	1100.0		0.0000	0.0000
S17-961218-2130	200272786				0.0000	0.0000
S17-961220-0435	200272880	251.3	41.8		0.0000	0.0000
S17-961220-1112	200272879	1110.0	119.0		0.0000	0.0000
S17-961220-1515	200274754	279.5	BDL		0.0000	0.0000
S17-961220-2040	200274770	2972.0	BDL		0.0000	0.0000
S17-961220-2147	200274771	2324.0	BDL		0.0000	0.0000
S17-961221-0930	200272986	324.0	57.0		0.0000	0.0000
S17-961221-1535	200272987	276.0	54.0		0.0000	0.0000
S17-961221-1850	200274806	419.7	BDL		0.0000	0.0000
S17-961221-2308	200275450	421.4	BDL		0.0000	0.0000

Total Pb discharged from Sump Tank (lbs): 0.351

Total Ba discharged from Sump Tank (lbs): 0.020

Campaign 4
Lead & Barium Tracking: Water

Table V
p3 of 3
Pb & Ba Discharged from Buiding Sump Tank

Customer Sample I.D.	FACTS Sample I.D.	Concentration of S-17 Sample (ug/L)		Run time of BST Pump (min)	Total Pb Discharged (lbs)	Total Ba Discharged (lbs)
		Lead	Barium			
S17-961222-0405	200273047	300.0	52.9		0.0000	0.0000
S17-961222-1300	200273048	265.0	49.0		0.0000	0.0000
S17-961222-2201	200274784	347.7	BDL		0.0000	0.0000
S17-961223-0800	200273910				0.0000	0.0000
S17-961223-1230	200273911				0.0000	0.0000
S17-961223-2235	200276329	62.9	24.1		0.0000	0.0000
S17-961224-0015	200276338	99.7	BDL		0.0000	0.0000
S17-961224-0605	200276368	327.0	BDL		0.0000	0.0000
S17-961224-0920	200273932	230.0	38.1		0.0000	0.0000
S17-961224-1957	200273933				0.0000	0.0000
S17-961225-0930	200273948	38.6	15.6		0.0000	0.0000
S17-961225-1532	200273949	37.7	19.2		0.0000	0.0000
S17-961225-1747	200276489	93.0	BDL		0.0000	0.0000
S17-961225-2334	200273986	2040.0	24.0		0.0000	0.0000
S17-961226-0520	200275964				0.0000	0.0000
S17-961226-1019	200275986				0.0000	0.0000
S17-961226-1445	200275837	30.5	BDL		0.0000	0.0000
S17-961226-2353	200275839				0.0000	0.0000
S17-961227-0107	200275830	53.0	BDL		0.0000	0.0000
S17-961227-0408	200276490				0.0000	0.0000
S17-961227-0845	200274026	1975.0	263.0		0.0000	0.0000
S17-961227-1005	200276491				0.0000	0.0000
S17-961228-0125	200274083	3293.0	204.2		0.0000	0.0000
S17-961228-0350	200274084	211.0	110.9		0.0000	0.0000
S17-961228-0815	200275541	645.0	56.8		0.0000	0.0000
S17-961228-1030	200275547	765.7	42.3		0.0000	0.0000
S17-961228-1230	200275548	1502.0	62.7		0.0000	0.0000
S17-961229-0237	200274118	2191.0	233.8		0.0000	0.0000
S17-961229-0352	200274119	4470.0	351.6		0.0000	0.0000
S17-961229-1000	200275522	734.0	84.2		0.0000	0.0000
S17-961230-0900	200274190	2806.0	148.9		0.0000	0.0000
S17-961230-1700	200275785	3093.0	76.4		0.0000	0.0000
S17-970102-0830	200274588				0.0000	0.0000
S17-970103-0830	200274589				0.0000	0.0000
					0.0000	0.0000
					0.0000	0.0000

Total Pb discharged from Sump Tank (lbs): 0.351

Total Ba discharged from Sump Tank (lbs): 0.029

Campaign 4
Lead & Barium Tracking: Filters

Table V
Pb & Ba Accumulated on Filters

Customer Sample I. D.	FACTS Sample I.D.	Sample Concentration (mg/Kg)		Dry Filter Weight (Kg)	Total Lead on Filter (lbs)	Total Barium on Filter (lbs)
		Lead	Barium			
S64PAF-961130-1230	200270899			1.346	0.000	0.000
S64HAF-961130-1235	200271004	BDL	3.12	1.704	0.000	0.000
S48PAFA-961201-1050	200270486	23791.00	4746.00	1.346	0.071	0.014
S48PAFB-961201-1039	200270487	334.00	84.19	1.346	0.001	0.000
S48PAFA-961202-0920	200270755	25400.00	3300.00	1.346	0.075	0.010
S48PAFA-961202-0900	200270758			1.346	0.000	0.000
S48PAFA-961203-0900	200270939	3.36	0.62	1.346	0.000	0.000
S48PAFB-961203-0825	200270942			1.346	0.000	0.000
S48HAFB-961203-0827	200270943	4.37	0.60	1.704	0.000	0.000
S48PAFA-961204-2000	200271181			1.346	0.000	0.000
S48AAFP-961205-0440	200271413	351.00	58.00	1.346	0.001	0.000
S48BAFP-961205-1420	200271415	47500.00	5800.00	1.346	0.141	0.017
S48HAFA-961206-1020	200271688			1.704	0.000	0.000
S48PAFB-961211-1512	200273755			1.346	0.000	0.000
S48PAFA-961212-0900				1.346	0.000	0.000
837	200271327			1.500	0.000	0.000
848	200271328			1.500	0.000	0.000
S48PAFB-961215-0810				1.346	0.000	0.000
S48A-961216-0800				1.500	0.000	0.000
S48PA-961217-1325	200273721			1.346	0.000	0.000
S48PB-961217-1330	200273722			1.346	0.000	0.000
837PS-961218-0830	200274580			1.346	0.000	0.000
S48HAFA-961218-0835	200274581	53611.00	5168.00	1.704	0.201	0.019
S48HAFB-961218-0840	200274582	55719.00	4483.00	1.704	0.209	0.017
S48PAFA-961219-0835	200274504	43494.00	5553.00	1.346	0.129	0.016
S48PAFB-961219-0840	200274505			1.346	0.000	0.000
S48PAFB-961220-0830	200274753	51672.00	4852.00	1.346	0.153	0.014
S48PAFA-961221-0840	200274762			1.346	0.000	0.000
S48PAFA-962122-0935	200275453			1.346	0.000	0.000
S48PAFB-961222-0940	200275454			1.346	0.000	0.000
S48AFB-961223-0905	200276287			1.346	0.000	0.000
S48AFB-961223-0915	200276288			1.346	0.000	0.000
S48AFA-961223-1035	200276289			1.346	0.000	0.000
S48PAFB-961224-0835	200276482	45685.00	5110.00	1.346	0.135	0.015
S48PAFA-961225-0845	200276484	77200.00	5401.00	1.346	0.229	0.016
S48PAFB-961226-1310	200275699			1.346	0.000	0.000
S48PAFA-961227-0830	200276487			1.346	0.000	0.000

Total Lead accumulated on filters: 1.3
Total Barium accumulated on filters: 0.1

MELTER STEADY STATE IDLE POWER REQUIREMENTS

Idle Dates	Idle Times	Idle Duration Hours	Avg Power KW	Avg Bath Temp Deg. C	Watt/Deg C Idle Power
6/9 - 6/9	1000 - 2200	12	70	1090	64
6/12 - 6/13	1800 - 1000	16	82	1124	73
6/13 - 6/15	1700 - 2300	54	84	1130	74
6/24 - 6/24	0300 - 2300	20	115	1260	91
7/3 - 7/3	0200 - 1800	16	89	1121	79
7/12 - 7/13	0400 - 2200	42	99	1138	87
8/5 - 8/6	1600 - 0100	9	96	1130	85
8/11 - 8/13	1200 - 0900	21	100	1130	88
8/28 - 8/28	0000 - 1500	15	130	1265	103
8/30 - 8/30	0300 - 1500	12	125	1260	99
9/12 - 9/12	0700 - 2300	16	125	1260	99
9/15 - 9/15	0000 - 0900	9	153	1265	121
9/25 - 9/25	0100 - 1200	11	141	1177	120
9/27 - 9/27	0200 - 1500	13	122	1151	106
9/29 - 9/30	1600 - 0900	17	122	1150	106
10/3 - 10/4	1100 - 0600	19	114	1138	100
10/9 - 10/10	0100 - 1300	36	121	1171	103
10/13 - 10/14	1800 - 1200	19	125	1173	107
10/19 - 10/19	0200 - 1200	10	169	1255	135
10/25 - 10/26	1600 - 1400	22	121	1175	103
11/2 - 11/3	0700 - 0400	21	116	1145	101
11/3 - 11/5	1800 - 2300	53	115	1145	100
11/9 - 11/10	1200 - 2300	35	136	1202	113
11/17 - 11/19	1700 - 0900	40	99	1100	90
11/27 - 11/28	0800 - 0800	22	105	1120	94
12/5 - 12/5	0500 - 1900	14	147	1250	118
12/24 - 12/25	2000 - 0500	15	150	1148	131
12/25 - 12/26	1800 - 0600	14	150	1155	130

APPENDIX H

TEAM COMPOSITION

Gail E Bingham, Consultant

Mr. Bingham has a B.S. Chemical Engineering from Oregon State University. In addition, Mr. Bingham has 26 years experience at the Idaho National Engineering Laboratory (INEL) Chemical Processing Plant responsible for engineering and project management. Positions held include Manager Strategic Affairs, Manager Major Projects, FPR Project Manager, NWCF Project Manager. Mr. Bingham also participated in the Operable Unit 4 Value Engineering study.

Joel B. Bradburne, Vitrification Pilot Plant Operations Manager

Mr. Bradburne has a B.S. in Biology from Bridgewater College, and is completing his M.S. degree in Environmental Management from the University of Findlay. Mr. Bradburne has over 15 years of experience in the nuclear industry. This includes the overhaul and new construction of nuclear powered submarines, construction, start-up, and operations of commercial nuclear facilities, and acted as Operations Manager for the Fernald Vitrification Pilot Plant. Mr. Bradburne also worked as a consultant for NUS Corporation in their Field Operations and Training Division, servicing both DOE and NRC clients.

Hamid Hojaji, Manager Melter Operations

Dr. Hojaji has his Ph.D. in Materials Science, M.S. in Chemical Engineering and Materials Science from the Catholic University of America, and a B.S. in Chemical Engineering from Tehran Polytechnic Institute. Dr. Hojaji has over 19 years' experience in the laboratory setting specializing in direction of glass melting and forming lab facilities. He performs research on fluorides and high-transition temperature superconductors and directs the development of wastewater treatment systems to remove contaminants from the water. Dr. Hojaji manages the pilot testing of nuclear waste glasses, characterizes the vitrified waste, and manages selective ion exchange media development for mixed waste streams.

Vijay Jain, Ph.D. MBA, Senior Engineer

Dr. Jain has his Ph.D. in Ceramics from Alfred University and M.B.A. from St. Boneaventure University. He is currently employed at West Valley Nuclear Services Company. Dr. Jain is a recipient of the 1995 George Westinghouse Signature Award (Individual) in addition to the 1995 George Westinghouse Signature Award (Team). He is Vice Chairman of the Nuclear and Environmental Technology Division of the American Ceramic Society. Dr. Jain has authored more than 30 publications in technical journals and conference proceedings, in addition to editing three books on Nuclear Waste Management. Additionally, Dr. Jain has nine years experience in vitrification process development and engineering.

Ronald Joseph, Team Technical Specialist

Mr. Joseph is currently employed as a Team Technical Specialist in the Emergency Preparedness Group and has been employed at the FEMP since 1989. Ron's primary duties involve the investigation and reporting of occurrences and performing root cause analyses for events experienced at the FEMP. He has been trained in accident investigation and root cause analysis techniques and is the lead instructor for the FEMP's Root Cause Analysis Course. Ron has been an Investigation Team Leader on numerous investigations performed at the FEMP, including Type C Investigations and the Investigation of Adverse Radiological Trends conducted in 1995. Ron's experiences at the FEMP and from other nuclear power plant operations have given him a broad base of knowledge in a number of processes required to analyze events for root cause and corrective action determination.

Joe Metzler, Electrician

Mr. Metzler has over 18 years of electrical experience which included trouble shooting electrical equipment used in process and manufacturing of food products at the Kroger Company. Joe has been an electrician at Fernald since 1984. His responsibilities included testing and maintaining equipment used in production. Joe has been with the Vitrification Pilot Plant since start-up in March, 1996.

Donald Paine, Ph.D., Silos Project Manager

Dr. Paine has a B.S. in Mathematics and Biology, an M.S. in Health Physics, and a Ph.D. in Radiology and Radiation Biology. As Silos Project Manager, Dr. Paine is responsible for the programmatic and functional operation for the environmental clean-up and restoration of Operable Unit 4 (OU4). He has more than 20 years of ES&H and project management experience, including emergency services management, in all aspects of the nuclear fuel cycle applicable to the FEMP site. This includes fuel manufacturing, safe shutdown, waste management, decontamination and decommissioning (D&D) of contaminated facilities, and remediation of hazardous and mixed waste sites at DOE, and several NRC licensed facilities. Dr. Paine has successfully managed ES&H programs at an NRC Category I, Licensed Special Nuclear Fuel Fabrication Facility.

Raymond Reinhart, Security Manager

Mr. Reinhart has a B.S. degree in Business Administration, with a minor in Industrial Management from Franklin University. Additionally, Mr. Reinhart has recently completed his Masters' in Environmental Management at the University of Findlay. He has over 20 years of experience in management. This experience includes the last five years at Fernald as the Manager of Self Assessment and his recent appointment to the position of Manager of Security. Mr. Reinhart directed the development of the Self Assessment program for the Safety and Health Division. He is trained as an auditor/lead auditor and has managed the

activities of four audits in the past four years. In addition, Mr. Reinhart functioned as the Team Leader on the Readiness Assessment (RA) for the UNH Transfer D-1 to D-2 conducted in March/April 1994. He also served as a team member of the UNH Neutralization Project, Operational Readiness Review (ORR) in May of 1995. Most recently, he served as Team Leader for the Thorium Overpack Project Operational Readiness Review (ORR) completed in early 1996.

Kareld Solomon, Vitrification Pilot Plant Engineering Manager

Mr. Solomon holds a Bachelors Degree in Mechanical Engineering Technology. He has over 20 years of experience at DOE facilities. During his ten years at the Fernald Site, Kareld has held many management positions within the company, ranging from Project Engineer to Manager of the Engineering Division. These assignments have included technical direction, contract technical management, project management, and engineering policy development. He is the current Silos Project Engineering Manager.

Ronald C. Worsley, Facility Technical Engineering Manager

Mr. Worsley has a B.S. in Mechanical Engineering from Newark College of Engineering. He Worsley also has 35 years of experience in technical, construction, and project management, engineering design, specification preparation, and start-up of major equipment and processes. Ron is currently coaching and directing technical personnel to provide complete design engineering, perform design reviews, develop conceptual designs and design scopes on an array of technically diverse tasks. Additionally, he participated in the investigation of numerous incidents related to failures of equipment and processes on-site.

REFERENCES

REFERENCE 1

Can the corrosion of tank bottoms be prevented?

P P BOGGUM

The phenomena of upward drilling and downward drilling in refractory materials by metals and bubbles in the glass melt is one of the most irritating causes of corrosion in tank furnace bottoms. In this article, P P Boggum of Didier-Werke AG, explains the nature of the phenomena and some of its causes and suggests a way in which it could be avoided.

Flux level corrosion, upward drilling (by bubbles) and downward drilling (by metals) can all be attributed to boundary layer convection, although externally they present totally different corrosion patterns.

The problem is fourfold:

1. To avoid upward drilling in the bottom by any measures which prevent the glass from penetrating into the horizontal joints of the bottom. Suggested methods include the use of large, plane, smooth shapes; accurate determination of a minimum number of vertical joints arranged to overlap; and the use of the thinnest intermediate layers of mortar or cement.
2. To reduce downward drilling by metal in the bottom by controlling the raw materials and the cullet for the glass batch, and by installing a further protective course for the bottom of direct-bonded 'Corundum-Chrome' tiles (Supral AR90).
3. To construct the bottom of large, ground, dry-laid bottom blocks.
4. To construct the insulation of tiles with a good compressive strength, sufficiently high application temperature, and low thermal conductivity (Legral 35/2G).

The attack on the tank bottom takes place by two different processes of destruction:

a: Upward drilling by the glass; and
b: Downward drilling by the metal.

Metal is generally introduced into the tank in the raw materials, the batch mix and cullet. In the case of lead glass tanks, other 'carriers' of metal in the tank bottom can be the reducing melt or, in the case of electrically heated tanks, molybdenum electrodes. These problems are not always solved by using well-sorted cullet or magnetic separators, for these attract only iron and nickel but not stainless steel or non-ferrous metals. As is shown by an example which is discussed later, magnetic separators which have failed over a longer period can lead to disastrous results in the tank bottom. The problem of re-cycling cullet must not be neglected either, because this is used increasingly by glass works both for environmental reasons and because of the shortage of raw materials. The sorting of non-ferrous metal bottle tops is, in this instance, an economic problem.

Conditions of upward drilling

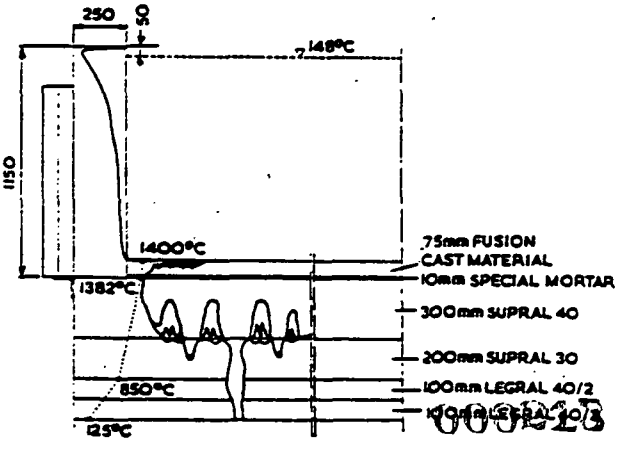
The appearance of upward drilling is well-known and has been discussed in detail in many reports.[1] This particular corrosion process has been extremely well illustrated in a film by Busby.

Upward drilling is a reaction at the boundary of the three phases: gaseous, liquid and solid. The gaseous condition is given by a gas bubble, the liquid condition by the glass and the solid condition by the refractory material. Attack by upward drilling occurs when the glass and refractory material, which is more or less soluble in the glass melt, come into contact at a sufficiently high temperature, and when the construction at this point of contact is such that the refractory face is at an angle between the horizontal, and slightly less than vertical.

Another condition is a gas bubble which escapes from the melt or the refractory material and attaches itself to the underside of the material. This bubble now causes a drilling effect on the refractory material. The severity of the attack depends upon the solubility of the refractory material, the surface tension of the glass melt and the dissolved refractory material, the density and viscosity of both melts, and on the size of the bubble. In the case of a small bubble, the attack is relatively forced and a narrow channel is formed, whilst with a larger bubble a wider channel is formed. The rate of the drilling effect of the latter compared to that of the small bubble is lower. The surface tension and the viscosity of the melt escaping from the drilled channel influence the severity of the attack of this drilling process.

Fig 1 shows diagrammatically the appearance of a bottom

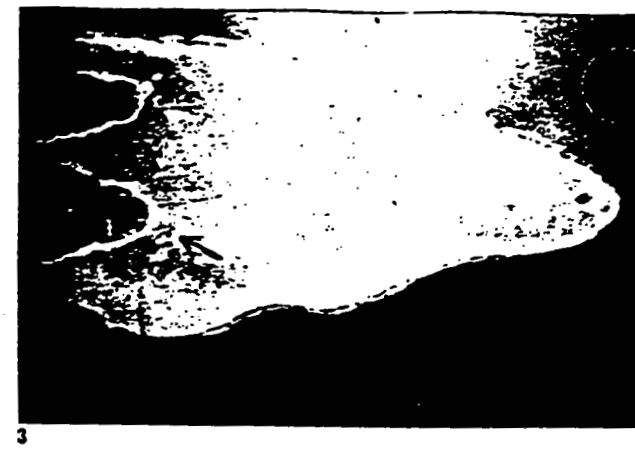
1 Metal drilling in the tank bottom. This particular tank furnace has a melting area of 45 m², cross-fire oil burners, and pulls 80-100 tons of white flint glass per day. The depth of the tank is 1100 mm and the temperature of the bottom 1400-1450°C. The furnace was on stream for 32 months.



GB-13



2



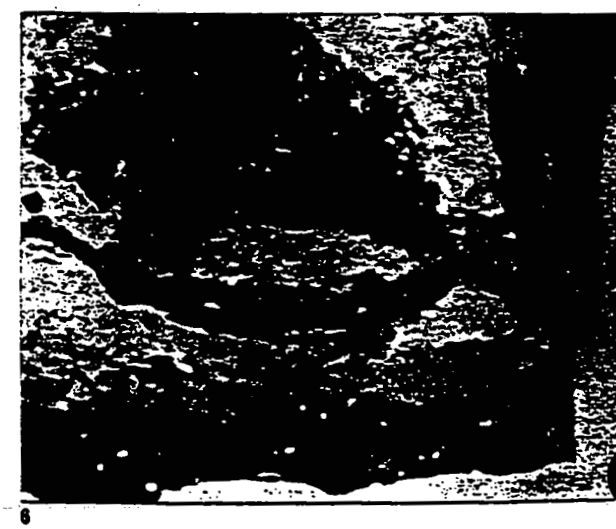
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4



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6

destroyed by upward and downward drilling. Fig 2 illustrates the 'jelly-like' structure of the fireclay bottom block remains; and fig 3 the cross section of such a 'jelly'. Solidified bubble in the drilled channel is clearly visible.

In tank bottoms, corrosion by upward drilling always occurs in those areas where glass has penetrated beneath the bottom paving. This can be the case when glass has infiltrated into the joints of the paving courses, or has penetrated into channels which intersect the horizontal joints of the bottom and which are the result of downward drilling by metal. Even when glass or liquid metal penetrates the areas of higher porosity of fusion cast tiles after the dense crystalline outer skin has been breached, this type of attack must be expected.

Conditions of downward drilling

Downward drilling is mainly caused by metal parts introduced into the tank. These metals can be divided into two groups:

1. Metals which liquefy in the appropriate temperature range between 1000 and 1400°C.
2. Metals which are still solid at these temperatures.

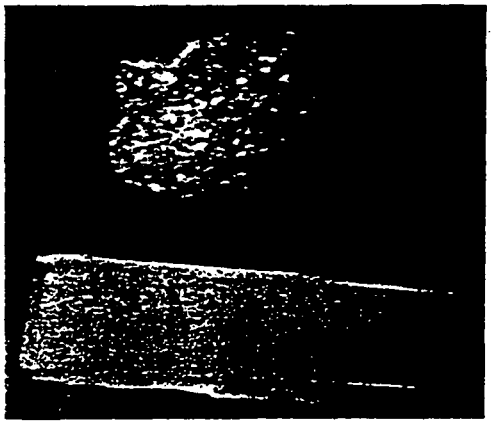
Compared to upward drilling, which is based on the material boundaries, gaseous–liquid–solid (gas–glass–refractories), the attack by metal is determined by the material boundaries, liquid–liquid–solid (metal–glass–refractories) or solid–liquid–solid (metal–glass–refractories).

On the three-material boundary, bubbles can also occur which do not participate in the corrosion of the refractory material but which migrate upwards into the glass. Brückner found a certain adhesion of these bubbles during laboratory tests. On prepared samples of metal inclusions in the bottom, we were also able to demonstrate this appearance of bubbles in the glass in the immediate vicinity of the metal. A drilling effect can hardly be ascribed to these bubbles.

Fig 4 shows a piece of fireclay bottom block with metal deposits; fig 5 a broken off metal (iron) with fireclay plug (button shape); and fig 6 the break area of this metal inclusion. Bubbles are embedded in the adjacent glass.

Metals	Melting temperature	Specific gravity	Electrical conductivity M/OHM MM² at 20° C	Magnetic properties
ALUMINIUM	659	2·7	37·6	No
ANTIMONY	630	6·6		
BRASS (70% Copper)	930	8·5		No
CADMIUM	320	8·6		
CALCIUM	842	1·5		
CHROME	1890	6·9		
COBALT	1495	8·9	16·1	
COPPER	1083	8·9	60·0	No
GOLD	1063	19·2		
IRON	1535	7·8	10·3	Yes
LEAD	327	11·3	4·8	No
MANGANESE	1260	7·2	0·5	
MAGNESIUM	651	1·7	22·0	
MOLYBDENUM	2621	10·2	19·4	
NICKEL	1455	8·9	14·6	Yes
POTASSIUM	760	0·9		
RHODIUM	1966	12·5	1·0	
SILICON	1420	2·3		
SODIUM	97	0·9	23·8	
TANTALUM	2991	16·6	8·1	
TIN	232	7·3	12·9	
TITANIUM	1800	4·4	1·2	
VANADIUM	1710	6·0	3·8	
ZINC	420	7·1	17·0.	No
ZIRCON	1857	6·5	2·4	

7



8



9



10

Metals in downward drilling

Whilst upward drilling is described in numerous scientific papers, downward drilling by metals has so far been investigated very little. The first investigations of this appearance have been published by Begley, Brückner, Herndon, Löffler, Müller, Sandmeyer and Schmidt.

Fig 7 gives a list of the most commonly appearing metals, together with their specific gravity, melting temperature, electrical conductivity and magnetic properties.

This figure is concerned exclusively with those metals which have previously been found in the bottoms of glass melting tanks. Tests and observations have shown that corrosion by these metals is of decreasing severity in the following order: lead, brass, copper and iron.

Zinc and aluminium

Zinc appears to be excluded as a corrosion component because it evaporates at relatively low temperatures, and to date it has not been demonstrated whether aluminium has a destructive influence on the refractory material. However, it has not been shown either whether aluminium is oxised into Al_2O_3 thus reducing SiO_2 to Si. As a metal, Si could then have a destructive influence on the bottom. Similar processes are known from aluminium melting.

Metal deposits

As yet, there is no satisfactory explanation of the influence of metal deposits in electrically heated tanks. A piece of a molybdenum electrode (see fig 8), which had broken off during operation, was deposited on the bottom, and heavy corrosion of the tank bottom developed around this piece of metal. However, whether the corrosion of the bottom was the result of the molybdenum as a metal, or whether there could have been electrical fields occurring which accelerated the corrosion process, is not known.

The piece of metal was about 8–9 cm long. What is known is the influence of glass in the joints of the bottom paving with respect to the path of the electric current on the behaviour of the refractory material. It is quite feasible that metallic deposits of iron and copper, etc, on the bottom can result in uncontrollable electrical fields which contribute to the severity of the attack.

Metal silicates

In addition to the properties such as density and melting temperature, the formation of metal silicates can also be of importance during the attack on the tank bottom. For example, lead silicates are formed at about 750°C, copper silicates and silicate compounds of brass components at about 1050°C, and iron silicates at around 1170°C. According to current knowledge, bottom corrosion is largely determined by the formation temperature of these silicates, the specific gravity of the metal and the melting temperature of the metal.

Fig 9 shows the underside of a paving tile destroyed by upward drilling, and fig 10 the upper side of the same paving tile with metal deposits. Also visible is the formation in part of iron silicates drilled into the tile.

Depth of drilling

The state of the metal – liquid or solid – is also decisive for the depth of the metal drilling.

When, depending on the bottom temperature, the liquid

The list of metals in fig 7 was taken from the 'Stahl-Eisen-Calender', Germany.

metal has again solidified, its readiness to attack should be greatly restricted. This is supported by metal deposits which were found at about half the block thickness in fireclay bottoms, which were un-insulated but paved.

However, the problem again arises that measurements of the rate of attack by metal in the bottom are rather difficult. It is not feasible to determine how long the metal has accumulated on the bottom, and how far it has penetrated in a certain unit of time. Under certain circumstances, metal melts which are found in the bottom could have occurred directly after the tank has heated up, or they could be only a few days old.

Begley, Herndon and Schmidt[2] investigated the attack by various types of metal on fusion cast AZS material. The test samples had a content of 34% ZrO_2 and crucible tests were carried out at 1500°C for a period of 72 h. AZS material was selected for the tests as, according to previous experience, this material has a relatively high corrosion resistance to glass and metal. It must be pointed out here that our subsequent investigations for the development of qualities with improved corrosion resistance were based on this material.

Investigations

During these tests, the linear penetration depth of the metal drilling and the surface loss were measured by means of a polar planimeter. Fig 11 shows the results of the tests.

The differences in the sequence of linear downward drilling and in the sequence of the surface loss were attributed to the different solution behaviour of metal in glass.

As Brückner has already demonstrated the liquid metal assumes various forms, notably circular cross sections with relatively smooth surfaces, often in the shape of a button with a raised outer rim and a dished inner surface, the underside of which is in contact with the refractory materials. In the centre of the attacked area the refractory material shows a thornlike protrusion.

Other melted metals exhibit irregular shapes, generally having a thornlike downward protrusion, which has penetrated into the refractory material. Which metals tend to form 'buttons' or 'thorns' is at present not known. Nor is it yet clear which type of corrosion is more severe – circular buttons or thornlike metal protrusions.

These types of attack, *ie* glass attack by bubbles with upward drilling and glass attack by metals with downward drilling, pose the question of this paper: can this type of attack be reduced or prevented?

We have carried out crucible tests with fusion cast material and with our special materials Supral AR 90 and ISO-KB. Fig 12 illustrates the finger test of Supral AR 90, ISO-KB and fusion cast material. In order to combat the problem, the following specification was drawn up.

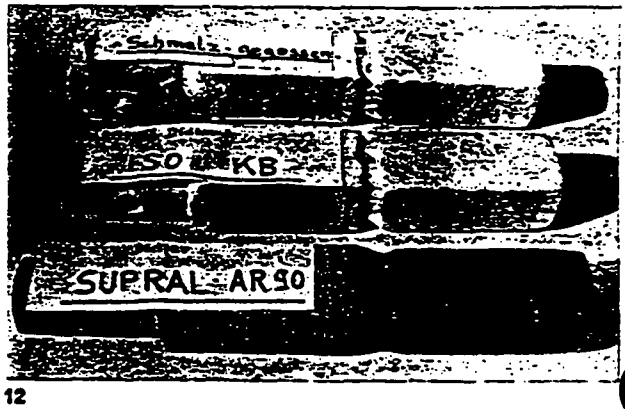
The requirements with respect to quality are high corrosion resistance to metal and metal silicate melts, low tendency to form bubbles to prevent upward drilling of the middle paving course. The discoloration is of no importance and does not appear with isolated spot-like attacks; with strong metal influence and large attack areas, discoloration should be regarded as a warning signal.

The requirements with respect to shape are plane smooth surfaces with sharp edges and accurate dimensions; rectangular form; and, depending on the manufacturing process, largest possible sizes. The size must not be obtained at the expense of planeness.

11 *Drilling effect of several metals on fusion cast: AZS-ZrO_2-Al_2O_3-SiO_2 material. Results obtained by Begley, Herndon and Schmidt.*

TEST CONDITIONS: 1500°C – 72 hours – Crucible Test
PORTION: Metal 0·177 cm³. Glass 32 Gm (Soda Lime Glass)
CORROSION MEASUREMENT:
(A) Linear Downward Drilling – mm
(B) Area Loss

	(A)	(B)
ALUMINIUM	0·00 mm	0·00
BRASS	1·39 mm	18·43
COPPER	1·29 mm	16·02
INCONEL	0·79 mm	1·82
IRON	1·63 mm	10·56
LEAD	0·95 mm	14·26
NICKEL	0·93 mm	4·88
STAINLESS STEEL No. 304	0·93 mm	10·86
STAINLESS STEEL No. 330	0·84 mm	3·46
ZINC	0·00 mm	0·00



12

For high resistance to downward drilling by metals, high corrosion resistance to glass and glass-like melts, Didier recommends high-fired, direct-bonded corundum-chrome glass contact materials such as Supral AR 90. This material has an exceptional corrosion resistance to glass, glass-like melts and to metal attack.

Choice of size depends on the thickness, *eg* 32 mm = P 4 tiles, available for 200 mm thicknesses up to WA-size dimensions (1000 by 500 by 100 mm).

References

1 R Brückner: *Einige Besonderheiten der vertikal bevorzugten Korrosion (Lochfrass).* Glastechnische Berichte 40 1967 No 10.2.371–374.

T S Busby: *The solution of refractories by molten glass.* International Glass Congress, Brussels 1965.

B L Schmidt, R S Harris: *Glass furnace melter bottom problems.* 32nd Annual Conference on Glass Problems 1971, University of Illinois.

H Jebsen-Marwedel: *Deutung der zusätzlichen Korrosion an der Spülkante von Glasschmelzen und durch Lochfrass von Blasen als Transportvergang durch permanente Umbenetzung.* Glastechnische Berichte 39 1966.

J Löffler: *Physikalische und chemische Reaktionen, die in Glasschee Glasschmelzwannen zur Ausbildung von Spülfugen oder zum Lochfrass führen.* Glastechnische Berichte 38 1965.

K H Sandmeyer and A Miller: *Laboratory studies of the glass furnace refractory structure.* Glastechnische Berichte 46 1973.

2 E O Begley, P O Herndon, R T Schmidt: *Metal drilling in tank bottom.* Cohart Refractories, Kentucky, USA.

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REFERENCE 2

Ceram. Eng. Sci. Proc., 9 [3–4] pp. 253–263 (1988)

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First Campaign of a Lead Crystal Glass Electric Furnace in Poland

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The Julia furnace produced high quality lead crystal glass over a wide range of operating conditions. Long furnace life was obtained by the unique concept of matching block electrode life with sidewall refractory life. The long campaign life more than pays back the additional cost of the tin oxide block electrodes. It is estimated that the second campaign will last at least 1 yr, with 10-12 yr being possible. A relatively simple pelletizing process was proven effective, resulting in a dust-free batch charger operation.

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Introduction

Since the discovery that lead oxide added to glass resulted in more light refraction or brilliance, lead crystal glassware has been highly regarded and sought by modern society. In earlier times this glass demand was supplied mostly from day tanks and pot furnaces. However, demand increased and people became more sophisticated about quality and consistency. They also learned that lead compounds are not as good for the environment as they are for glass characteristics.

Consequently, many regulations and restrictions have been placed on lead crystal fuel-fired furnaces with respect to air pollution, which makes an all-electric, cold top furnace the best choice. Fortunately, electric furnaces produce excellent glass quality which means there are multiple benefits associated with electric melting. Of course, there are downside factors associated with electric melting of lead glass. For example, the normal molybdenum electrode material cannot be used in lead glasses as there is a reaction between lead oxide and moly which rapidly corrodes the electrodes. This leads to many problems, such as Mo streaks, rapid electrode wear, and exudation of molten metallic lead through the furnace bottom.

It has become accepted practice to use tin oxide rod electrodes in lead glass applications, but this usually results in other problems, such as relatively short (18 to 24 mo) furnace life. The short life is associated with tin oxide rod electrodes and the ensuing relatively high current density which often causes

Reference 2-900218

early failure of an electrode. Normally, these electrodes cannot be changed without draining and shutting down—thus the short campaign life.

TECO/Elemelt, Ltd. has taken a different design philosophy with respect to lead glass furnaces. This involves tin oxide block electrodes, as shown in Fig. 1, instead of the more frequently used rod electrodes. The origins of this particular design concept are based on the single phase molybdenum plate electrode furnaces, as shown in Fig. 2, developed by Elemelt, Ltd. which have been successfully used for many years. This concept incorporates proven design features, including the following:

- -Facility for establishing a designed temperature profile from the batch crust to the high temperature melting and refining zone by suitable location and sizing of electrodes.
- -Low watt and current densities at the electrode/glass interface to prevent overheating and thus reduce local thermal convection cells, seed and bubble formation. The lower interface temperatures also reduce electrode wear and, therefore, achieve longer electrode and furnace life.

Tin oxide block electrodes have been used for many years in certain types of optical glass furnace premelters. These premelters are usually mixed energy melters, and the glasses melted include a wide range of compositions. Although tin oxide materials have been available in the United States for many yr, they were not widely used commercially until the 1970s. This is about the time that tin oxide, known as "Stannex," became available. The manufacturers developed various systems which could be used for incorporating block electrodes into furnace designs.

From studies of electrical and physical properties of this tin oxide, it was believed the material could be used as electrodes and sidewall refractories simultaneously under the right conditions. Included in the electrical studies were the electric power-induced temperature gradients through the block, electrode-to-glass interface resistances, and other relevant properties. The corrosion rate of the blocks is primarily a function of glass interface temperature, which is also a function of current density. Therefore, the surface area parameter of the electrode block is critical to having the electrode wear rate the same as the sidewall wear rate.

This design philosophy was tested in 1978, when we were commissioned to design a lead crystal furnace by the Polish Glass Industry. The furnace was located in southwestern Poland, in the Sklarska Poreba Plant of Huta Skla Krystalowego "Julia."

The design goals for this project were overall good economics, good glass quality, long furnace life (minimum 4 yr), flexible furnace operation compatible with hand working parctices, possible noncontinuous operation, and environmental acceptability.

Furnace Design

The furnace was designed to pull up to 12.0 tonne/d of 24% PbO glass. The furnace is illustrated in Fig. 3. Melting and fining rate considerations were used to determine the furnace size and configurations.

Melter

The melter was 3.340 m (11 ft) wide by 1.760 m (5.8 ft) long, a melting area of 5.88 m^2 (63 ft^2), which results in a melt rate of about 2t/m^2d (4.8

ft²/ton). The melt rate was deliberately kept low in order to achieve the desired furnace life. The electrical requirements for this particular glass were determined based on experience with molybdenum plate furnaces. The criteria used for this were maximum current and watt densities, permissible maximum operating voltages, and thermal gradient requirements. Having determined the total melter area, the glass depth needed for good melting and refining in terms of thermal gradients, together with the electrical parameters, the furnace configuration, including electrode placement and area, could be clearly defined.

The electrode configuration consisted of a number of stacks of tin oxide blocks forming the complete electrode assemblies, as shown in Fig. 3. Among the patented features was interleaving with silver foil to provide good electrical contact between individual blocks, which allows uniform current transmission to the glass. Power to the stacks was supplied by clamped connectors from the cables leading from the secondary of the transformer/regulator set. The electrodes and surrounding refractories must be installed to strict dimensional tolerances. However, each stack is surrounded on the outside cold face by a water-cooled frame as a safety device, in the event there is any glass leakage between the electrodes and the adjacent refractories [ER1711RT (AZS)].

The melter was designed for cold top operation with a relatively simple superstructure. Materials were used which could tolerate frequent thermal shock for rapid heat-up or cool-down and hot holds as required for cyclic or interrupted operation.

Throat

The throat was a submerged type constructed with AZS refractory. The rod electrodes installed in the throat were intended for use only during start-up and periods of low pull or soak. An electrically heated bottom drain was installed in the lowest part of the throat channel. This was designed for intermittent use to periodically remove any contaminated glass or metallic lead should it occur. There were no occurrences of contaminated bottom glass nor was any metallic lead found during the campaign. In the beginning of the furnace operation, the drain was used once per week, then once per mo, and now about once every three mo as a precautionary measure.

Riser

The riser, constructed with AZS refractory, again used stacked tin oxide electrodes. Temperature was low enough at this location to allow the use of plug type electrical connectors.

Working End

In order to hold costs down, the working end was designed in an open arrangement with six gathering holes. This did not allow much individual control for each work opening but was sufficient for this application.

Above-glass energy was provided by molydisilicide elements. Above-glass temperature zoning was required to maintain glass quality and consistent operation, even in this relatively small working end, i.e., 2.500 m (8.2 ft) long by 1.700 m (5.6 ft) wide.

Under-glass energy was introduced through a number of stacks of plug-connected electrodes on each side. The essential criterion for the design and arrangement was current density. In order to maintain seed-free glass, the current density had to be kept below critical values. It is significant to note that

every time the critical current density was exceeded by the operators, for whatever reason, seedy glass was the result.

The working end was separated from the riser by a submerged weir block. This was used to eliminate return flow from the working end to the melter, particularly to prevent the return of dirty glass as commonly occurs in hand gathering operations. Also, there was a surface overflow drain heated with a gas burner, intended for a number of functions:

- -Assured positive atmospheric pressure above glass, thus preventing ingress of cold air at the gathering holes
- -Continuous removal of contaminated or lower quality surface glass
- -Simple method of glass level control by ensuring a small continuous glass flow over the weir block, which was an integral part of the design of the surface drain
- -Means of maintaining a minimum glass flow at all times, even when there is little or no production, such as on weekends.

Originally, no stirring mechanism was installed in the working end. However, cord quality was less than desired. A refractory stirrer was added and cord and ream were reduced to achieve high quality product levels.

Batch Charging

A reciprocating travelling boom charger with conveyor belt was used and has worked quite well for over eight yr with relatively little maintenance. The charger is shown in Fig. 5.

Although powdered red lead (Minimum) was specified as the PbO source, it was believed that pelletized batch would not be necessary. The additional cost was thought to be unwarranted, but subsequent events proved otherwise. Dusting at the charger was unacceptably severe, even when using the water spray system, which is part of this charger design.

If a similar situation arose today and non-pelletized batch had to be used, a totally enclosed tubular vibrating charger would be specified. This, it is believed, would improve the situation, but one cannot be sure that additional dust collection or suppression equipment would not be required.

Cullet

As is normal in the hand-made crystal industry, substantial amounts of cullet arise during manufacture and processing and during non-productive periods of furnace operation. The Julia furnace has operated with 60–70% cullet and operates normally with about 50%. The furnace can also operate with 100% batch, allowing great flexibility.

Pelletizing

Shortly after start-up it became obvious that the dusting at the furnace charging end was unacceptable. Fortunately, the Glass Institute in Krakow had been doing extensive work on the batch pelletizing and could provide a disc pelletizer on short notice. TECO/Elemelt had conducted pelletizing studies in their laboratory with this particular batch using a mini-disc pelletizer. The batch was found to be easy to pelletize using only water for the binding medium. When the water-wet pellets were allowed to air dry, they formed a hard outer skin which maintained their integrity during handling and charging. When

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heated to 200°–300°C (400°–575°F), they became rock hard, but it was found unnecessary to use this additional heating process in this case.

Pelletizing on site was accomplished by using water binder and manually charging the disc pelletizer, as shown in Fig. 6. Dusting at this stage was controlled by liberal spraying of water. Pellet sizes were quite variable, depending on the operators, the variable amounts of water used, and the discontinuous nature of the operation. However, these size variations did not adversely affect melting behavior or glass quality. The pellets were acceptable, but improvements to automate the process could be made relatively inexpensively.

The immediate effect of introducing pelletized batch was to increase the melt rate and capacity of the furnace about 25% (up to 15.0 tonne/d). The capability to reduce pull drastically while maintaining a crust and good glass quality remained. Adjustments made to the batch formula also helped the low pull conditions. Until the batch formulation and other steps could be implemented, the melting area of the furnace was reduced by floating silica blocks around the edge of the melter, as shown in Fig. 7. Using such pure silica blocks did not cause any glass problems and the blocks lasted for several weeks in this lead glass.

Observations

Operation

During the first campaign, the average daily pull was 7.5 tonne/d. The pull ranged from 4.0 to 15 tonne/d. The target glass composition was as follows:

SiO_2	58.6%
PbO	24.4%
K_2O	13.1%
Na_2O	3.8%

Antimony oxide (Sb_2O_3) and arsenic oxide (As_2O_3) were used as fining agents.

There were periods of five d per week of operation, with little or no glass flow over the weekends. During such times, the charger was withdrawn, the charger opening was closed, the crust was allowed to melt, and the crown temperature was allowed to rise. With a normal batch crust, the crown temperature was in the 120°–150°C (250°–300°F) range; during soak periods it rose to about 1100°–1200°C (2000°–2200°F). Over the 75-mo period, this intermittent cycling did cause cracks in the crown but not so severely as to impede operation or necessitate repairs. The preferred method used during weekend holds was to maintain a crust and reduce pull to about 4.0 tonne/d through the surface drain.

The melter operation was manually controlled to a resistance range versus pull. The resistance values were determined by calibration at various pulls. Typical operating values for the melter are as follows:

Voltage	290 V–300 V
Current	1,550 A–1,600 A
Power	450 kW–480 kW

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Typical specific energy consumption figures were as follows:

Energy/tonne @ 4.5 t/d: 1.85 kWh/kg (1,678 kWh/t)
Energy/tonne @ 7.5 t/d: 1.5 kWh/kg (1,361 kWh/t)
Energy/tonne @11.0 t/d: 1.0 kWh/kg (907 kWh/t)

At low pulls of less than 7 tonne/d, about 11 kW is used in the throat power system.

To regulate the temperature of the glass entering the working end, the riser power was used. At 7 tonne/d the typical power was 70–75 kW. In the working end about 60 kW was required below glass with 100–120 kW above glass during normal operation.

The total power requirement for the unit (melter, throat, riser, and working end) was around 690–745 kW, and the metered power consumption, including all services (cooling water, air cooling, and batch charging), was 840–850 kW.

A batch crust thickness between 60 mm (2½ in.) and 100 mm (4 in.) was maintained in the 4–15 tonne/d pull range. The quantity of water sprayed at the batch charger played a significant part in establishing and maintaining the crust. The additional water used to maintain the crust did not require more energy. The additional energy used was apparently offset by lower heat losses through the wet crust.

Furnace Condition At End of Campaign

The Julia furnace was shut down for rebuild in August, 1985, after mo (6.25 yr) of continuous operation. The reason for stopping the campaign was that the integrity of the electrical insulation became marginal after six yr of exposure to high humidity. The insulation available at the time of the furnace construction in 1978 was not impregnated to protect against its hygroscopic properties whereas modern versions of the material are impregnated.

After cooling the furnace for repair, it was realized the campaign could have continued for a considerably longer time, even yr. In the melter, the average wear of the AZS sidewall blocks was about 55–60%; of the original 300 mm (12 in.), about 130 mm (5 in.) remained. The block electrodes had worn about 30 mm (1 in.) more than the sidewall block. The original electrode blocks were 340 mm (13.4 in.) thick and about 140 mm (5.5 in.) remained. The melter sidewall and electrode condition after drain is shown in Fig. 8. The furnace bottom looked practically new. Also, the throat and riser refractories showed little wear and the throat and riser electrodes looked new.

Refractory wear in the working end was minimal as was the electrode wear, except in one electrode block where a slice about 20 mm (3/4 in.) thick had delaminated. This is believed to have happened during a power failure early in the campaign.

Glass Quality

Glass quality was adjustable, depending on market and product requirements at any one time. For example, by altering the furnace operating parameters, seed levels could be affected. Glass quality, in end product terms, was judged to be good throughout the furnace campaign. Occasional seed or blister problems arose when the working end under-glass power was increased to such a level that the permissible maximum electrode current density levels

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were exceeded. Batch stones were not encountered, but some refractory and other extraneous stones occurred, the former associated with the hand gathering operation, the latter apparently caused by possible contamination during batch and cullet handling. Since the installation of a stirrer in the working end, plus the continuous operation of the surface drain, cord and ream ceased to be problems.

Conclusions

The Julia design concept furnace produced high quality lead crystal glass over a wide range of operating conditions. Long furnace life was obtained by the concept of matching block electrode and sidewall refractory wear rates. The furnace could be operated in a continuous or discontinuous manner, depending on production requirements. The turndown range was large, i.e., from 15.0 to 4.0 tonnes/d.

The long campaign life more than pays back the relatively high initial investment cost of tin oxide block electrodes. The Julia plant estimates that the second campaign will last at least 8 yr or more. The furnace is presently in the third yr of its second campaign. A relatively simple pelletizing process has proven effective, resulting in a dust-free batch charger operation. The concepts used in the Julia furnace design can be used for other oxidizing glasses and/or glasses requiring a high degree of quality.

Bibliography

G. B. Shaw, "Properties of Tin Oxide Electrodes for the Glass Industry," *Glass Int.*, Sept., 39–42 (1987).

J. Stanek, Electric Melting of Glass. Elsevier Scientific Publishing Co., NY, 1977.

W. R. Steitz and C. W. Hibscher, "Design Considerations for All-Electric Glassmelters: I," *Ceram. Eng. Sci. Proc.*, 1 [1–2] 92–97 (1980).

W. R. Steitz and C. W. Hibscher, "Design Considerations for All-Electric Glassmelters: II," *Ceram. Eng. Sci. Proc.*, 2 [1–2] 1–13 (1981).

Fay V. Tooley, The Handbook of Glass Manufacture, 3rd Edition, Section 6, Electric Melting of Glass, pp. 387–400/28.



Fig. 1. Tin oxide electrodes.

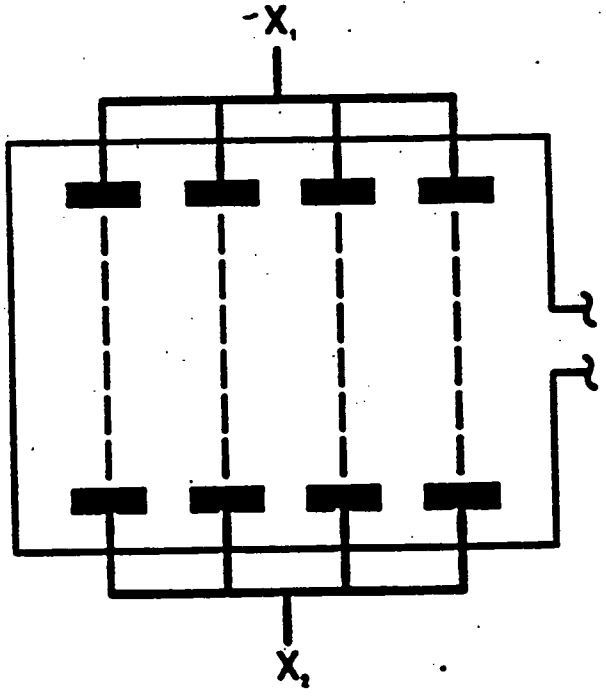


Fig. 2. Plate electrode furnace schematic.

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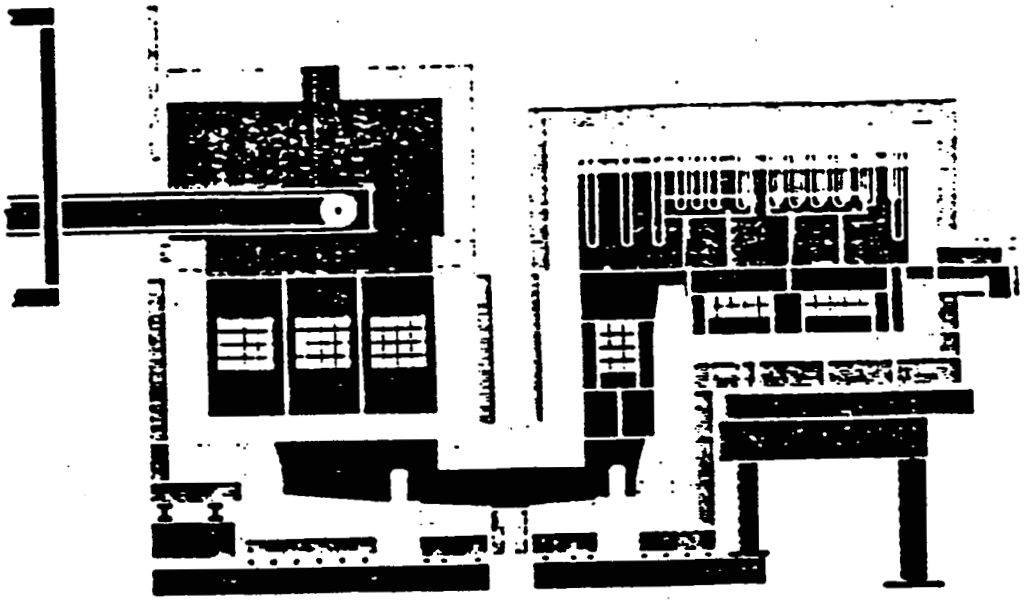


Fig. 3. Lead glass electric furnace.

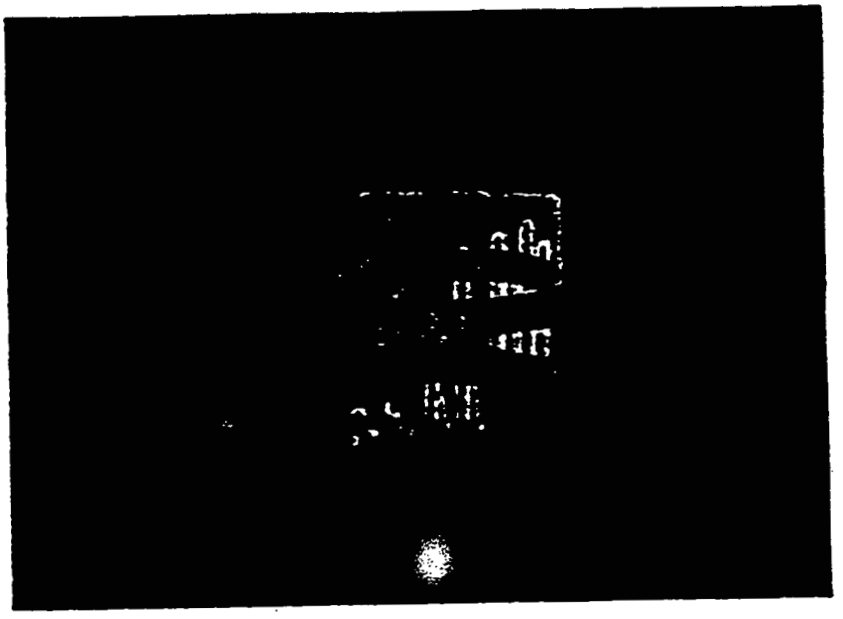


Fig. 4. Above-glass heating, working end.



Fig. 5. Batch charger.



Fig. 6. Worker charging peiletizing disc.



Fig. 7. Melter with silica floaters.



Fig. 8. AZS sidewall with tin oxide electrodes after 72-month campaign.

REFERENCE 3

Glastech. Ber. 57 (1984) No. 4, pp. 81 - 95

CORROSION OF REFRACTORY BRICKS RESULTING FROM METAL DROP BORING

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(Received on December 22, 1983)

1. INTRODUCTION AND OBJECTIVE

In the container glass industry, the refractory materials of the tank bottoms, particularly in the melting chamber are attacked through the introduction of usually nonmagnetic, low-melting point heavy metals with the recycling glass as well as through metallic abrasion and scale from glass treatment [1 through 4]. This affects the quality of the products and shortens the duration of the tank campaign.

Several corrosion paramters, such as diffusion, convection, surface tension, density, temperature and chemical composition of the melts, have frequently been thoroughly examined in the past and to this date are the subject of concentrated research [2 through 42]. It is the objective of the present paper to establish quantitative correlations among interface-convective variables (interfacial tensions and spreading pressures of variously composed glass melts with metal melts at high temperatures) and corrosion rates for the so-called metal drop boring on four highly refractory bricks frequently used today and to discuss the associated secondary phenomena.

2. INTERFACIAL TENSION, SPREADING PRESSURE AND INTERFACIAL CONVECTION

Interfacial tensions between fluid phases occur like the surface tension

Reference 3-1

versus a gas phase at the interfaces of not completely miscible liquids or melts. In a three-phase ternary system without miscibility there results, because of the three interfacial tensions, a spreading pressure which may be positive, negative or at times even zero. But in a two-phase ternary system two miscible liquids or melts also develop generally different interfacial tensions towards the third immiscible phase, so that the difference in interfacial tensions results in the spreading pressure. Applied to the system Metal Melt/Glass Melt + (glass melt with dissolved refractory material) this means

$$P_{sp} = \gamma_{M/HG} - (\gamma_{M/ffG} + \gamma_{HG/ffG}) \gtreqless 0. \qquad (1)$$

P_{sp} denotes the spreading pressure, $\gamma_{M/HG}$, $\gamma_{M/ffG}$, $\gamma_{HG/ffG}$ the interfacial tensions of metal/hollow-glass melt, metal-/glass melt enriched with refractory material, and hollow-glass/glass melt enriched with refractory material (hollow-glass melt $\hat{=}$ soda lime glass) [40]. Due to the complete miscibility of the container glass melt with the glass melt enriched or respectively saturated with refractory material, $\gamma_{HG/ffG}$ is negligibly small compared to the other interfacial tensions, simplifying the equation (1) thus:

$$P_{sp} = \gamma_{M/HG} - \gamma_{M/ffG}. \qquad (2)$$

The spreading pressure which constantly changes in time and locale because of the material transfer of refractory components from ffG to HG causes the interfacial convection, since the glass melt with the lower interfacial tension tries to surround the metal droplet. This results in the signs for spreading pressure and sense of rotation of the spreading eddies of interfacial convection. The sense of rotation of the eddies is negative if the spreading pressure is negative and vice versa [26 to 29 and 43].

2.1. Determining the Interfacial Tension between Metal- and Glass Melts

To determine the interfacial tension the method of the floating drop was used [2 and 44] where the observation of the wetting angles in a state of equilibrium with the surface tensions of the two melts is utilized according

to the vectorial equation (3) attributed to Young:

$$\frac{\sigma_1}{\sin(\alpha + \beta)} = \frac{\sigma_2}{\sin \alpha} = \frac{\gamma_{12}}{\sin \beta} . \qquad (3)$$

Here σ_1 and σ_2 denote the surface tensions of melts 1 and 2, γ_{12} the interfacial tensions between the melts, α the angle between the tangents at the surface of melt 1 and the interface and β the angle between the surfaces 1 and 2 perpendicular to the boundary line.

Equation (4) is a solution of equation (3) as per γ_{12}

$$\gamma_{12} = \left| \sqrt{\sigma_1^2 + \sigma_2^2 \pm 2\,\sigma_1 \sqrt{\sigma_2^2 - (\sigma_2 \cdot \sin \beta)^2}} \right| . \qquad (4)$$

The sign change before the inner root is a function of the value of angle β. For $\beta > 90°$ a subtraction takes place, for $\beta < 90°$ an addition. The equations are illustrated in Fig. 1.

The experiments to determine interfacial tensions were conducted in a SiC-tubular furnace with "Alsint" protective tube, which had been purged with repurified argon-hydrogen inert gas mixture. In a suitably fashioned Al_2O_3-crucible the sample metal and the glass bead were placed into the furnace. The glass bead was held in position on the center of the metal drop by an alloyed tungsten wire. The wetting angles were magnified by means of an appropriate lens system and photographed with a large-image camera. Natural radiation was used in part, and an electronic flash was also used in part. Fig. 2 shows a glass melt bead on molten lead at 1395° C photographed in this manner. Temperature at the bead was measured with a PtRh/Pt- or EL 18-thermoelement, respectively.

P.83 The angle of contact β was determined in the following manner from the photos magnified about 20 times: The surface segments in the vicinity of the triple phase line (or in the projection: of the triple phase point) were approximated by parabolas and the angle between the tangents calculated in the intersection. Analogous, the angle of contact α was determined after the image of the glass bead underside on the metal drop

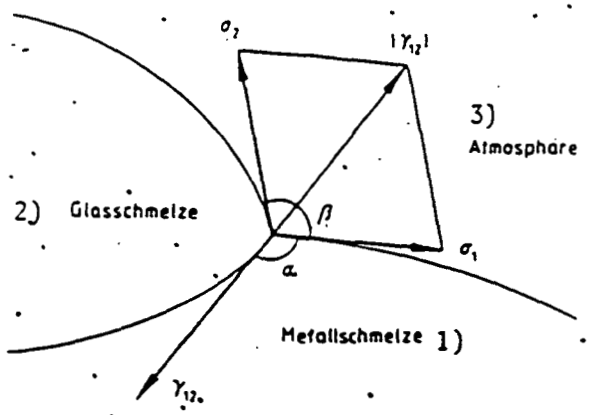


Bild 1. Parallelogramm der Oberflächen- und Grenzflächenspannungskräfte am schwimmenden Tropfen und Youngsche Beziehung. 1: Metallschmelze, 2: Glasschmelze; 3: Ofenatmosphäre, σ: Oberflächenspannung, γ: Grenzflächenspannung, α: nicht direkt beobachtbarer Randwinkel, β: direkt beobachtbarer Randwinkel.

Fig. 1. Parallelogram of surface- and interfacial tension forces at floating drop and Young-equation. 1: metal melt, 2: glass melt, 3: furnace atmosphere, σ: surface tension, γ: interfacial tension, α: angle of contact not directly observable, β: directly observable angle of contact.

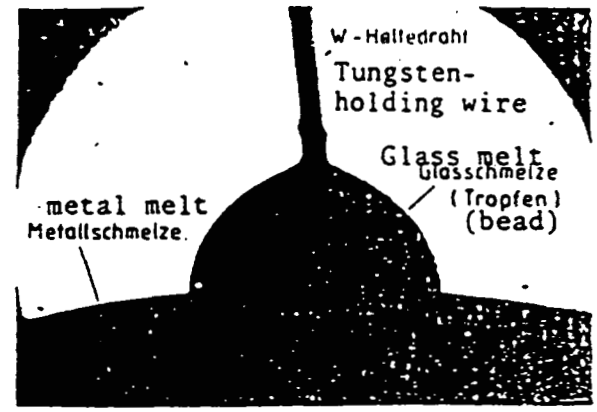


Bild 2. Benetzung einer Bleischmelze durch einen Tropfen Hohlglasschmelze, der durch einen Wolframdraht in Position gehalten wird; Versuchstemperatur 1395 °C.

Fig. 2. Wetting of a lead melt by a drop of hollow-glass melt held in position by a tungsten wire; test temperature 1395° C.

had been measured under the microscope. The surface tensions of the metal melts were found in literature references [45 through 48]. The surface tensions of the container-glass melt and of the melts saturated with refractory material [40] were measured with the bead pressure method in [41] (Table 1).

2.2. Measuring Results and Discussion

The interfacial tensions were determined between the metal melts of lead, tin, copper, silver, and gold - which in part are of technical significance and in part interesting from the aspect of systematic - on the one hand and the pure hollow glass melt and the synthesized melts of boundary layer compositions [40] on the other hand.

Fig. 3a to e show the α- and β-values of the wetting angle measurements, the α-values computed after equation (3), and the interfacial tension values computed as per equation (4) for the hollow glass melt saturated with various fireproof materials and for the pure hollow glass melt. The abbreviations

denote: HG pure hollow glass melt; SKS, CKS, Z33 and Z41 glass melts of the boundary layers at fused-cast corundum brick, chromium oxide corundum brick, corundum-zircon oxide bricks with 33 or 41%, respectively, of ZrO_2. The standard deviations of the experimentally determined angle values and of the computed interfacial tension values have been entered. In the mean, the latter are for copper 5; for gold 4;, for silver 5; for tin 7, and for lead 6%. For reasons of legibility, the standard deviation was not entered for the values calculated for contact angle α. It varies between ± <1° for copper and ± <6° for lead. The measurements with lead were made difficult by the high vapor pressure of the lead melt (approx. 100 mbar at 1400° C). The result was that during the measuring procedure a significant melt fraction evaporated and the bead of the glass melt deformed between the wire holding it and the metal drop. In addition, the precipitate dimmed the viewing windows of the furnace. The values in Fig. 3a to e largely follow the same pattern: Angles β and interfacial tensions γ have opposite trends and change to a greater degree than angles α. The calculated values of angle α generally range within the variations of the measured values, but are usually lower than the mean value of the respective α-determinations.

TABLE 1. Surface tensions at 1450° C of metal melts [45 to 48], of a commercial pure (HG) flint container glass (hollow glass) melt and one saturated with various refractory materials (SKS, CKS, Z33, Z41) [40 and 41]

	Surface Tension in mN m^{-1}
Metal Melt	
Cu	1 267
Au	953
Ag	842
Sn	381
Pb	322
Glass Melt	
HG	321
SKS	378
CKS	370
Z33	374
Z41	383

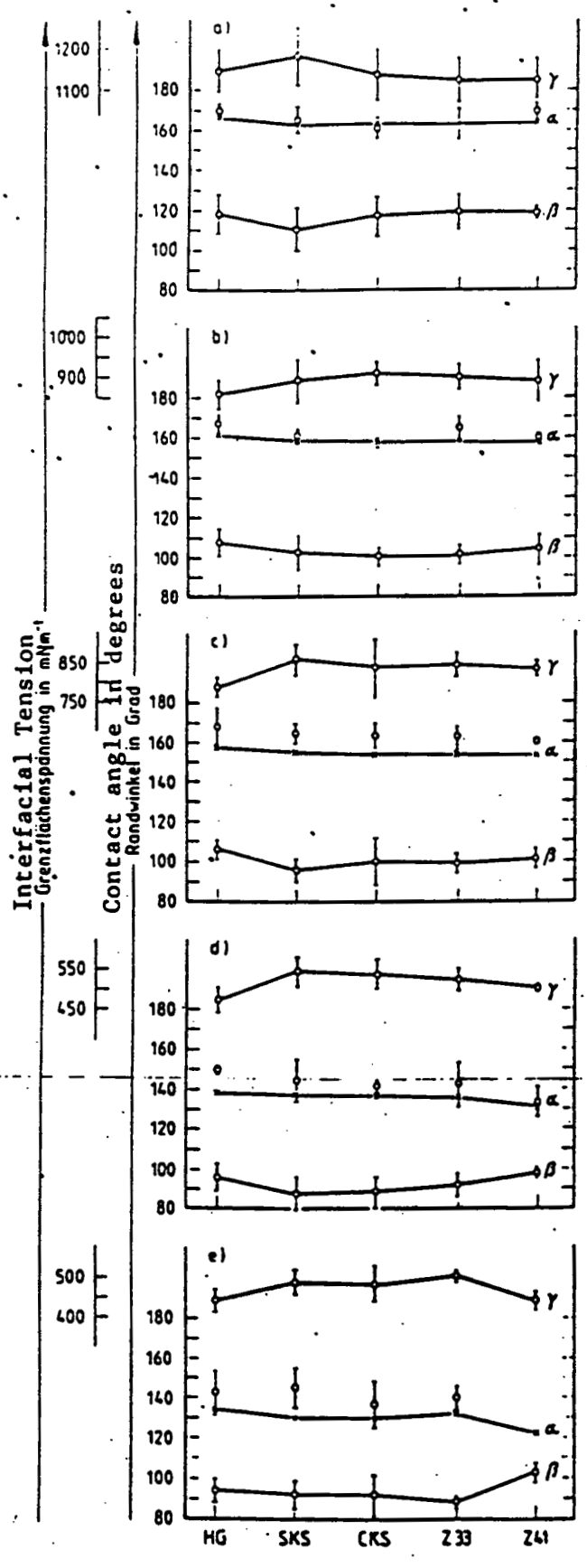


TABLE 2. Spreading pressures in $mN\ m^{-1}$ at 1450° C of melts saturated with refractor materials versus pure hollow glass melt on metal melts as interfaces and on air as surfaces

Metall	Steinmaterial, mit dem die Hohlglasschmelze gesättigt wurde – Brick material with which the ~~hollow gl. melt was saturated~~ –			
	SKS.	CKS	Z33	Z41
Cu	−40	(+ 5)[1]	(+18)[1]	(+20)[1]
Au	−37	−48	−43	−32
Ag	−72	−45	−52	−42
Sn	−74	−63	−49	−29
Pb	−46	−40	−62	(+ 5)[1]
Mittelwerte[1]	−54 ± 16	−49 ± 9	−52 ± 7	−34 ± 6
Air	−57	−49	−53	−62

1) Mean values: The values in parentheses and those against air were disregarded in taking the mean; however, they would not modify the trend of the systematics. Regarding the mean standard deviations of interfacial tensions $\Delta\gamma$ from which the spreading pressures were calculated, see Fig. 3a to e.

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Fig. 3a to e. Contact angles α, β and interfacial tensions γ, calculated as per equation (4) for five metal- and five glass melts at 1450°C. The standard deviations of the β-determinations are taken into account for t γ-calculation; the α-values calculated from Young-equation are linked to this, and the measured α-values entered with their standard eviations. Definitions are as follows: HG = hollow glass melt, SKS = hollow glass melt nriched with fused-cast corundum brick, CKS = enriched with chromium oxide corundum br., Z33/41 = enriched with zircon oxide corundum bricks (33 and 41% ZrO_2). a) copper melt, b) gold melt, c) silver melt, d) tin melt, e) lead melt.

Reference 3-6

The deviation towards smaller values is essentially a result of the fact that it was not possible to also determine from the cooled sample the bending of the metal drop surface when calculating the values for angle α, since outside of the metal surface covered by the glass bead the surface was too grooved by dendritic crystal growth. An estimate from the photos indicated that the values for angle α are smaller by 0 to about 7°. This has to be taken into consideration particularly with measurements for silver, since they were performed on relatively small metal droplets with greater surface bending.

The interfacial tensions increase in the sequence of Pb < Sn < Ag < Au < Cu (Fig. 3a to e). The spreading pressures calculated after equation (2) are listed in Table 2. In contrast to the interfacial tensions, they do not produce any systematics with regard to different metals. In part this can be attributed to the relatively large error in determining the interfacial tensions and to the fact that the relatively low spreading pressures are derived from the differences of the high values of the interfacial tensions. However, a partial reason is also that there is no significant metallo-specific effect upon the spreading pressures. However, taking the mean of the spreading pressure of a respective glass melt saturated with refractory material reveals throughout all metal melts a systematic trend according to which the spreading pressures are to be classified as follows: SKS ≈ CKS ≈ Z33 > Z41. The negative sign of the spreading pressures is in agreement with schlieren-optical photos of metal drops in glass on refractory material, where in all the systems studied there appeared eddy formations with a negative sense of rotation, therefore also with negative spreading pressure. Fig. 4 shows a typical schlieren-optical photo of copper on SKS in hollow glass melt after a test at 1450° C.

Table 2 illustrates that within the measuring accuracy there is no discernible specific influence of the metal type; i. e. all of the metals used here have an inert effect within the range of the measuring error[2]. This does not mean, however, that differences smaller than the measuring error could not exist, which could become obvious during long-term processes, as is the case with metal drop penetration.

2) The present results were obtained on the basis of repeated and very careful analysis and critical evaluation and cancel the preliminary results reported in two previous papers [50 and 51].

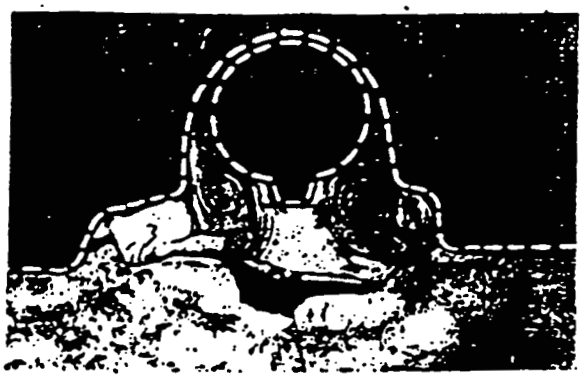


Bild 4. Schlierenoptische Aufnahme eines Kupfertropfens auf SKS in Hohlglas nach einem Versuch bei 1450 °C. Deutlich ist, besonders am rechten Tropfenrand, die Ausbildung von Wirbeln mit negativem Drehsinn zu erkennen.

Fig. 4. Schlieren-optical photo of a copper drop on SKS in hollow glass after a test at 1450°C. The formation of eddies with negative sense of rotation is clearly visible, especially at the right edge of the drop.

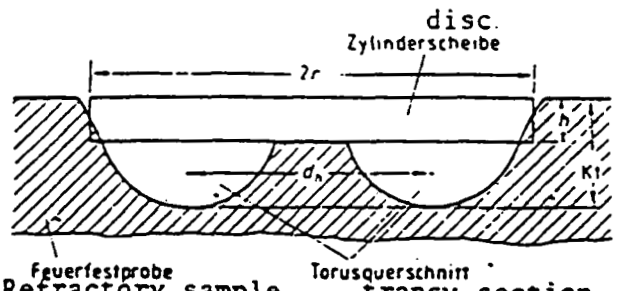


Bild 5. Prinzipskizze zur Auswertung eines Korrosionsloches mit Zapfen.

Fig. 5. Diagrammatic sketch to evaluate a corrosion hole with cone.

3. PREFERRED VERTICALLY DOWNWARD ORIENTED CORROSION (METAL DROP BORING)

Because of the interfacial convection about the metal drops resting on the tank bottom, the refractory material within the area of the metal drops is being preferentially dissolved. For the experimental quantification of pitting the five metals mentioned in section 2.2. - lead, tin, copper, silver, gold and also aluminum - were selected, with the precious metals having basic significance because of their chemically inert behavior and their specific physical properties, and the others having practical significance. The representative selection of highly refractory bricks is the same as in section 2.

3.1. Test Procedure and Evaluation

The corrosion studies for the refractory bricks with metals lead, tin, copper, silver, and gold were performed in platinum tanks filled with 300 g of hollow glass melt. Drop sizes were 15; 100; 500 and 2000 mm^3 at testing temperatures, so that corrosion holes without and with center cones [27] as well as their transitional shapes appeared. The tests were conducted at 1450° C (with

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variations of ±10 K) and lasted from 6 to 192 hours. At the end of the test the samples were cooled in air. For the evaluation the samples were sawed and polished so that the polished section showed exactly the cross section through the drop and possibly the cone. From the ten-fold magnified photograph of the polished section which was photographed together with a precision scale, the parameters Depth and Longitudinal Section of the corrosion hole were determined. The corrosion depth (Kt = Korrosionstiefe) is the distance between the reconstructed surface at the start of the test and the lowest point of the disintegration front on completion of the test. The longitudinal section (Kf = Korrosionsfläche = corrosion area) of the bored hole was determined with a pole planimeter taking into consideration the magnification factor, and the corrosion volumes (Kv = Korrosionsvolumina) were calculated; if a cone forms, this is taken into account. Bores without cones in a first approximation represent a cylinder with the volume

$$V = \pi \cdot r^2 \cdot h \qquad (5).$$

Using values Kt and Kf equation (5) becomes

$$Kv = \left(\frac{Kf}{2\ Kt}\right)^2 \cdot \pi \cdot Kt. \qquad (6)$$

P.86 The bored hole with cone is again at first approximation a toroidal cross section or a figure composed of a cylinder disc and a toroidal cross section (Fig. 5). The volume (Vt) calculated from the torus cross section is

$$Vt = \frac{1}{2} \cdot \pi \cdot Kf' \cdot d_h \qquad (7),$$

where d_h is the distance between the centers of the circle segments and $Kf' = Kf - 2\ r \cdot h$. For the compound figure the corrosion volume is equal to the sum of Vt plus the volume of the cylinder disc calculated according to equation (5). To be entered for h is the distance from the tip of the cone to the reconstructed surface and for r half of the mean width of the corrosion hole above the cone.

Reference 3-9



Bild 6. Siliciumtropfen (Durchmesser: 3 mm) in einem SKS-Hohlzylinder mit Hohlglasschmelze, der in einer Redoxreaktion bei 1450 °C aus einem Aluminiumtropfen entstand.

Fig. 6. Silicon drop (diam: 3 mm) in a SKS-hollow cylinder with container glass melt, which formed in a redox reaction at 1450°C from an aluminum drop.

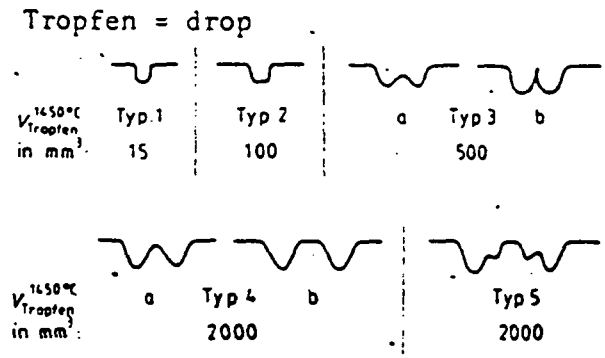


Bild 7. Ausbildungsformen der Korrosionslöcher, hervorgerufen durch Metallschmelzen in vier Tropfengrößen.

Fig. 7. Forms of corrosion holes caused by metal melts in four drop sizes.

For the second procedure the glass is tapped off the brick sample, the glass residue dissolved in hydrofluoric acid, while the metals - insofar as they do not separate - are dissolved in mineral acids. The cavities were sounded with a micrometer dial gauge. The advantage of this procedure lies in the efficient determination of the anisometric (frequently not quite rotationally symmetric) corrosion profiles. One disadvantage is the less accurate determination of Kf and Kv. The second method could only be applied for SKS, since the ZAC-bricks disintegrated in the hydrofluoric acid and in the case of CKS the glass residues with the boundary layer composition did not dissolve or else, upon extended reaction time of the hydrofluoric acid, the glass residue was also loosened from the cracks and the sample disintegrated into many small fragments.

3.2. Within the glass melt the metal drops - depending on their volumes, interfacial tensions and densities - assume various shapes and in this manner affect the geometry of the corrosion hole in the fire brick. Aluminum holds a singular position, since it does not make any noticeable contribution to the vertically downward oriented corrosion, as has been observed in tests with SKS in accordance with [3 and 4]. In addition, the observation was made in the

present paper that in a redox reaction aluminum oxidized completely and a ball of elemental silicon formed (Fig. 6). Due to its low density ($\varrho_{Si,\ 1450°C} = 2.5\ g\ cm^{-3}$) the silicon ball floats in the diffusion layer enriched with refractory material ($\varrho_{ff.-glass,\ 1450°C} = 2.5\ g\ cm^{-3}$). Therefore, drop boring by aluminum could be factored out in the studies. Silicon also does not seem to present a corrosion hazard. The other metals produced holes in the firebrick shaped as illustrated in Fig. 7.

The smallest (15 mm^3 at 1450°C) light droplets (tin, copper) form globules and produce cylindrical holes in the firebrick with a hemispherical dissolution front. The smallest drops of the heavier metals (silver, lead) are flattened out on their underside, so that the cylindrical corrosion hole exhibits a flattened bottom area. The smallest gold droplets, after tests with homogenous finely crystalline refractory material, are concave on their undersides and indicate the incipient formation of cones in the corrosion area.

In the case of the larger (100 mm^3 at 1450°C) drops the curvature is more pronounced and the incipient formation of cones is more frequently detectable in the corrosion holes which continue to be cylindrical in shape. In the case of drops 500 mm^3 in size there always appear cones within the corrosion pits which may have two physical forms. All of the metals, except gold, form with fused-cast corundum brick and with chromium oxide corundum brick hole-/cone shape type 3a, gold and at times also lead type 3b (see Fig. 7). Hole type 3b is predominant with corundum zirconium oxide brick. With regard to development the two types 3a and 3b represent two stages. In the case of type 3a the refractory material below the drop was not shielded against the dissolving attack of the glass melt to the same extent as for type 3b. With
P.87 prolonged testing time the hole shape changes from type 3b to type 3a and becomes more similar to type 2.

The really large drops (2000 mm^3 at 1450° C) frequently deviate from the rotational symmetry and produce elliptical holes with cones. The hole cross sections correspond to form types 4a and 4b in Fig. 7. It should be added here that the greatest hole depths vary within a hole and are lower for sections along the long ellipse axis than perpendicular to it. Form type 4a is predominant with fused-cast corundum brick and chromium oxide corundum brick,

form 4b with corundum zirconium oxide brick. The appearance of forms 4a and 4b depends on the shielding property of the metal melts, as is the case with forms 3a and 3b.

As became apparent even with the smallest droplets, gold with its high density effects the best shielding, while metals with markedly lower density - copper and tin - are less adequate for this.

Bricks which contain little or no glass phase - fused-cast corundum brick, chromium oxide corundum brick - have a tendency toward stronger corrosion of the cones than the corundum zirconium oxide bricks rich in glass phase, where the cone surface is not corroded even after 96-hour tests.

In the case of all brick types individual tests revealed, especially with gold and lead, the development of specific cone shapes: Instead of one cone a central cone had formed plus a cone ring which was broken at two opposing points. This shape corresponds to type 5 in Fig. 7. This shape of hole develops when the metal drop spreads above the cone and a new ternary boundary forms where the interfacial convection starts as well. The eddy and spreading processes at the inner side of the annular metal melt and at its outer side during the further course of corrosion have the effect that this annular secondary cone is partially removed. In several tests with fused-cast corundum and chromium oxide corundum brick there appeared with gold, copper and ~~tin at the edges of the corrosion holes upward oriented corrosion grooves~~ which indicate gas bubbles, i. e. a promotion of drop boring through bubble boring.

The time sequence of corrosion takes place in a similar manner for all metals on refractory material in a hollow glass melt. At the onset of drop boring the vertical corrosion rate is high since, on the one hand, the protective diffusion layer enriched with refractory components has not yet formed and, on the other hand, corrosion products can still be freely carried off. The large differences of interfacial tensions in the region of metal drop boring cause a high spreading pressure with the result that the fresh glass melt is abundantly transported along the metal drop to the firebrick. After a few hours the corrosion rate diminishes, since the diffusion layer has developed and the viscosity of the glass in the boundary layer has

increased by the factor 10 [41].

The smallest droplets with the greatest surface/volume ratio have the strongest interfacial interaction and rapidly bore into the refractory material; they sink into the boundary layer which is greatly enriched with refractory components, and their drilling action weakens because of increasingly reduced differences in interfacial tensions about the drops (Fig. 8a and b), since the corrosion products - because of their higher density than that of the pure glass melt - are being transported out of the bore with increasing difficulty as the hole gains in depth.

The large drops penetrate more slowly since a fraction of their surface, which is lower relative to their volume, is effective as active ternary boundary; furthermore they partially shield the refractory material on which they rest against the pure glass melt. However, the larger the drops the longer the ternary boundary is maintained causing the larger drops to further penetrate into the refractory material before they too sink into the boundary layer enriched with refractory material and the corrosion finally comes to a halt, as was especially evident in tests with fayalite melts [42].

The representation of the volume of the corroded refractory material (Fig. 8c and d) permits a clearer observation of the corrosion development than is possible with the representation of the depth of the corrosion hole. The nonlinear corrosion curve of the large lead drops (2000 mm^3) on chromium oxide corundum brick is interpreted to mean that during the first hours of corrosion the rather porous firebrick is being infiltrated by the glass melt. The result is, as described in [33], an accelerated starting effect in this instance as well. After a phase of slowing constant corrosion rate a cone has formed under the drop which is still enveloped by the metal, but causing the surface of the metal melt to enlarge. The interfacial convection takes effect on a larger circular area and therefore the corrosion rate begins to rise again after about 35 hours. Then the lead melt separates over the cone causing another ternary boundary to become active on the inner side of the thus created ring, whose (the ternary boundary's) spreading eddies contribute to volume corrosion in particular, but not much to depth corrosion. After

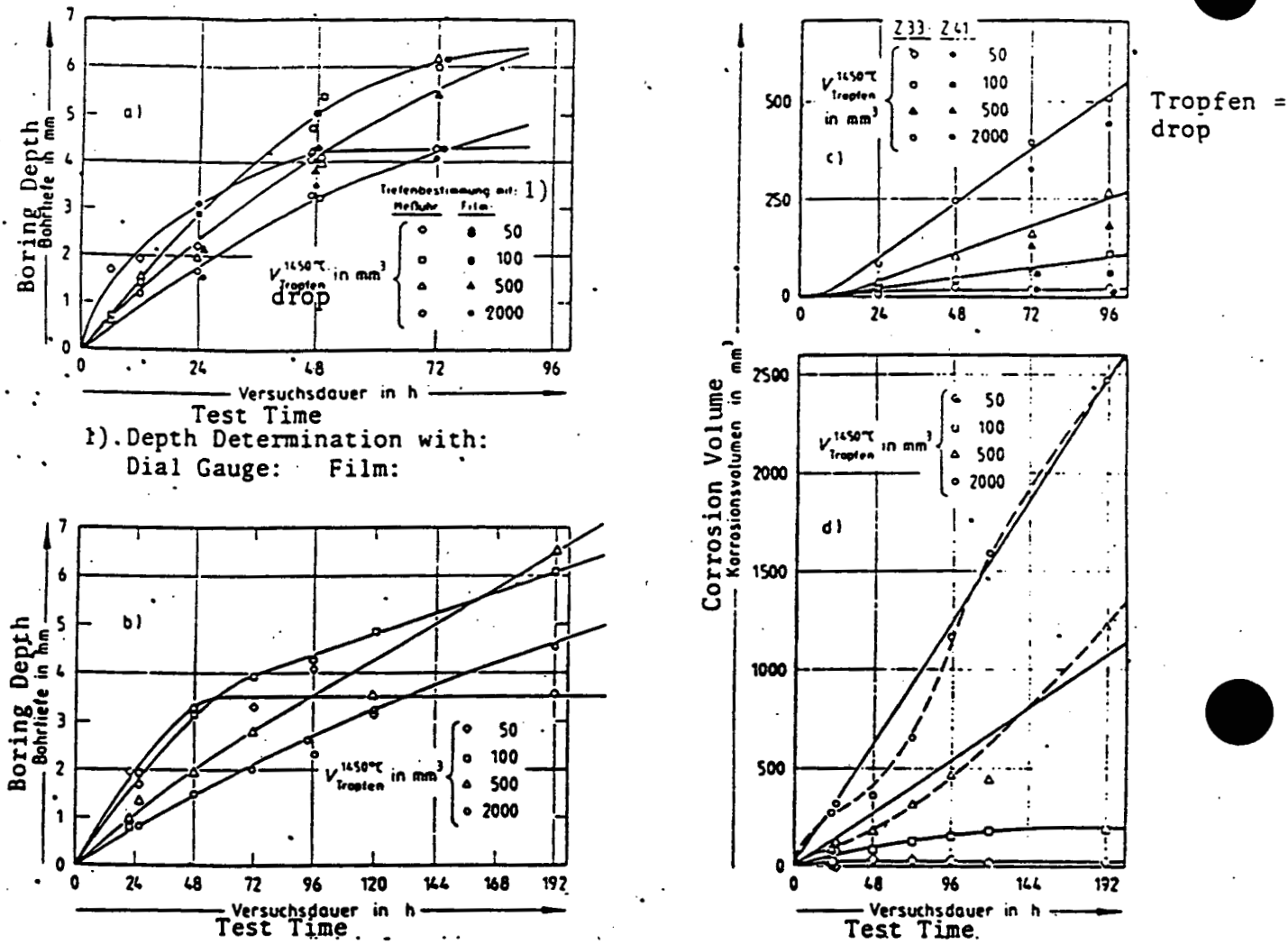


Fig.8a to d. Examples for vertically downward oriented corrosion on firebricks in the presence of various metal melts by a hollow glass melt at 1450° C: a) corrosion hole depth with a fused-cast corundum brick in the presence of a copper melt; in this Fig. a comparison is made between the results of depth determination with a dial gauge - identified by open symbols - and the results from the photographic method - identified by solid symbols -; b) corrosion hole depth with a chromium oxide corundum brick in the presence of a lead melt; c) corrosion volume with zirconium oxide corundum bricks (Z33 = open symbols, Z41 = solid symbols) in the presence of a tin melt; d) corrosion volume with chromium oxide corundum brick in the presence of a lead melt.

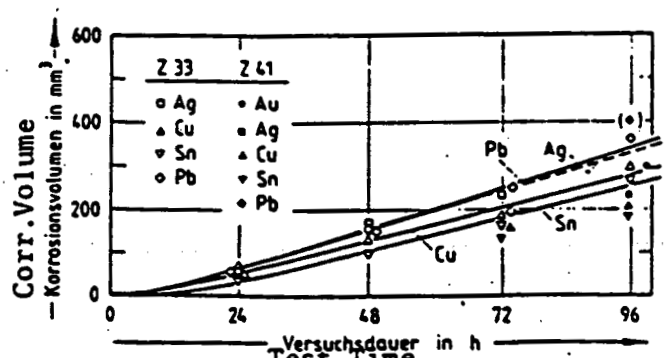


Bild 9. Vertikal abwärtsgerichtete Korrosion (Korrosionsvolumen) von Z33- und Z41-Steinen durch Hohlglasschmelze bei 1450 °C in Gegenwart von 500 mm³ großen Tropfen der Metalle Kupfer, Gold, Silber, Zinn und Blei

Fig. 9. Vertically downward oriented corrosion (corrosion volume) of Z33- and Z41-bricks through hollow glass melt at 1450° C in the presence of 500 mm^3-sized drops of the metals copper, gold, silver, tin, and lead.

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about 100 h the shielding by the glass melt enriched with refractory material increases to such a degree that corrosion is slowed down once more.

Drops with a volume of 500 mm^3 at 1450° C exhibit a particularly steady corrosion behavior. Their corrosion rates change negligibly over the duration of the tests. The drop shape remains nearly rotational symmetric in most tests, so that the measured values range within a narrow spread. The vertically downward oriented corrosion is therefore being discussed with examples for this drop size.

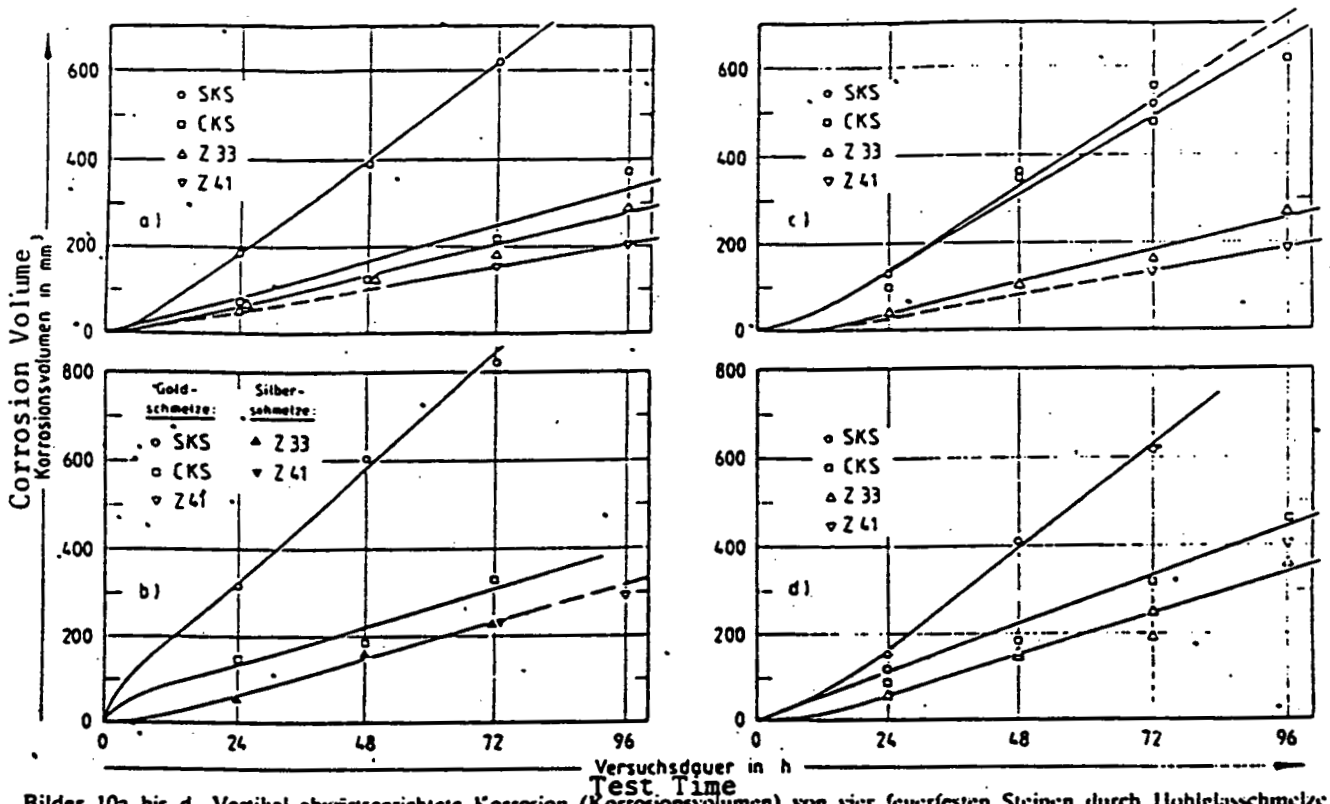
Fig. 9 shows the corrosion rates of Z33- and Z41-bricks through the hollow glass melt in the presence of the various metal drops. With these bricks corrosion developed with comparably small interfering secondary effects. The corrosion rates vary only slightly with the different metal melts; i. e. the metal melts in a first approximation perform inertly within the limits of measuring accuracy and taking into account the secondary effects mentioned in section 4.1. The
P.89 corrosion values of Z33 are in a higher range overall than those of the Z41-brick which is richer in ZrO_2; the values for SKS are equally higher than those for CKS. The higher corrosion value of Z41 with lead is neither in agreement with the corrosion results for the other drop sizes nor with the corrosion hole depths. It is a result of the frequently observed anisometric formation of lead drops which led to the fact that the longitudinal section through the sample for the corrosion evaluation was parallel to the longitudinal dimension of the drop. By contrast, the corrosion values of the other lead drops on Z41 are clearly in a lower range. No such extremely asymmetrical corrosion holes were detected for the former.

When comparing all of the firebricks one finds that corrosion rates differ substantially for drop penetration (Fig. 10a through d). Corrosion resistance increases in sequence SKS < CKS < Z33 < Z41. The starting processes of corrosion are again clearly recognizable.

4. SECONDARY EFFECTS

4.1. Formation of Cones and Bubbles

Corrosion is affected by various secondary effects. In addition to the drop size, it depends on the density and interfacial tension of the metal melt whether a cone will form under the metal drop and when, in the case of larger



Bilder 10a bis d. Vertikal abwärtsgerichtete Korrosion (Korrosionsvolumen) von vier feuerfesten Steinen durch Hohlglasschmelze bei 1450 °C in Gegenwart von 500 mm³ großen Tropfen verschiedener Metallschmelzen: a) Kupfer, b) Gold und Silber, c) Zinn, d) Blei.

Fig. 10a to d. Vertical downward corrosion (corrosion volume) of four firebricks through hollow glass melt at 1450° C in the presence of 500 m^3 drops of different metal melts; a) copper, b) gold and silver, c) tin, d) lead.

drops, the cone will penetrate the drop so that a ring is created. High density combined with a low interfacial tension causes the drop to continue to spread, so that the cone pierces the drop at an earlier stage than in the case of metal drops with low density and high interfacial tension. Thus the formation of rings was observed in prolonged tests with decreasing frequency for 2000 mm^3 drops of Au > Pb > Ag > Cu; with gold rings already occurred in part with 500 mm^3 drops. With tin there was no instance of ring formation to be detected.

While corrosion is very strong with CKS and in part also with SKS at the beginning of the test, it starts at lower rates with ZAC-bricks, since these firebricks exude part of their melting phase at the start of the test [41], which protects them well against the first corrosion attack. Only after this retarded starting process [33] does corrosion increase. Two deviations are seen in Fig. 10b and c. Corrosion of CKS in the presence of tin is about as much as that of SKS.

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This means that the corrosions rate of CKS, which normally corresponds to about the rate of ZAC-bricks, is very high. The corrosion of gold drops starts with a greatly increased beginning corrosion and subsequently remains considerable for SKS. At the end of the tests both the tin drops on CKS and
P.90 the gold drops on SKS were covered with little bubbles that were the size of pin heads for tin and 1 to 2 mm for gold. They are given the responsibility for increased corrosion since they enlarge the interfacially active area and remove melt containing refractories in addition to interfacial convection when they separate from the metal drop surface. Origin and composition of the gases are not known. They might originate from the pore volume of the refractory material; however, galvanic potentials might also be the cause for the formation of bubbles [52 to 55]. Electrolysis experiments revealed [56 to 60] that even small potential differences can cause gas bubbles.

4.2. Galvani Potentials (EMF) and Corrosion in the Outer Electric Field

According to the Nernst-equation with R = gas constant, T = absolute temperature, F = Faraday constant

$$E = \frac{RT}{2F} \cdot \log \frac{\sqrt{p_{O_2}(1)} \cdot a_{O^{2-}}(2)}{\sqrt{p_{O_2}(2)} \cdot a_{O^{2-}}(1)} \qquad (8)$$

the EMF (E) between two electrodes of a galvanic cell depends on the oxygen ion activities ($a_{O^{2-}}$) and the oxygen partial pressures (p_{O_2}) at the electrodes (1) and (2). In addition, the unknown diffusion potential principally enters at the electrodes [60 and 61]. When determining the EMF one has to make sure that

a) the cell reaction is reversible,
b) thermoelectric potentials are disconnected,
c) the state of equilibrium is present,
d) except for redox processes, there do not occur any valency modifications,
e) the electrolyte is only ion conducting, and
f) secondary reactions can be eliminated.

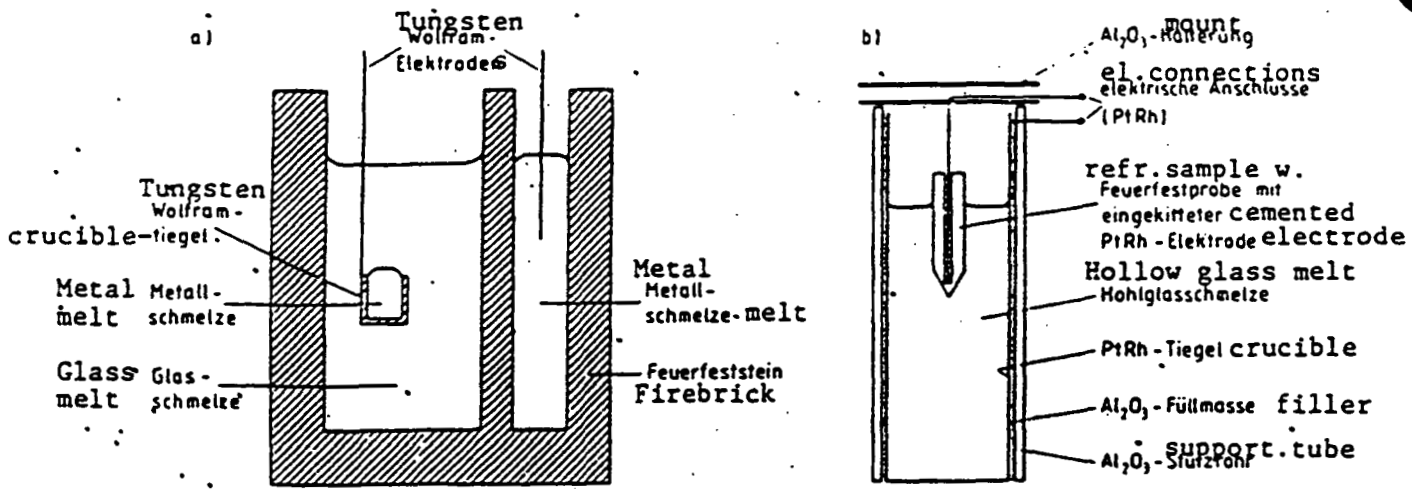


Fig. 11a and b. Sample array to determine EMF; a) series tungsten electrode/ metal melt/refractory material/metal melt/tungsten electrode; the thin wall of firebrick serves as diaphragm, b) series (Pt/Rh)-electrode/refractory material/ hollow glass melt/(Pt/Rh)-electrode; the same arrangement was also used to conduct corrosion tests in an electric field.

The first requirement can be verified with repeated measurements at a temperature which is selected among higher and lower temperatures. The second requirement is met with the lowest possible temperature gradient in the furnace. Difficulties are encountered in awaiting a state of equilibrium in tests which are to correspond to conditions in the case of corrosion of fireproof material, since the melt corrodes the crucible material. Since a conventional hollow glass melt is used for measuring the EMF, an undesirable valence change, e. g. in As^{3+}/As^{5+}-, Ce^{3+}/C^{4+}-, SO_4^{2-}/SO_3^{2-}-ratios, has to be expected. With elevated temperatures, there occurs in part an electron conduction within the glass melt, which might affect the EMF-measurement. By the same token, the measurement at elevated temperatures may be impaired by the electrical conductivity of the crucible material and chance values of EMF. Part of the tests had to be conducted under reducing conditions where the system underwent a change. The results of EMF-measurements are therefore expected to yield only minor accuracy under the stated conditions, so that merely tendencies and indirect deductions as to the corrosion processes are to be anticpated from these.

The measured EMF in the system glass melt-metal melt-firebrick is composed of several individual values. In the case of the selected crucible arrangement

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for the determination of the EMF between metal- and glass melts (Fig. 11a), the series of tungsten electrode/metal melt/glass melt/firebrick (diagphragm)/
P.91 metal melt/tungsten electrode is formed. Expressed as an addition of the single potentials the equation for total EMF is

$$E = k' + E_{W/MS} + E_{MS/GS} + E_{GS/ff} + E_{ff/MS} + E_{MS/W} \cdot *) \qquad (9)$$

Constant k' takes into account the single potentials for melt/atmosphere, glass melt/tungsten electrode and other single potentials, as for example diffusion potentials, which are not being varied when performing the tests. The $E_{W/MS}$- and $E_{MS/W}$-values cancel each other, so that for EMF there remains the equation

$$E = k' + E_{MS/GS} + E_{GS/ff} + E_{ff/MS} \cdot \qquad (10)$$

In order to minimize the changes in the chemical process of the glass melt, part of the EMF-determinations were performed in double hollow cylinders of refractory material with the larger hollow cylinder having an interior diameter of 35 mm with a capacity of about 50 g glass melt. The smaller hollow cylinder (inside diameter 7 mm), which held the metal melt, had a wall thickness of only 7 mm, so that the wall could act as a membrane. Part of the tests was executed in double hollow cylinders with a smaller volume for the glass melt, since in this instance hollow glass melt saturated with refractories was used.

The semiquantitative interpretation of the tests shows that the EMF of the pure hollow glass melt with metal melts within a few hours from 500 to 1000 mV at the start of the test declines rapidly at first and then gradually and finally reaches a value of between 150 and 80 mV. The voltage values are in a lower range for glass melts saturated with refractory material. The high voltages at the start of the test are interpreted to be the effect of the pronounced temperature drop. The EMF-measurements performed in system hollow glass melt/firebrick were taken under the aspect of learning the quantity of the system-inherent EMF-values for corrosion tests in the outer electrical field.

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*)W = tungsten; MS = Metallschmelze = metal melt; GS = Glasschmelze = glass melt ; ff = feuerfest = fireproof

The EMF-values between pure hollow glass melt and fused-cast corundum brick or fused-cast zirconium*corundum brick, respectively, with 41% ZrO_2 were determined in a Pt/Rh-crucible (diameter 40, height 120 mm) which was charged 3/4 full with refined hollow glass melt. The cylindrical test specimen of refractory material (diameter 10, height 40 mm) was cemented to a Pt/Rh-electrode and about 2/3 of it submerged in the glass melt (Fig. 11b). Measuring temperature was 1500° C. The voltage was conducted from the furnace with Pt/Rh-wires of equal alloy and recorded on a recorder. With both firebricks the voltage is positive versus the glass melt and declines after temperature equilibrium has been established.(*oxide-)

The EMF of the fused-cast corundum brick declines about exponentially starting from 650 mV to a value of 435 mV after about 2 h. From here on there follows a second nearly exponential voltage drop to the final value of 90 ± 5 mV (Fig. 12).

The EMF of ZAC-41 brick amounts to 580 mV at the start of the test. It also declines nearly exponentially to a value of 310 mV, drops again after 3.5 h and assumes the final value of 40 ± 2 mV (Fig. 12).

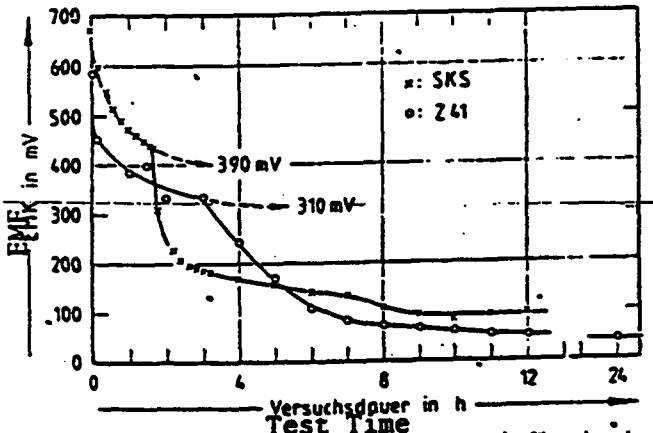


Bild 12. EMK von Hohlglasschmelze/Feuerfeststein für schmelzgegossenen Korundstein und Zirkonoxid-Korundstein (41 % ZrO_2) bei 1500 °C.

Fig. 12. EMF of hollow glass melt/ firebrick for fused-cast corundum brick and zirconium oxide corundum brick (41% ZrO_2) at 1500° C.

The first limiting values (extrapolated to $t^{-1} \rightarrow 0$, i. e. $t \rightarrow \infty$, corresponding to 390 mV for the fused-cast corundum brick and 310 mV, respectively, for the ZAC-41 brick) can be assigned to the present testing array. The cause for the second voltage drop after approximately 2 and 3 h is judged to be the infiltration of the refractory material and the formation of an ion bridge between the hollow glass melt and the Pt/Rh-electrode in the fireproof material. The

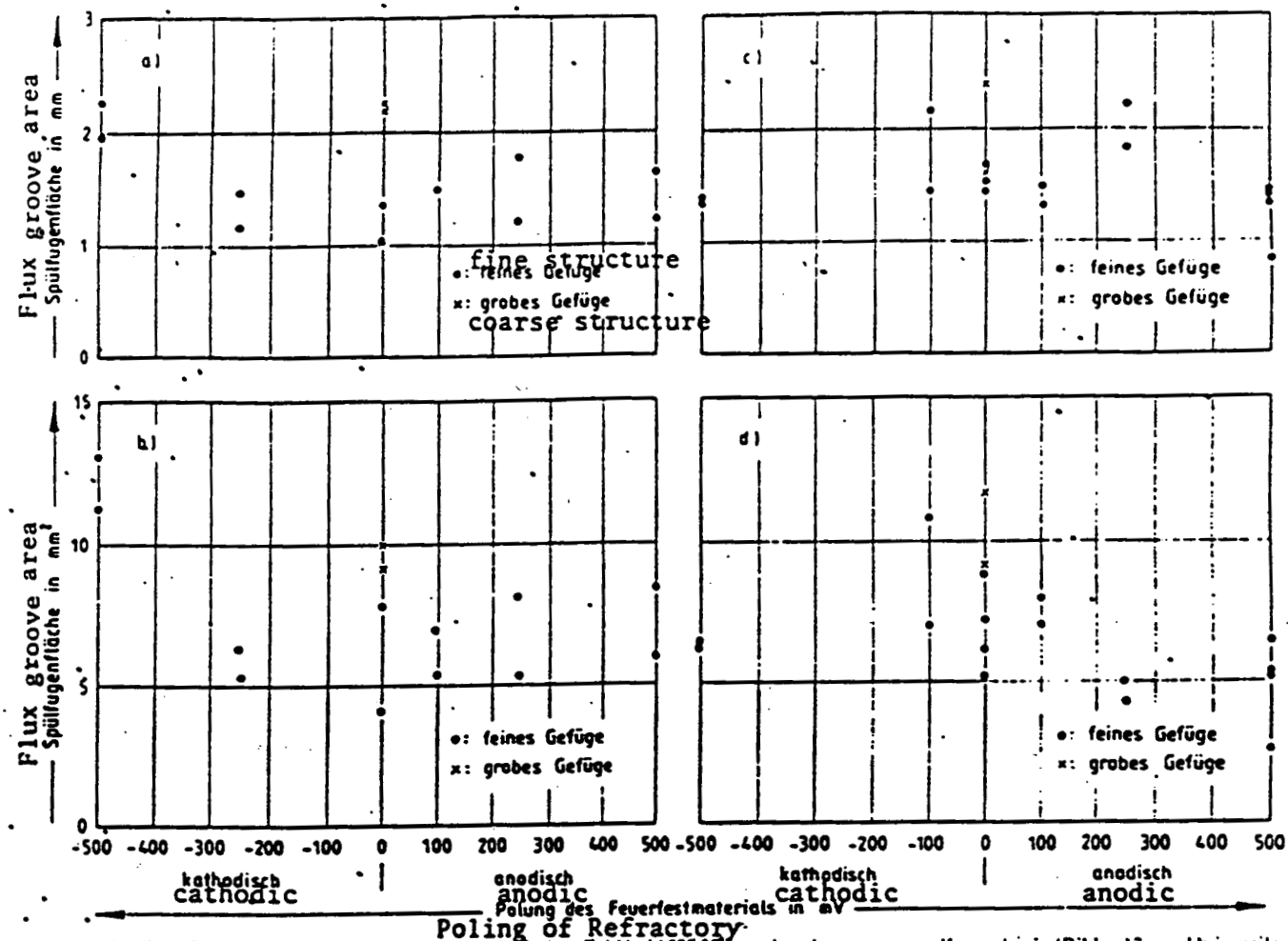
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reason for the smaller final value of ZAC-41 brick might be that the infiltrated melt has a composition more similar to the hollow glass melt than the infiltration melt of the pure corundum brick (also compare [4]).

In determining the corrosion of refractory material in a hollow glass melt in the outer electric field one has to take into account that the refractory appears as cathode and contains a higher fraction of alkalis compared to its environment. Therefore SnO_2 corrodes less strongly in a borate melt if it is poled anodic [62]. Studies should therefore be made on fused-cast corundum- and zirconium oxide bricks (41% ZrO_2) to determine whether these results can be transferred to other fireproof materials as well.

P.92 Zylindrical refractory specimen as electrodes were subjected to flux groove corrosion with voltages between -500 and +500 mV. It was not expedient to apply higher voltages since a distortion of the test results by bubble boring is to be expected because of gas generation through electrolysis. A Pt/Rh-electrode was cemented into the cylindrical specimen without making contact with the glass melt. The tests lasted for 12 (corundum brick) and 24 h (ZAC-brick) at 1500° C. After about four to six tests the 300 g of glass melts were renewed, so that corrosion would not be affected by shifts in concentration. Following the corrosion, the specimen were sawed apart, polished and photographed. The sectional photos were used to determine the flux groove depth (Fig. 13a and c) and the area of the flux groove cross section (Fig. 13b and d). It was not expedient in this instance to determine the corroded volume, since it would merely have produced a different scaling of the surface values in view of the constant radius of the samples.

The outer electric field does not significantly reduce corrosion rates with anodic and cathodic poling; rather, there seems to exist only a certain tendency that corrosion rates are increased with cathodic poling of the refractories, which is characteristic for the significance of alkali ions in corrosion. A reverse poling does not reveal any corrosion-reducing effect, on the other hand.



Bilder 13a bis d. Spülfugenkorrosion im äußeren elektrischen Feld bei 1500 °C an schmelzgegossenem Korundstein (Bilder 13a und b jeweils 12 h Behandlungsdauer) und Zirkonoxid-Korundstein (41 % ZrO_2) (Bilder 13c und d jeweils 24 h Behandlungsdauer): es ist keine Minderung der Korrosion im elektrischen Feld erkennbar; die Korrosion nimmt bei kathodischer Polung des Feuerfestmaterials SKS tendenziell zu.

Fig. 13a to d. Flux groove corrosion in the outer electric field at 1500° C on fused-cast corundum brick (Fig. 13a and b with 12 h treatment each) and zirconium oxide corundum brick (41% ZrO_2) (Fig. 13c and d with 24 h treatment each); no reduction of corrosion can be detected in the electric field; there is a tendency toward increased corrosion with cathodic poling of the refractory SKS.

5. SUMMARIZING DISCUSSION

The tests to determine metal drop corrosion rates reveal the sequence of corrosion resistance for firebricks: SKS < CKS < Z33<Z41. On the other hand, no significant differences were measured in the corrosion rate of one type of brick versus various metal melts.

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The studies on interfacial tensions between the metals and glass melts demonstrate that the spreading pressures between a melt enriched with refractory material and the pure hollow glass melt vary only within the error margins with regard to the various tested metals, which is in agree-
P.93 ment with the fact that the corrosion rates for each respective type of firebrick in combination with the five metal melts are also very close; i. e. there does not exist any dependency beyond the error margin of the metal types upon the spreading pressures and the metal drop corrosion. In other words: The metal melts behave similarly inertly as a non-reactive gas bubble; they merely provide the phase interface necessary for interfacial convection, which satisfies the condition of a fluid two-phase ternary boundary for the occurrence of interfacial convection [27 and 28]. The differences observed in the spreading pressures are therefore to be regarded only as the differences of the surface- and interface tensions between the pure hollow glass melt and that saturated with various refractories, and furthermore practically independent of the magnitude of single individual surface- and interfacial tensions which can differ greatly.

The corrosion resistance of firebricks only partially depends upon the magnitude of the spreading pressure and thus the vehemence of interfacial convection. With equal spreading pressure or equal activity of interfacial convection (Table 2 e. g. for SKS and CKS), corrosion resistance also depends upon other parameters, such as upon the effective diffusion coefficient, the saturation concentration and thickness of the Nernst diffusion layer [34]. These parameters are more favorable for CKS because of the high resistance of the Cr_2O_3-Al_2O_3-solid solution toward the hollow glass melt compared to SKS. The same holds true accordingly for ZAC-brick with regard to the high resistance or low solubility, respectively, of baddeleyite and its distribution.

The corrosion rate depends greatly on the size of the drop volume and upon whether the corrosion hole depth is being studied or the corrosion volume. While very small droplets (15 mm^3) initially have a very high corrosion rate for the bore hole depth which later declines strongly and finally returns to zero, large drops (2000 mm^3) initially show a lower rate which subsequently declines only slightly, meaning it is substantially greater

than that of the smaller drops during the later development of corrosion. On the other hand, when observing the corrosion volumes there does not occur any overlapping and the sequence for all test times corresponds to the metal drop volumes.

Superposed upon the determined consistencies are several statistical secondary effects, such as structures differing from charge to charge, inhomogeneities within the refractories as well as corrosion-promoting effect of occasional gas bubbles which originate from the porosity of the refractories and possibly are generated as a result of local or local-element type electrochemical processes. If the potential difference between the refractories and the glass melt is compensated by an electric field, no reduction of the corrosion rate is effected within the limits of measuring accuracy. This is an indication of the significance held by the high reactivity between alkali- and aluminum oxide. With reversed poling of the electric field there is a tendency toward increased corrosion rate for SKS-material.

[Translated from the German original
by I. Payne - 274-7127]

6. BIBLIOGRAPHY (TRANSLATION OF GERMAN AND FRENCH TITLES):

[1] Glass melt interferences through ferrous inclusions of foreign bodies in refractories.

[2] Some characteristics of primarily vertical corrosion ("pitting").

[4] Is it possible to avoid corrosion of metals in tank bottoms?

[5] Corrosion of refractories through glass melts.

[6] Dissolution of quartz glass in a Na_2O-SiO_2-melt.

[7] Dissolution of silica glass in a Na_2O-CaO-SiO_2-melt.

[8] Dissolution of silica glass in a Na_2O-CaO-SiO_2-melt with forced convection.

[11] Physical and chemical reactions which result in the formation, in glass tanks, of flux interstices or pitting.

[12] Interpretation of additional corrosion at the flux line of glass melts and through pitting by bubbles as carrier process through permanent wetting.

P.94 [13] Images on the generation of a flux interstice in glass melting tanks. Part I. Corundum furnace brick.

[14] Advanced bottom corrosion in tank furnaces through metals.

[18] Effect of ZrO_2 on the viscosity of glass melts containing Al_2O_3 and their significance for the corrosion of sintered corundum and sintered zirconium oxide.

[19] Electron microanalytical studies on the dissolution properties of fused-cast glass tank brick.

[20] Electron microanalytical studies on the dissolution properties of fused-cast glass tank brick. Part I: Diffusion tests after a simplified model system. Part II: Test results.

[21] Observations on the glass attack of a fused-cast zirconium corundum brick.

[22] On the glassphase in fused-cast aluminum oxide-zirconium oxide bricks.

[23] On the cause for glass issue and bubble formation on fused-cast brick at high temperatures.

[24] Fused-cast brick for glass furnace construction - properties and application.

[25] Bonded chromium corundum brick in the glass industry.

[26] Elementary processes in the corrosion of refractories by glass melts.

[27] Basic mechanisms of interfacial convection resulting in erosion on model substances and refractories.

[28] Mechanisms and systematics of elementary processes of interfacial convection.

[29] Interfacial convection and substance transport.

[30] Critical phenomena when using refractory materials for the glass melt.

[31] The partnership of glass industry - refractory industry. State of the art and development trends.

[32] Bottom protection in glass melting tanks by bonded ZIRMUL.

[33] Determination of the corrosion rate of firebrick by glass melts under conditions of free laminar density- and interfacial convection.

[34] Interactions among glass melt and refractories.

[35] Reactions between SiO_2 and a sodium silicate melt. Part 1. Dissolution of SiO_2 in the melt.

[36] On the question of the fused phase in Corhart-Zac bricks.

[37] Fluid phase in fused-cast bricks.

[39] Elevated temperature behavior of interfacial compositions formed during the corrosion of baddeleyite corundum refractories by alkali silicate glass melt.

[40] Saturation concentrations at the phase boundary of refractories and hollow glass melt.

[41] Corrosion study on refractories by glass melts under special consideration of convection.

[42] Corrosion of firebricks by fayalitic melts in the presence of iron drops.

[43] On the kinetics of material transfer at the interfaces between silicate glass- and fused salt baths and of the material transport in silicate glass melts, giving special consideration to the behavior of Na_2SO_4 and its decomposition products.

[44] Physics and chemistry of interfaces.

[45] Interfacial tension measurements on molten metals.

[49] Glass. Nature, structure and properties.

[51] Influence of various parameters upon corrosion of refractories by glass melts.

P.95 [52] Electrochemical measurement of the oxygen partial pressure in glass melts. Oxidation equilibrium studies.

[53] Study of the contact phenomena between glass and oxide at high temperatures by measuring the electric potential.

[54] On the causes of electromotive forces between glass systems.

[57] The influence of atmospheric oxygen upon the formation of bubbles in the galvanic series platinum/glass - refractory/platinum.

[60] Glass technological manufacturing defects.

REFERENCE 4

2 are given in Fig. 4, which shows that the influence of Cr_2O_3 on the ultra-violet absorption of glass is negligible.

Chromium oxide even seems to have a reducing influence on the ferric ions in glass, as can be seen from the results recorded in Fig. 4 for base composition 2 with additions of 0·02 per cent. Fe_2O_3 and 0·015 per cent. Cr_2O_3. In consequence of the solarisation process, the transmission of this sample returned almost to the same values as were found for this glass without chromium oxide. These results confirm again that the glass without chromium oxide.

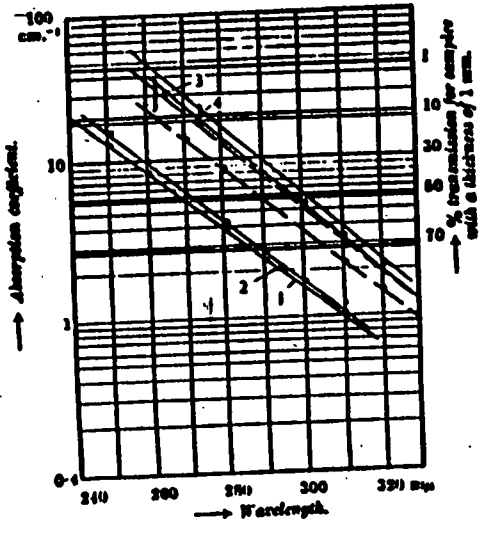


FIG. 4.

The Influence of Chromium Oxide on the Ultra-violet Transmission of Base Composition 2.

From left to right the curves are :—

1. *Without either* Fe_2O_3 *or* Cr_2O_3.
2. *Containing* 0·04% Cr_2O_3.
3. ——— 0·02% Fe_2O_3 + 0·015% Cr_2O_3 *before solarisation.*
   ——— *The same after solarisation.*
4. ——— 0·02% Fe_2O_3 *before solarisation.*
   ——— *The same after solarisation.*

influence of chromium oxide on the transmission properties of glass in the ultra-violet is negligible. For this reason studies on the positions and valency states of chromium ions in glass were not continued.

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REFERENCES.

1. J. E. Stanworth, this *J.*, 1950, **34**, 153 T.
2. O. Gott, *Glastech. Ber.*, 1951, **24**, 36.
3. J. M. Stevels, *Philips Research Reports*, 1948, **3**, 325.
4. J. H. van Vleck, *Theory of Electric and Magnetic Susceptibilities*, Oxford, 1932, 285.
5. W. A. Weyl, this *J.*, 1943, **27**, 133 T.
6. J. M. Stevels, *Verres et Réfract.*, 1948, **2**, 4.

VIII.—*Some Distinctive Contours Worn on Alumina–Silica Refractory Faces by Different Molten Glasses: Surface Tension and the Mechanism of Refractory Attack.*

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Abstract.

Characteristic types of attack produced isothermally by some mobile glasses on alumina–silica refractories are listed in Table I. Holes and scores worn beneath the glass are narrower than arches formed above. The different kinds of attack are explained by hypotheses involving surface forces. Some complications often found in manufacture (coarse refractories, large temperature differences etc.) were excluded as far as possible, in order to obtain a clearer picture of the main types of refractory attack.

TABLE I.

Different Types of Refractory Attack.

Nature of Glass.	Distinctive Refractory Attack.	Figures in Text.
Non-spreading, large surface tension.	Everywhere below glass-line.	2, 3
Wetting.	Pin-holes.	5
Spreading.	Horizontal row of shallow archways above glass-line.	6, 10, 13
Small surface tension.	Row of flux-line pits.	12, 13
Dense, small surface tension.	Bottom pitting.	16
Reducible to metallic beads.	Bottom drilling by metallic buttons.	19
Dense, small surface tension.	Set of downward scores.	20, 21

I. SURFACE FORCES AND GLASS TECHNOLOGY.

The tension, more precisely the tangential tensile force, in a liquid surface is called surface tension. To stretch the surface this force must be overcome. It is the cohesive forces in the liquid which cause surface tension. Thus when blowing a bubble, work is needed to drag liquid continuously into the ever-growing surface. Cohesion, manifesting itself as surface tension, accounts for the compact shape of drops.

The greater the cohesive forces in the liquid, the greater is the pull required to stretch the surface, and the greater is the surface tension. For liquid/gas interfaces there is usually an increase in surface tension on passing from easily evaporating liquids like laboratory solvents to salts, commercial glasses and metals. Some examples are given in Table II.

The surface tension of a glass depends on its composition. In glass

Reference 4-1

eyes or glaze patterns the boundary between different glasses is blurred if the surface tensions be too unbalanced. With commercial glasses, surface tension is small for enamels and mobile solders but large for slags. Mobile solders and enamels are rich in powerful fluxes (alkali,

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TABLE II.

Approximate Surface Tensions of Liquids in Air.

Liquid.	Surface Tension (newtons/metre).	Liquid.	Surface Tension (newtons/metre).
Petroleum	0·025	A silicate glass.....	0·3
Water	0·07	Mercury	0·5
Common salt	0·10	Silver.....	0·8

lead oxide, boric oxide, fluoride), while slags * have plenty of polyvalent metal oxide but little further flux.

Besides cohesion within a liquid itself there is adhesion between the liquid and other bodies as when syrup sticks to a spoon. Where a liquid meets a wall there is adhesion. If this adhesion overcomes the cohesion of the liquid, then liquid spreads up the wall; this can occur with molten glass against ceramics. But if cohesion predominates, then the upper surface of the liquid will be drawn downwards on approaching the wall, as happens with molten glass against carbon.

A slag glass which attacked ceramics aggressively gave a plunging meniscus when founded in a carbon pot. The carbon pot was protected outside by a refractory sheath with lid. After teeming, no glass was left sticking to the carbon, nor was there any attack. But the glass was darkly coloured, and iron had been reduced to metallic beads.

Surface forces operate throughout glass-manufacture, as for example in the following processes, which are listed without elucidation or discussion: dressing of sand by froth-flotation; moulding, drying and vitrifying of refractories; compacting of damp batch; "atomization" of fuel oil; sintering of hot batch; fining; drawing of sheet, tube and fibre; unwanted rounding of moulded sharp edges; melting-off; glass-blowing; sealing; fire-polishing.

During manufacture it is in the furnace that temperatures are hottest. Here the glass is most fluid and therefore offers least resistance to cohesion and adhesion. The purpose of this memoir is to show how such forces help to explain the attack of molten glass on refractories.

II. METHOD OF EXPERIMENT.

During recent years the author has founded a variety of glasses in open pots for industrial purposes. For the hundreds of exploratory melts which formed most of the work, pots of ½-litre capacity were usually used. Often different kinds of refractory attack were encountered with

* Metallurgical slags usually contain less than half silica, with alumina and much lime: thus an [illegible] blast-furnace slag gave 34% SiO_2, 14% Al_2O_3, 43% CaO and 7% (Mg, Mn, Fe) [illegible]phide being expressed as oxide.

different types of glass. Attack by the more fluid glasses was most marked, and was examined further.

Open pots were slip-cast and refractory fingers tamped from -250μ mixtures composed partly or entirely of calcined kyanite ($Al_2O_3.SiO_2$) and plastic white clays ($Al_2O_3.2SiO_2.2H_2O$). Such fine material passing through a screen of sixty meshes to the inch gives a closely textured refractory surface which reveals delicate kinds of attack that might be masked by using coarse grog. Usually rather more than half by weight of the refractory composition was alumina.

To increase their resistance to the mobile low-silica glasses melted, the pots for founding were matured at 1500° C. Test-pots and refractory fingers were arched to the temperature of the trial.

The molten glass was founded free from batch and seed, and was then ladled into a pre-heated test-pot of a few tenths litre capacity, sometimes with a refractory finger resting obliquely between the bottom corner and the edge. Tests were often carried out at about 1200° C. Roomy furnaces heated by town's gas enabled a fairly even temperature to be maintained in the small pots used. Here the glass was not markedly hotter on top as it is, for example, in an industrial fuel-fired tank furnace. The glass doubtless absorbed a trace of sulphur from the furnace gases.

During the trials there was usually a slow fall in glass level mainly caused by pot solution, which sometimes became quite noticeable. To accentuate any pattern formed on the refractory, the glass was often coloured; cobalt oxide, for example, being used sometimes to impart a blue colour. After testing the glass was normally teemed, but occasionally the pot was left to cool in the furnace.

The fluidity of the glasses tested discouraged viscous drag, leaving them responsive to unbalanced surface and "vitrostatic" forces. As will be explained later, this then led to clear-cut refractory attack, particularly before any complications arose from changing conditions. Also uniformity of temperature was encouraged by the easy-flowing glass.

III. SOLUTION OF REFRACTORY IN MOLTEN GLASS.

Molten glass gradually dissolves alumina-silica refractory. The glass, besides becoming richer in alumina, is also diluted by the refractory. Because of this dilution there is a decrease in the proportion of the fluxes and commonly of silica. The proportion of silica in the dissolved refractory is not usually sufficient to maintain, in the diluted glass, a silica content at seventy or more per cent. With many of the low-silica glasses studied in the present experiments, the change of silica content was only slight. Dissolved refractory thus increased alumina at the expense of fluxes and perhaps of silica.

The properties of solvent glass and refractory solution are contrasted in Table III, and will now be discussed.

Increase of alumina at the expense of the corrosive fluxes raise[s] [illegible]cosity

Reference 4-2

and lessens chemical attack on the refractory. Consequently, the viscous film of solution which slowly forms at the refractory face gives some protection against the solvent glass.

TABLE III.

Silicate Glass and Alumina–Silica Refractory.

Solvent Glass.	Solution of Refractory.
Markedly more fluid.	Markedly more viscous.
More erosive.	Less erosive.
More corrosive.	Less corrosive.
Lesser cohesion.	Greater cohesion.
Lesser surface tension.	Greater surface tension.
Penetrates easier into pores.	Penetrates more slowly into pores.

The viscous aluminous film differs in density from the main solvent. In ordinary manufacture this film is denser with a light * glass like high borosilicate of low basic oxide content, but the lighter with a dense glass like low-silica lead glass.

In a dense glass at a uniform temperature the lighter solution is pushed slowly upwards by unbalanced vitrostatic pressure. In a less dense glass the process is reversed. To counteract accumulation and withdrawal of glass, currents, whatever their cause, are accompanied by return currents elsewhere, thus leading to a continuous circulation.

The contributions of different glass components to the total surface tension are listed by A. Dietzel.† There is a decrease in surface tension when silica is replaced weight for weight by the aggressive fluxes potash, boric oxide, lead oxide, soda or fluoride (reckoned as silicon fluoride), and an increase when replaced by the polyvalent metal oxides alumina, magnesia or lime, the rise with alumina being very marked.

As already mentioned, dissolved refractory increases the alumina at the expense of fluxes. Therefore refractory solution increases surface tension unless circumstances be quite exceptional, as when slags are in contact with less aluminous refractory.

Adhesion also affects refractory attack, but unfortunately little is known of its variation with glass composition. As yet the strengths of adhesion to refractory of solvent and solution cannot be properly contrasted.

It is explained later how the slow dissolving of refractory and continuous withdrawal of the solution thus formed from the face lead to continuous processes of refractory attack.

IV. Overall Attack.

Low silica slag glasses containing little or no alkali are employed for insulating and textile fibre, and welding fluxes. They are quick-setting and need to be tested up to 1500° C., at which temperature they are fluid

* To avoid repetition the words "heavy" and "light" are used to mean dense and less dense.

† A. Dietzel, "Praktische Bedeutung und Berechnung der Oberflächenspannung von Gläsern, Glasuren und Emails," *Sprechsaal*, 1942, 82.

and aggressive.* Types of attack described later were eliminated because the slags investigated were reluctant to creep up an alumina–silica face (see Fig. 1), and because their large surface tension was little altered by dissolved refractory.

The large proportion of corrosive basic oxides causes pronounced and general pot-attack everywhere beneath the glass, as illustrated in Fig. 2. Falling glass level leaves the side-wall slanting backwards at the flux-line (Fig. 3). If the surface tension be altered by refractory solution, then surface forces may cause intensified attack near the glass-line, as happened with other glasses (see, for example, Sections VI and VII).

The somewhat lighter solution against the wall moves upwards, while in the interior a compensatory current of solvent flows downwards. The speed and position of this downward stream depend on the conditions. Attack is aggravated where the stream impinges on the pot face.

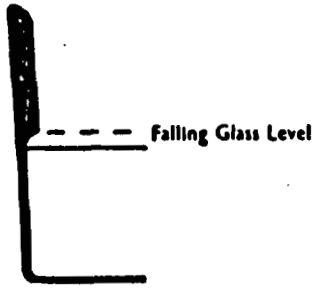


FIG. 2.

Overall Attack by Non-spreading Glass of Large Surface Tension.

V. Pin-holes.

Unlike slags of the kind just mentioned, most other glasses, particularly those of very small surface tension, wet alumina–silica refractory. If the refractory be porous from inadequate firing, then it soaks up mobile glass by capillarity (see Fig. 4).

Corrosion is resisted better by aluminous than by siliceous refractory compositions. But if green siliceous and aluminous pots be arched side by side so as just to mature the siliceous body, then the aluminous body still remains porous with but little glassy matrix, and is more easily penetrated by glass.

Fig. 5(*a*) shows pin-holes worn by enamel on entering the pores of under-fired refractory. The enamel as it oozes further, dissolves material from the body and becomes more viscous. Eventually aluminous spots may collect on the outer wall as in Fig. 5(*b*).

Even with a well-vitrified refractory, pin-holes may be formed at the ferruginous darker specks on the face. Such pin-holes were formed for example when a glass of percentage composition 71 SiO_2, 27 B_2O_3 and 2 alkali was founded at 1500° C. or hotter.

* The term "aggressive" is used to include both corrosive and erosive.

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Reference 4-3

VI. Arching Above the Flux-line.

Leadless low silicate, dense barium crown, lead barium borate, boric oxide and many other glasses spread up the refractory wall to form "arches." As shown in Fig. 6 (*a*) and (*b*), such arches vary considerably in size and form. This remarkable phenomenon depends neither on the density nor the depth of the glass, but can be explained in terms of surface forces.

It is necessary to remember that:

(*a*) the boundary between two liquid films is drawn towards the liquid of greater surface tension; and

(*b*) because of cohesion a liquid, given the opportunity, tries to gather into a compact shape (this lessens the surface area, thus decreasing the free surface energy).

A film of glass spreading up the pot side from the meniscus dissolves refractory and loses fluxes by evaporation. Consequently, as it creeps

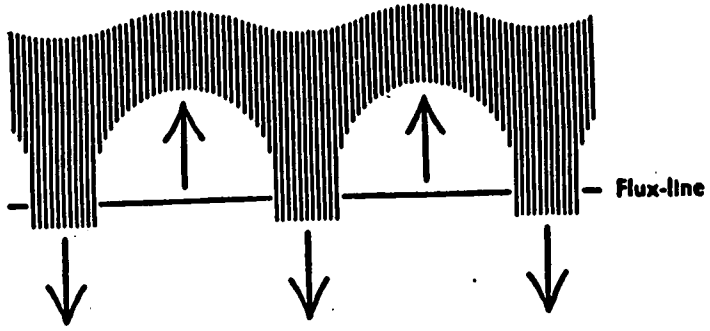


Fig. 7.

Circulation of Glass during Arching.

upwards, the film becomes more viscous and increases in surface tension. The viscous glass then draws more of the fluid solvent glass up from below.

In this way a band of aluminous glass gathers round the wall a centimetre or two above the glass-line; this then shrinks at intervals into waists, just as a jet of liquid breaks into drops. Fresh glass still wells up the face from below, swelling the bulges between the waists until the aluminous glass trickles down again into the main glass.

Eventually arches of viscous glass are left side by side around the wall at intervals of perhaps a centimetre or more. Boric oxide itself gave arches at 2·5-cm. intervals.

There is a continuous circulation of glass up inside the archways and down the columns back into the main glass (Fig. 7). The mobile glass from below scours a shallow depression on the refractory face inside each archway. [illegible] the viscous columns the wall is attacked to a less extent. A [illegible]d flux-line attack results when columns stretch down-

wards and shield ridges at the glass line (Fig. 8). The refractory attack within the archways is usually only feeble, the clear definition of the arches being due to the thicker layer of viscous glass.

Arching is encouraged by conditions which favour wetting, such as boric and lead oxides in the glass, and an oxidising atmosphere.

The clear green oxidised film which forms on the surface of copper ruby glass spreads easier than the main ruby, as can be seen by heating fragments of the glass on a flat refractory plate. In a reducing furnace atmosphere the glass melts down to a dark-red spot as in Fig. 9(*a*); but in an oxidising atmosphere the red spot becomes surrounded by an easier-spreading green fringe, as in Fig. 9(*b*).

Fig. 10 shows conspicuous green arches formed by copper ruby having a green surface film. These are similar to the arches formed when the same batches are founded under oxidising conditions to give a wholly green glass.

Arches arising solely from selective evaporation are produced by silico-borate melts in a platinum dish, and also by wine or spirit in a glass (Fig. 11). In these examples there is evaporation of flux and alcohol respectively as the liquid spreads up the wall, leaving residual arches and columns of increased surface tension.

Here might be mentioned surface adsorption in solutions. At the surface there is a detectable excess of those components which reduce surface tension. So that with glasses there is a readier migration from the interior of potash, boric oxide, lead oxide, soda or fluoride. This migration helps to oppose the loss by preferential evaporation of these fluxes from the surface.

VII. Flux-line Pits.

Another striking yet more destructive kind of attack is the etching after some minutes of pits a couple of millimetres or so in diameter, around the flux-line as illustrated in Figs. 12 (*a*) and (*b*). This pitting occurs with fluid glass of small surface tension. As with arching, it is still encountered with shallow pools of glass a few millimetres deep, and occurs irrespective of density with very light glasses containing boric oxide (including boric oxide itself), with high-alkali low-silicate containing alumina, lime or lead oxide, and with very dense optical glass. This flux-line attack occurred in these experiments even when the glass was not markedly hotter on the top surface, in contrast, for example, to the conditions in a tank furnace.

It is immaterial whether the solution formed at the refractory face is lighter or heavier than the solvent glass. Thus dense lead glass and light boric oxide itself both give flux-line pits as well as arches, although dissolved refractory lessens the density of the first and increases that of the second. Flux-line pitting is often accompanied by arching (see Fig. 6(*a*) and Fig. 13), and in the furnace brisk movements are so[illegible]times seen at and above the glass-line at intervals round the wall.

Reference 4-4

Solvent glass at the meniscus, besides creeping up the pot wall, also tries to creep downwards where it meets the layer of aluminous solution against the submerged face. In the case of denser glasses this aluminous layer is slowly moving upwards. Just beneath glass level the aluminous solution drags away from the pot side and gathers into threads because of its greater surface tension.

Jets of solvent glass penetrate between these threads and scour the side-wall at the flux-line, producing an aluminous film on the refractory face which is continuously drawn by cohesion into the surrounding aluminous solution (Fig. 14). The jets of solvent eat a horizontal row of holes, while from the ridges between flows the protective solution.

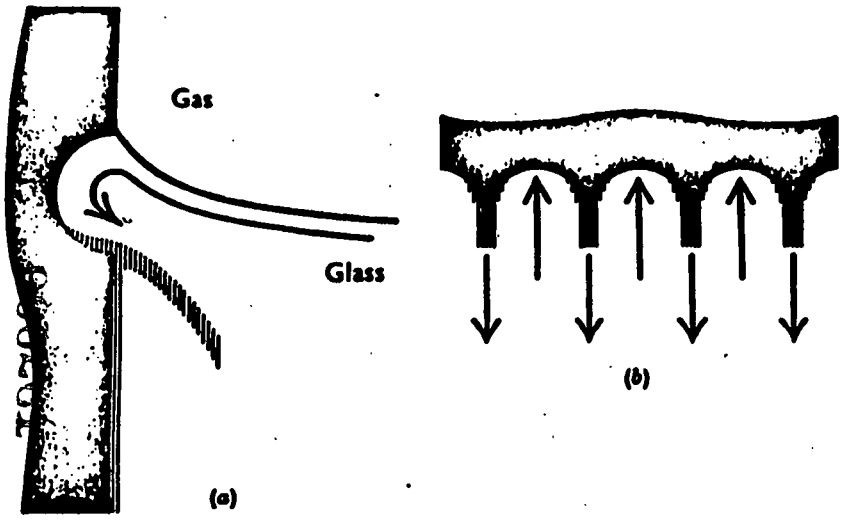


Fig. 14.

Mechanism of Flux-line Pitting: (a) *vertical section;* (b) *horizontal section.*

After a while with enlargement and coalescence of the holes, consequent lengthening of the flux-line periphery and derangement of the periodicity, changes in temperature and so on, individual pits become obliterated, leaving a groove at the glass-line. As the glass level falls there is formed on the lower ledge of this groove a new series of pits, visible at the finish after teeming.

With boric oxide glass there is very marked flux-line attack, including pitting. Here the aluminous solution is denser than the solvent, and it sinks down the wall, drawing fresh solvent towards the glass-line. Apart from this, the mechanism of the flux-line pitting resembles that just described for denser glasses.

Pits are sometimes eroded down the side-wall right beneath glass of quite small surface tension. For dense lead glass such holes were few and isolated, but for high potash borosilicates and very light (nearly

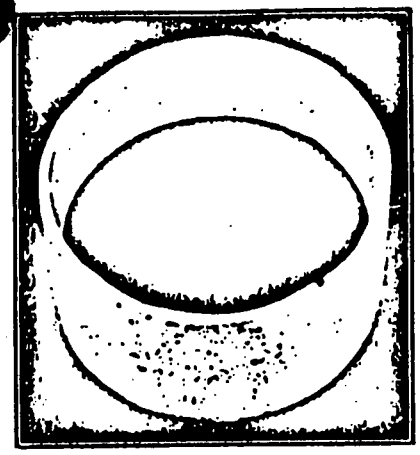


Fig. 1.

Untreated Pot with Glass Slug in the Bottom. The slug has not spread up the sides.

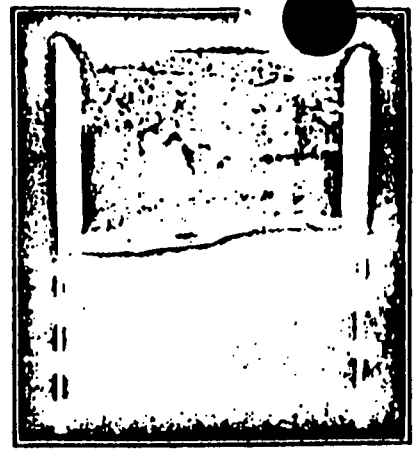


Fig. 3.

Wearing Back of Flux-line Refractory by Glass Slug.

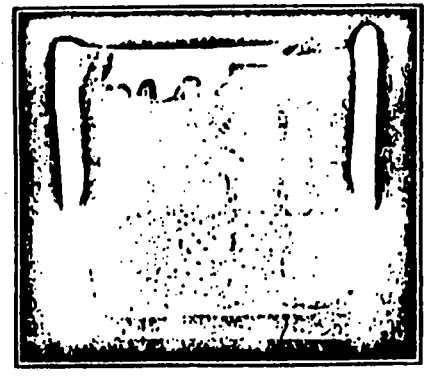
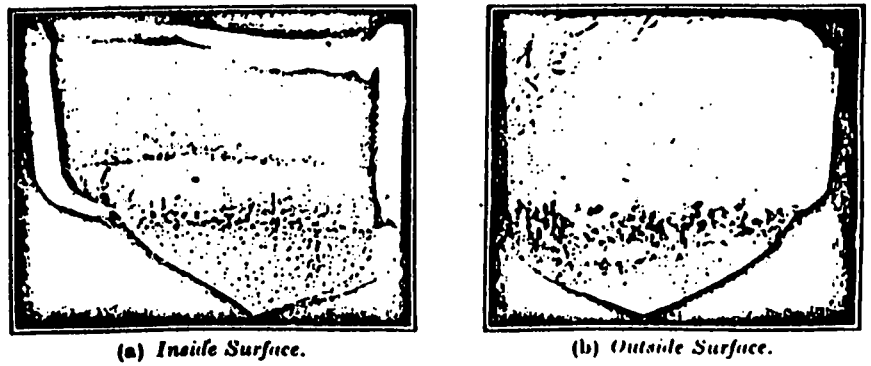


Fig. 4.

Section of Pot Soaked by Mobile Glaze.



(a) *Inside Surface.* (b) *Outside Surface.*

Fig. 5.

Under-fired Pot after Penetration by an Enamel.

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Reference 4-5

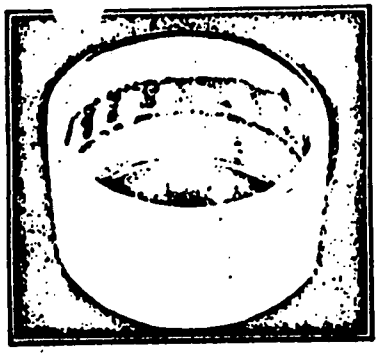


FIG. 6(a).

Arches from a Spreading Coloured Glass.

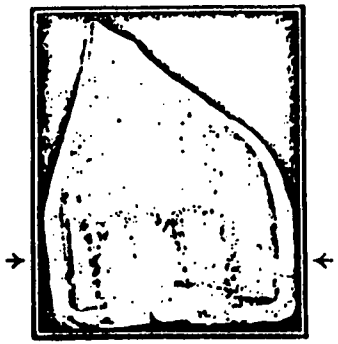


FIG. 6(b).

Arches from a Colourless Spreading Glass. The arches are just visible above the flux-line, marked by arrows.

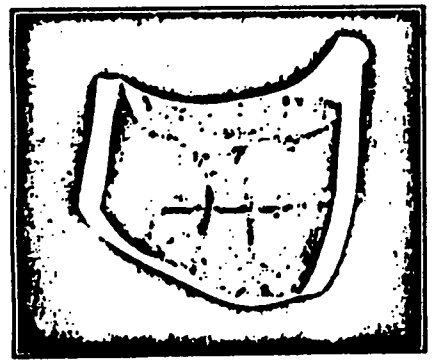


FIG. 8.

Slotted Flux-line Attack.

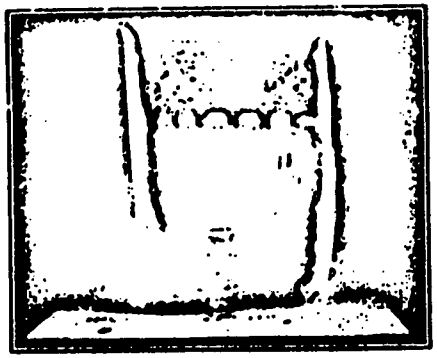
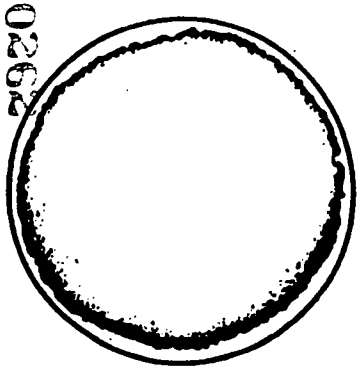
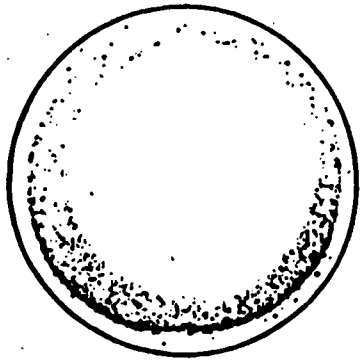


FIG. 10.

Arches from Lime Copper Ruby.



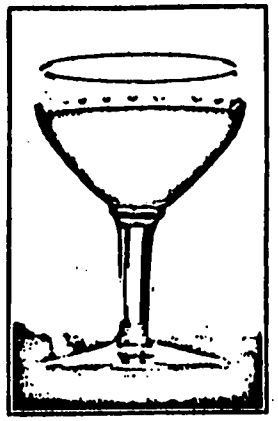
(a) *Reducing Atmosphere.*



(b) *Oxidising Atmosphere.*

FIG. 9.

Copper Ruby on Alumina-silica Refractory Base.



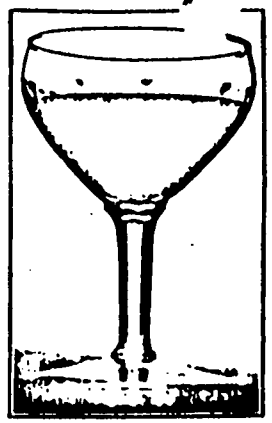
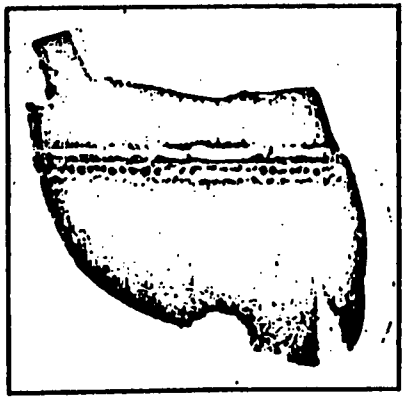
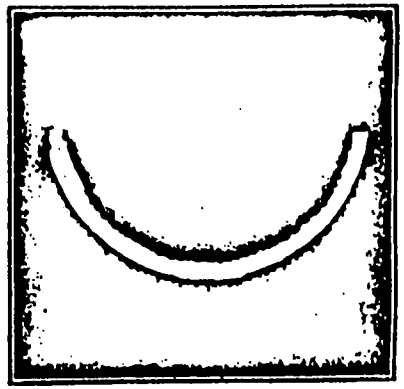


FIG. 11.

Different Forms of Tears (Arches) from Strong Wine.



(a) *Elevation.*



(b) *Cross-section.*

FIG. 12.

Typical Flux-line Pits.



FIG. 13.

Arches and Flux-line Pits.

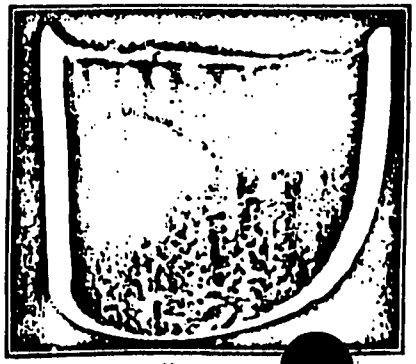


FIG. 15.

Side Pitting by Glass of Small Surface Tension.

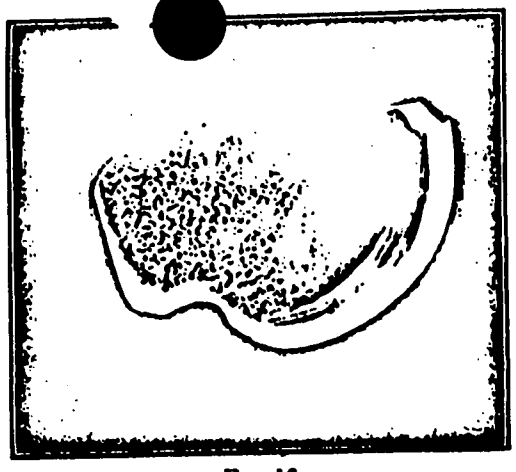


FIG. 16.
Bottom Pitting, Viewed from Above.

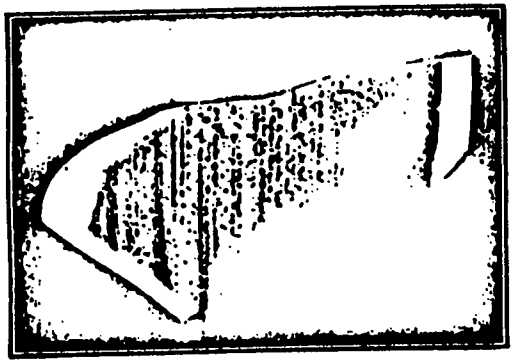


FIG. 20.
Vertical Lines on a Pot Wall.



FIG. 21(a).
Lengthwise View of Downward Scores under a Slanting Face.

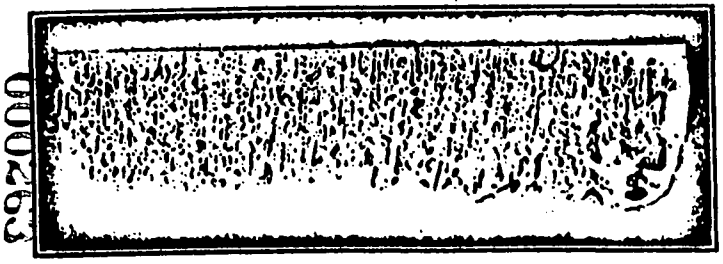


FIG. 21(b).
Cross-sectional View of Downward Scores under a Slanting Face.

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half boric oxide) glasses the holes were everywhere closely [illegible]vded (Fig. 15).

VIII. Bottom Pitting.

Close pitting of the pot bottom shown in Fig. 16 occurs with dense glasses of small surface tension. It is quite common with high-lead glass without any metallic lead separating. This type of pitting can again be explained by differences between the aggressive solvent glass and the protective aluminous solution formed at the refractory face.

On account of its greater cohesion the aluminous solution adhering to the bottom of the pot gathers into blobs. This aggregated solution

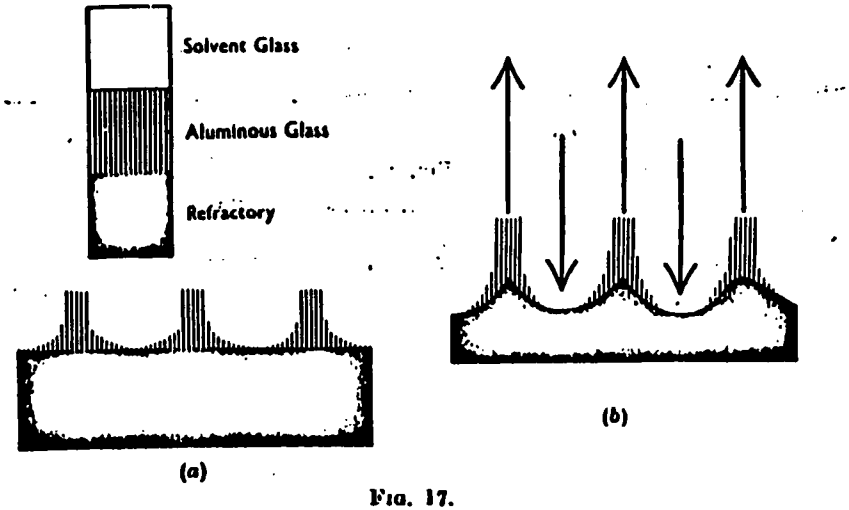


FIG. 17.
Mechanism of Bottom Pitting: (a) *aggregation of aluminous glass;* (b) *circulation.*

because of its low density rises into tongues much as a soft parison is drawn into rod. Counter-currents of denser penetrating solvent descend between the tongues and wear holes where they impinge on the bottom (Fig. 17).

The viscous film formed on the refractory surface inside the holes is continuously dragged into the main web of solution guarding the rest of the bottom. There is a circulation of glass down the middle of each hole and upwards from the ridges between. As each hole deepens the vitrostatic pressure differences between solvent and solution increase, thus reinforcing the downward jets scouring the bottom.

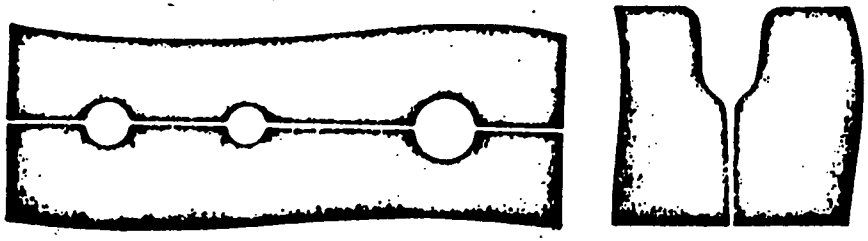
Similar bottom pitting is produced in a pot lined with paraffin wax and filled with carbon tetrachloride. The mechanism is here the same as that just described. Direct measurement showed that dissolving of the wax diminishes density and increases surface tension.

With dense glass in a refractory pot there is lighter solution against the side-wall. This results in a slow current which flows down in the interior

Reference 4-7

of the pot, then round and up against the side. The downward streams of solvent are thus intensified and diverted outwards, thereby accounting for the piercing of pot bottoms at or near the corner.

Related to bottom pitting, are pitting under a horizontal refractory face by high boric oxide glass, the aluminous solution here draining downwards, and the wearing of a row of holes along a crack through which glass is seeping (Fig. 18).



(a) (b)

FIG. 18.

Pitting Along a Crack: (a) *longitudinal section;* (b) *cross-section.*

IX. BOTTOM DRILLING BY MOLTEN METALLIC BUTTONS.

When glasses are melted under reducing conditions, heavy metals like gold, silver, copper, lead, tin, nickel, cobalt, even iron, or their alloys may separate. On sinking to the bottom of the pot, such precipitated molten metal penetrates the protective layer of aluminous glass and gathers into beads. Should ordinary bottom pitting described in the previous section precede precipitation of this molten metal, then the beads will collect in the holes already formed.

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FIG. 19.

Drilling by Metallic Beads.

If there be any appreciable quantity of oxygen dissolved in the beads, this may give corrosive metal oxide or by escaping as gas cause teetering. The spitting of silver and copper on solidifying is due to the escape of oxygen.

Downward streams of penetrating corrosive solvent envelop the mobile metallic beads as they grow, dance and turn. This solvent, together with any small pick-up of metal oxide, then attacks the refractory beneath. The aluminous solution formed is extruded by the heavy beads and frequently rises upwards, encouraging local circulation. The pot bottom becomes irregularly and often closely pitted by holes as in Fig. 19, and eventually may be drilled right through.

An analogous phenomenon is upward drilling by gas bubbles. A gas bubble, imprisoned under a refractory face, also extrudes aluminous solution: the bubble being agitated as refractory dissolves, by fluctuations of surface tension and vitrostatic pressure, much as a speck of camphor dances on water. Solvent glass of small surface tension, encouraged by

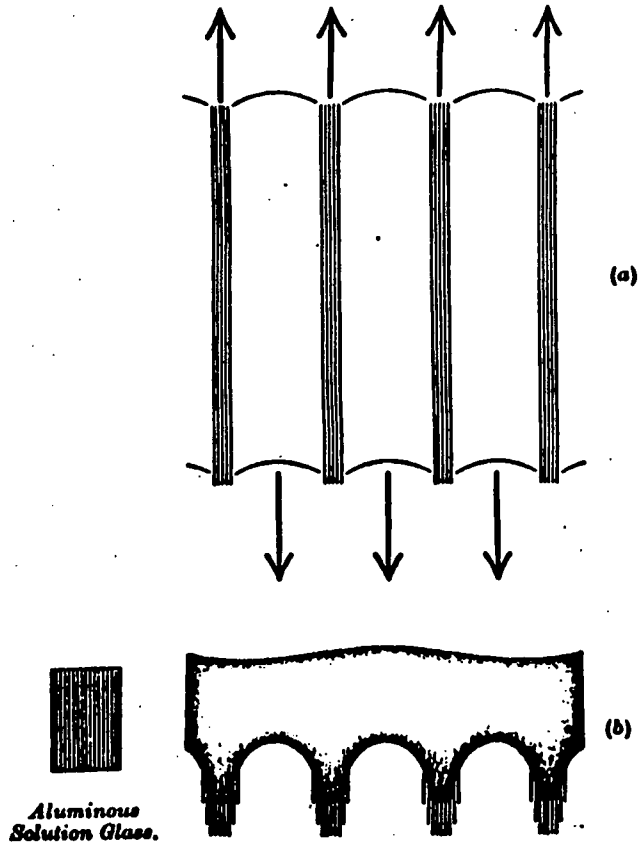


FIG. 22.

Mechanism of Downward Scoring. (a) *Lengthwise.* (b) *Cross-section.*

surface adsorption (already mentioned at the end of Section VI), envelops the bubble and drills an upward hole in the face. Such upward drilling occurs on the underside of rings, floaters and throat blocks.

X. DOWNWARD SCORES.

Another striking and distinctive type of contour again etched beneath dense glass of small surface tension is a close and regular downward scoring on an ascending refractory face. This was sometimes noticed on the side-wall of pots (Fig. 20) but was more marked on the underside of a sloping refractory block.

Reference 4-8

In a pot of dense lead glass a rectangular refractory finger was set diagonally. As expected, the top face of the block became pitted. But also, after some minutes, flutes were cut on the underface from the flux-line downwards as in Fig. 21 (*a*) and (*b*). The flutes were evenly spaced at millimetre intervals, and became fainter with depth: they were sharply etched as though engraved by a thin cutting wheel.

Again an explanation is to be found in the differences between solvent glass and the aluminous solution formed at the refractory surface. Because of its greater cohesion, the less-dense aluminous solution, slowly rising up the underface of the inclined block, gathers into filaments which move upwards side by side. A similar sort of aggregation helps to account for striae formed on mixing miscible liquids and in particular for streaks of cord in glass, aluminous cord being particularly persistent.

Dense penetrating corrosive solvent glass, including that creeping down the face from the flux-line, passes between the rising filaments and eats narrow channels in the refractory. The viscous film formed along the bed of each channel is continuously drawn by cohesion into the threads on either side.

Fig. 22 shows solvent etching the grooves while the ridges between are protected by viscous solution. Eventually the lighter aluminous threads might rise sideways into the channels. The original ridges would then become obliterated.

With a slanting block of paraffin wax in carbon tetrachloride there was again pitting on the upper face. But here the vertical sides of the block, and not the underface, were faintly marked by downward lines again at millimetre intervals.

XI. Conclusions.

The pitting and scoring described in this memoir depend on differences between solvent and solution, and would still be expected had the refractories employed been even smoother and more homogeneous. In industry such detailed patterns are often masked; * but surface forces are still persistently helping to wear away the refractories.

An important problem is to protect the refractory surface by a viscous film of solution. Incursion of solvent wears pits, scores, etc., and needs to be prevented. Where attack is greater, aluminous glass is produced more quickly; this viscous solution would be available to protect the face if it could be retained.

It would also be useful to know more about the adhesion of different glasses to refractory.

December 15th, 1952.

* There are many possible complications during glass-manufacture, including coarsely textured and unevenly fired refractory, corrosion, erosion and abrasion by batch, evolution of gas, foam and gall floating on the glass, glazing of the wall above the glass-line by dust and vapour, changes of glass level, temperature differences.

IX.—*Spectrophotometric and Magnetic Studies of Glasses Containing Iron in Relation to Their Structure. Part I. The Forms in which Iron Exists in Glass.*

By Abd-El-Moneim Abou-El-Azm, B.Sc., Ph.D., A.Inst.P.

Abstract.

The forms in which iron exists in glass were investigated by colour analysis, using Moore and Prasad's method. The results obtained from the colour analysis of a number of silicate and high-lead glasses showed that it was necessary to redetermine the specific optical densities of ferrous iron and of the brown ferric and the grey ferroso-ferric oxides given by Moore and Prasad. The specific optical densities of the ferrous iron were determined from a study of a number of silica–soda–lithia glasses, those of the brown ferric iron were calculated by studying the colours in high-lead and alkali-borate glasses and those of the ferroso-ferric oxide were determined from a number of silica–boric oxide–soda glasses. The magnetic properties of the various glasses were also determined, and the results obtained were in complete agreement with the conclusions drawn from the colour analysis, namely, that iron exists in glass in the following five forms:

(1) ferrous iron, contributing a blue colour, exists as Fe^{++} ions;

(2) ferric oxide which contributes a brown colour and is present as a colloidal dispersion;

(3) colloidal ferroso-ferric oxide which contributes a grey colour and is paramagnetic when present in small amounts (mass susceptibility = 425×10^{-6} at 20°), but shows ferromagnetic behaviour when it is present in high proportions or after the glass has been heated for 200 hours at 550° C.;

(4) colourless iron which exists as FeO_4 tetrahedra;

(5) a highly oxidised form of iron, provisionally identified as existing in the form of an alkali ferrate which produces an absorption in the region of 480—520 mμ additional to that produced by the brown colouring iron; this form is present only in glasses containing high proportions of the alkalis.

The results obtained from density measurements carried out on a number of silica–soda glasses indicated that the ferrous ions are probably enclosed in holes in the silica network.

Introduction.

The problem of "iron in glass" has been the subject of investigation by several authors. A review of the work published before 1949 is given by W. A. Weyl in the monograph *Coloured Glasses*.[1] H. Moore[2,3,4,5] and co-workers, H. Cole,[6] J. De Jong,[7] J. M. Stevels,[8] N. J. Kreidl[9] and W. A. Weyl[10] have also dealt with this subject, and the results obtained by them will be referred to at the appropriate stages in the present series of papers.

In the present paper the conclusions drawn from the colour and magnetic properties of the glasses, in so far as they give information on the state of iron in the glass, will be covered. In future papers it is proposed

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GLASS INDUSTRY

68th year

Longer Life for Electric Melters



REFRACTORIES

Reference 6-10267

Building Longer Life into Electric Melters

How advanced refractory materials have enabled designers to plan for balanced refractory life and an extended campaign

By ANTHONY J. MERCHANT
Senior Field Sales Engineer
Standard Oil Engineered Materials Co.

ELECTRIC furnaces used to produce insulating fiberglass have peculiar problems of their own, relating to both their design and the materials of construction. The primary problem is caused by the extreme turbulence created by the electric furnace's high thermal pumping action.

This pumping action constantly brings fresh glass to the refractory interface, resulting in severe corrosion for the full depth of the furnace sidewalls—unlike the condition created in a fossil-fuel-fired furnace, where corrosion is concentrated on the sidewall at a defined metal line.

Electric furnaces tend to be smaller by design and more heavily insulated than fuel-fired ones, resulting in higher interface temperatures and an increased rate of corrosion. The estimated refractory life for a typical gas-fired furnace is five years. In contrast, the estimated refractory life for a typical electric furnace is only two to three years.

The first-generation electric furnaces melting insulating fiberglass used a traditional AZS (alumina-zirconia-silica) lining for the glass contact sidewalls. The furnace's hot, turbulent conditions rapidly deteriorated the AZS lining. Operators installed air and water cooling systems in an attempt to extend the life of the AZS lining, but experienced only moderate success. Refractories with a higher level of corrosion resistance were needed.

To meet this need, researchers at Standard Oil Engineered Materials Co. developed two new types of chrome-bearing fused cast refractories: Monofrax K-3 (27% chrome-alumina) and Monofrax E (80% chrome-spinel). Both types are highly corrosion resistant, but their use is limited because they can produce coloration when they come into contact with glass in the melting process. With insulating fiberglass, however, color posed no problem. A typical chemical analysis for Monofrax K-3 and E is shown in Table 1. Fig. 1 shows a comparison of the metal line corrosion vs. temperature for soda lime glass for Monofrax S-5 (40% AZS), K-3, and E.

The superiority of the chrom products is immediately evident. Corrosion resistance is dramatically higher than that of the most resistant 40 per cent alumina-zirconia-silica product, Monofrax S-5, both at and below the melt line.

When an electric furnace's refractory lining is upgraded from an AZS to a chrome-bearing product, the

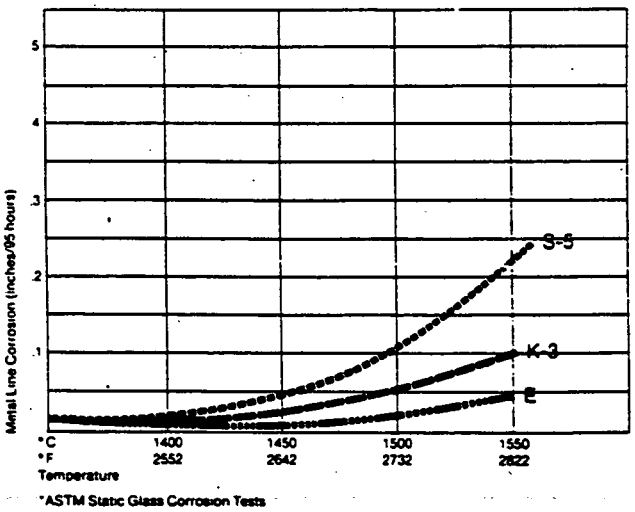


Fig. 1—Metal line corrosion vs. temperature for soda lime glass.

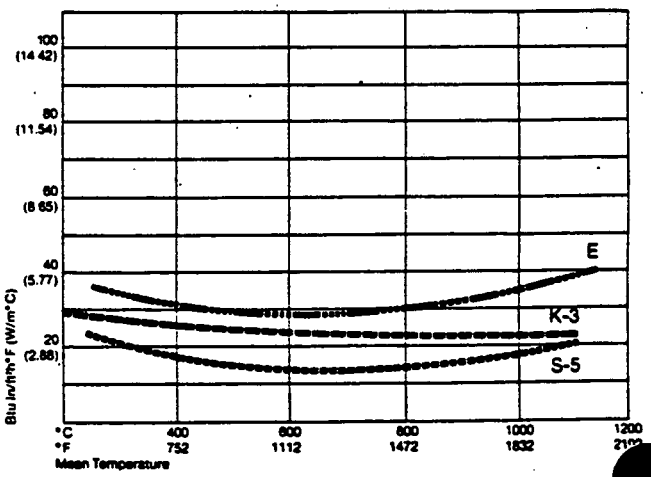


Fig. 2—Thermal conductivity.

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Reference 5-2

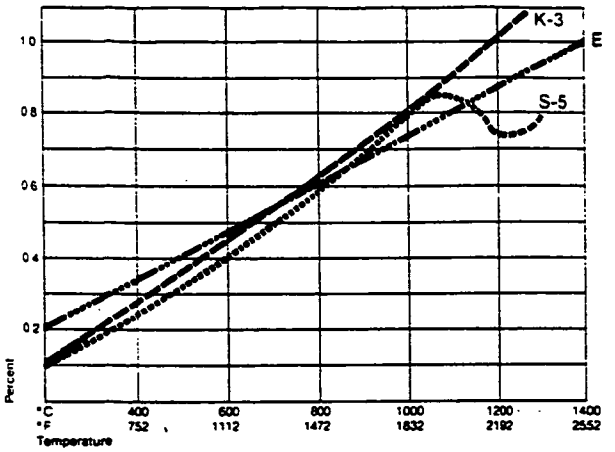


Fig. 3—Thermal expansion.

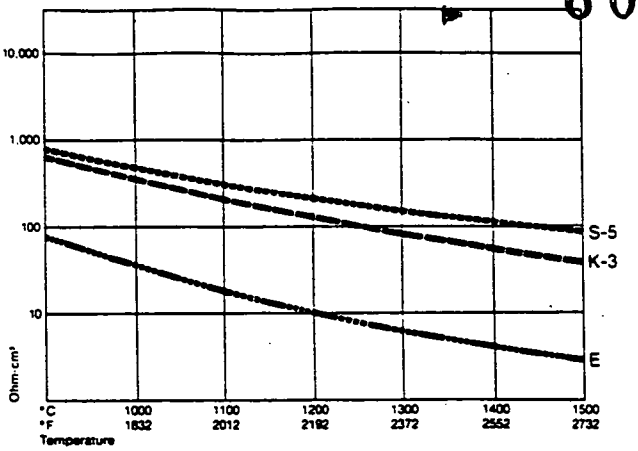


Fig. 4—Electrical resistivity vs. temperature.

physical properties are also important—in particular, thermal conductivity. As Fig. 2 indicates, both Monofrax K-3 and E refractories exhibit a slightly higher thermal conductivity rate. Added insulation may be desirable.

Another critical property is thermal expansion. As Fig. 3 indicates, Monofrax K-3 and E have a relatively uniform expansion-contraction behavior, while Monofrax S-5 has an erratic curve at high temperatures. This linear expansion allows for more rapid heat-up schedules. Shutdowns to perform intermediate repairs are also possible.

The electrical resistivity vs. temperature for Monofrax S-5, K-3, and E is shown in Fig. 4. Note that the electrical resistivity of Monofrax K-3 is similar to that of Monofrax S-5. Its high resistivity makes K-3 ideal for electric melting, where the use of electrode blocks is essential. Since the resistivity of Monofrax E is lower, it is used in electrode blocks only under extreme conditions.

The excellent corrosive properties, thermal conductivity, and thermal expansion characteristics of Monofrax K-3 and E have provided electric furnace designers with refractory materials that can withstand an electric furnace's harsh operating conditions. With K-3 sidewalls and E throats, a balanced life, with an even wear pattern, has become a reality. Although refractory performance may vary with temperature, glass composition and pull rate, fiberglass manufacturers are now able to achieve up to five-year furnace campaigns using

TABLE 1—Chemical Analysis: Monofrax K-3 and E Fused Cast Refractories

	K-3 (Chrome-Alumina)	E (Chrome-Spinel)
Cr_2O_3	27.1	77.7
Al_2O_3	58.6	6.5
Fe_2O_3	5.9	5.9
MgO	6.1	7.4
SiO_2	1.6	1.7
Other	0.3	0.2
Bulk Density ($lb./ft.^3$)	239.0	265.0
Apparent Porosity (per cent)	4.0	5.0

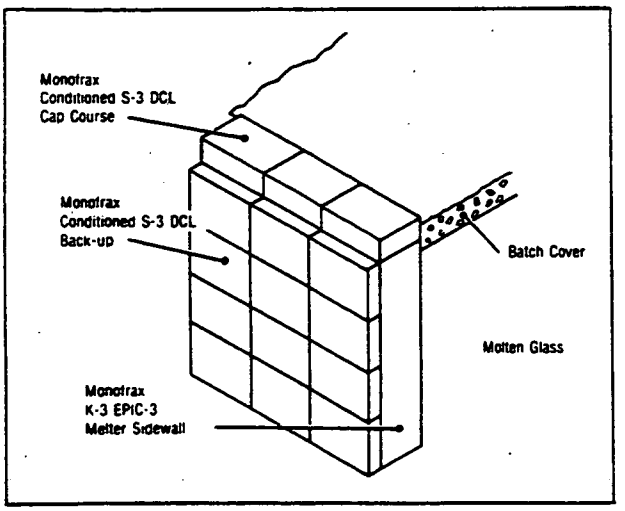


Fig. 5—Typical single-course sidewalls.

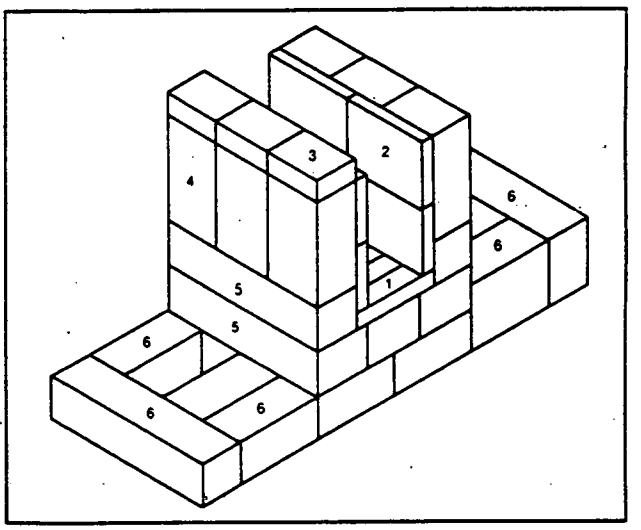


Fig. 6—Typical throat design.

Reference 5-3

Monofrax K-3 and E refractory.

In fact, in the past 12 years, more than 100 electric furnace linings using Monofrax K-3 and E have been installed in the U.S. and Canada. The following describes an example of a melter sidewall and throat design for a typical fiberglass-producing electric furnace, using Monofrax K-3 and E.

Melter sidewall design

In this design example (see Fig. 5), the glass-contact melter single-course sidewalls are constructed of voidless Monofrax K-3 EPIC-3. The EPIC-3 (end-poured, inhibited cavity) casting technique is an end-cast process using carefully timed multiple pourings into oversize headers. When the refractory is cool, the header is removed with a diamond saw. The EPIC-3 technique produces an essentially void-free casting with unsurpassed crystalline uniformity.

The blocks, which are 42 to 54 inches high, are installed with the cast surface in the down position. This ensures that the densest part of the blocks will be at the most corrosive area of the furnace.

Most electric melters are cold top in design, with a blanket of raw batch forming the upper six to eight inches. Since the materials in this area are not continually exposed to molten glass, a Monofrax S-3 (33% alumina-zirconia-silica) diamond cut lug (DCL) cap course is recommended. The properties of Monofrax S-3 DCL make it adequate for this application. However, in the case of a mixed melter with auxiliary gas firing from above, the cap course should be omitted and Monofrax K-3 continued for the full depth.

Voidless Monofrax K-3 EPIC-3 sidewalls can be used in both side and bottom electrode furnaces. In deep vertical-melt furnaces, where height may exceed 54 inches, two-course construction is used. In this case, all horizontal and vertical joints are diamond ground to ensure minimum upward drilling.

Monofrax K-3 sidewalls should be backed entirely with a three-inch overcoat of Monofrax Conditioned S-3 DCL tile. This serves as a thermal cushion for the chrome glass contact blocks and also as a barrier if glass penetration occurs later in the campaign. This envelope of DCL tile can be mortared into place with Monofrax S cement or applied dry for easy removal and reuse on subsequent rebuilds.

Throat area design

With the exception of sidewall electrode blocks, the throat area is the most vulnerable to glass attack, and the cover blocks over the opening are the most susceptible. In early designs, Monofrax K-3 was used for throat constructions. After a few campaigns, it was evident that, to balance overall furnace performance, the throat needed a refractory with corrosion resistance superior to that of the refractories used in the sidewalls.

Today, designers are using Monofrax E in the throat because of its superior corrosion resistance. Fig. 6 shows a typical throat design installed in the U.S., Canada, and Europe, using Monofrax E.

In this example, the cover blocks are Monofrax E, made by the EPIC-3 voidless-cast technique. The balance of the throat is Monofrax K-3, with the exception of the cap course. The covers are overcoated with Monofrax K-3 DCL tile and the facers, with Monofrax S-3 DCL tile. All mating surfaces are diamond ground to precise tolerances. Air or water-box cooling can be applied to further retard the corrosion rate.

Service conditions in the pavings of electric furnaces may vary, depending on the placement of electrodes, which may be located in the sidewalls or bottom of the furnace. As with all paving construction, the glass contact course should be a highly corrosive resistant material. Subsequent lower courses may have lesser resistance. For many applications, six inches of Monofrax S-5 DCL shapes should be used. Severity of operations may warrant an upgrade to Monofrax K-3 in extreme cases.

Where conditions are less demanding, Monofrax S-3 DCL may be the more economical choice. In all cases, the vertical joints should be lightly mortared and a bedding layer of Monofrax K heat-set cement should be used in order to minimize the downward penetration of molten glass.

Monofrax S-5 EPIC-3 electrode blocks are recommended for bottom electrode furnaces. If sidewall electrodes are used, Monofrax K-3 EPIC-3 is the recommended material.

The size of the riser section in electric melters can vary greatly. In risers with low-square-foot areas, the temperature of the glass in contact with the walls tends to be higher than those having larger areas, resulting in increased corrosion. Monofrax K-3 EPIC-3 is recommended for furnaces with confined designs. For melting units with larger square-foot areas, Monofrax S-5 in either the end-cast or tilt-cast method may be used.

The Monofrax line of products has given electric furnace designers added flexibility in the selection of materials to meet the wide variety of wear requirements that exist in a typical electric melter furnace. This flexibility enables designers to plan for balanced refractory life and an extended campaign. It also allows fiberglass manufacturers to better control refractory maintenance costs and reduce the amount of downtime for repairs.

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MONOFRAX® REFRACTORIES

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Refractories Division
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Monofrax® Bottom Paving System

Product Information

Background

For years the glass furnace bottom was considered an area of minimal wear with construction and design given little attention. The past two decades, however, have brought about major changes in furnace design which have drastically altered this picture and furnace bottoms have become extremely critical areas. Furnaces have evolved into larger, deeper and more highly insulated structures. Even as their output has increased, so has the furnace life, so campaigns of 6 to 10 years are not unusual and the furnace bottom must provide maintenance-free service throughout the campaign.

Multilayered Approach

Bottoms have become complex, multilayered structures consisting of a graduated series of refractory types ranging from the highly corrosion resistant fused cast glass contact paving down to the lower layers of insulation. Between are a variety of refractory types chosen to provide resistance to glass penetration between layers. Since bottoms are now insulated, temperatures in that region are higher and glass can remain molten and penetrate the joints more easily, getting between refractory layers. When this occurs, the upper glass contact refractories can be completely destroyed by upward drilling. With longer campaigns and efforts to recycle more cullet, tramp metal contamination and downward drilling also become a significant factor.

Resistance to Penetration

Attempts to control glass penetration down through vertical joints and subsequent spread horizontally beneath the paving tile have led to the development of Za-Ram plastic ramming mix as part of a complete paving system. This is a highly resistant material comprised of fused AZS grains in a high alumina matrix. It is rammed into monolithic layers as a subpaving which acts as an effective barrier both to glass and metal penetration as well as preventing the spread of glass into the horizontal joints.

Components of Monofrax System

The Monofrax paving system then consists of the glass contact layer composed of Monofrax® S DCL TILE, a thin bedding layer of S cement to seal the horizontal joint under the tile, and finally a rammed monolithic layer of Za-Ram plastic which will act as a barrier to glass and metal penetration.

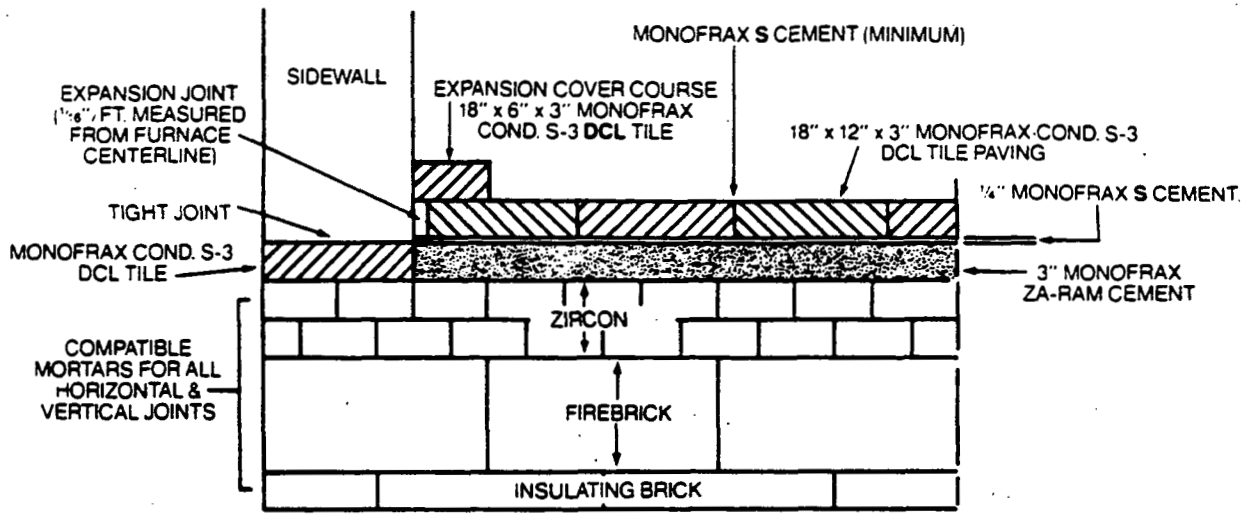
Two typical construction designs are shown on back.

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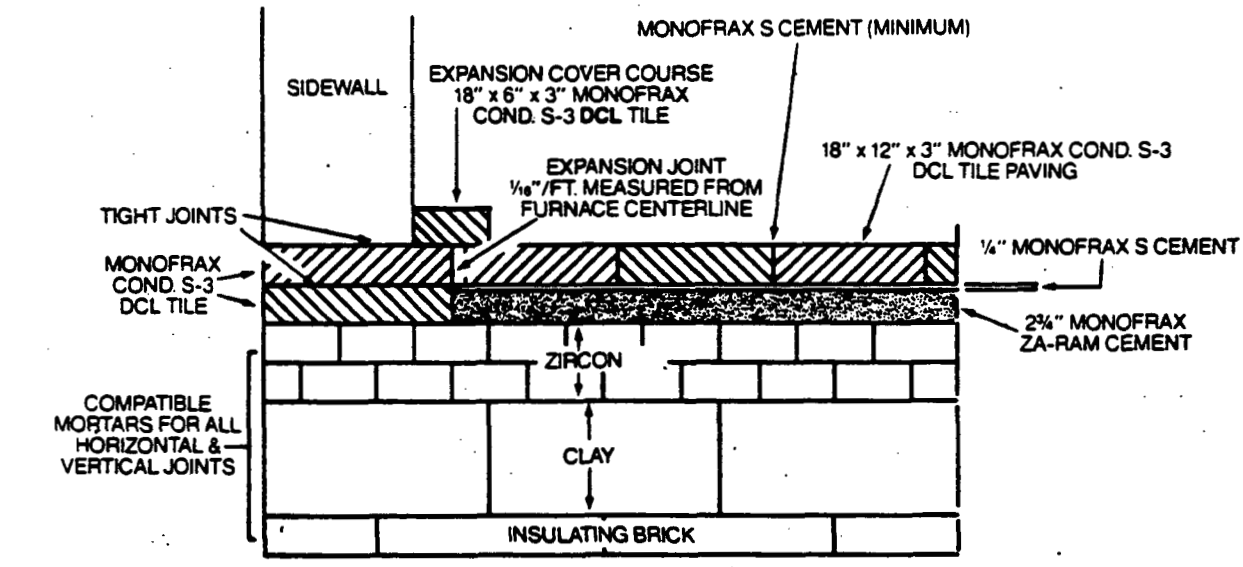
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Paving Installations



Paving installation with sidewall seated on a single Monofrax® DCL tile layer.

NOTE: Bottom arrangements shown are merely typical and not presented as a recommended construction.



Paving installation with sidewall seated on a double layer of Monofrax® DCL tile.

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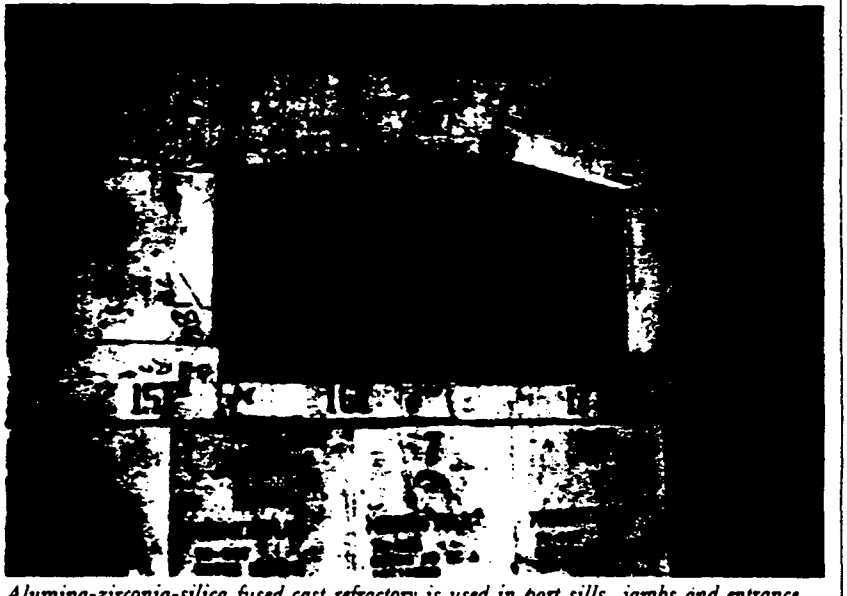
Selecting Refractories for Glass Tank Superstructures

Proper matching of refractories to various furnace environments helps cut down on glass defects and stretch out the campaign

By: Jerry Thompson,
Sales Manager, Refractories Div.,
Standard Oil Engineered Materials Co.

The glass tank has evolved into a large, highly productive and efficient melting furnace. It is capable of producing hundreds of tons of glass daily at quality levels unheard of only a few years ago. These production rates are being maintained for the life of the furnace, which can be anywhere from six to 10 years. Advances like these are due, in large part, to the quality and durability of their modern refractory linings.

Today's furnace design engineers can select from a variety of conventional bonded and fused cast refractories. To construct a balanced furnace, the engineer must be familiar with the properties of available refractories as well as the requirements of each area of the furnace. The concept of refractory balance refers to choosing materials that prevent the lining from developing weak areas that could lead to premature furnace shutdown or costly intermediate maintenance.



Alumina-zirconia-silica fused cast refractory is used in port sills, jambs and entrance arch. Beta alumina is selected for port necks.

Fused cast refractories

Because there is no all-purpose refractory, correctly matching refractories to various areas of the glass tank superstructure can be a complicated task. What follows is a brief survey of typical fused cast refractories and their applications.

Alumina-zirconia-silica (AZS) types of fused cast refractories provide the high corrosion and abrasion resistance required for service in the severe environments common to the charging end of the melter. These refractories, containing from 33 to 40% zirconia, are primarily glass contact refractories and are applied in the superstructure areas where batch dusting, slagging and rundown predominate. AZS refractories have a glassy phase (12 to 18 wt%) which is necessary to cushion the thermal expansion created by the phase transformation of zirconia, and to provide thermal shock resistance. However, this can exude from the refractory, particularly under heavily insulated conditions, and result in a rundown of zirconia-rich glass that may drip into the tank, causing such defects as stones and cords.

Aluminas also are used for superstructure applications. While not as corrosion resistant as AZS refractories, aluminas are materials of choice for downstream sections of furnace superstructures. Because they have little or no glassy phase, they can be heavily insulated without causing exudation and rundown. Aluminas also are zirconia-free, eliminating one source of cord and stone defects.

Alpha and beta aluminas

The refractory most extensively used in the flat glass industry is 100% beta alumina fused cast. Its coarse, monocrystalline structure consists of soda-saturated alumina, making it exceptionally stable in the glass tank's soda-rich atmosphere. It also provides considerable resistance to thermal shock. This refractory, however, is vulnerable to corrosive and abrasive attack, so it is primarily installed in downstream ports and breast walls where batch dusting is less severe. Its stays dry even when heavily insulated, preserving glass quality.

Mixing equal portions of alpha and beta alumina creates another high-alumina fused cast refractory. This mixed crystal phase results in a finer, denser structure having greater resistance to corrosion and abrasion. The denser crystal structure, however, detracts from thermal shock resistance. As with beta alumina, mixed alpha-

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Operating conditions affect selection

beta refractory has very little glassy phase, can be heavily insulated without causing exudation and is stable in soda-rich atmospheres. It is used in shadow walls, breast walls and end walls in some end port tanks. This refractory is more important in glass contact service in the refiners, forehearths and canal of flat glass and container furnaces.

Effects of tank life factors

Tank life factors are furnace conditions that adversely affect the service life of the refractory lining and the length of the campaign. In addition to causing severe degradation of the lining, these factors often produce refractory-associated glass defects.

• Batch carry-over. Resistance to flame-borne batch abrasion, melting and rundown requires a refractory offering maximum corrosion resistance. This explains why alumina-zirconia-silica fused cast refractories are selected for the charging end of the furnace.

• Thermal shock. The ability to withstand temperature fluctuations is critical to several areas of the furnace. Thermal shock and severe thermal gradients cause refractories to spall and fracture, producing more area for attack, as well as mechanical loss of lining material. The beta alumina fused cast refractory offers the highest resistance to thermal cycling, followed by 36% zirconia AZS.

• Alkali vapors. The alkali vapors present in the atmosphere of a soda-lime glass tank can attack and alter refractory materials. Refractories containing silica or a glassy phase are particularly vulnerable to alkali assault. The alkali vapors will react with the silica phases of the refractory, combining to form low-melting glasses that drain out of the refractory body and run down into the tank. Aluminas, particularly beta aluminas, are the most alkali resistant and will remain dry in soda-rich atmospheres.

• Glassy phase exudation. Refractories such as the AZS types, which contain an appreciable amount of glassy phase, exhibit exudation or migration of the phase to the hot face in service. This exudation is further aggravated by alkali vapors and insulation. It causes batch dust to adhere to the refractory surface, melt and increase the amount of rundown. Exudation is one of the leading causes of refractory related glass defects. Because they are free of glassy phase, alumina fused cast materials help prevent exudation-induced defects.

• Insulation. Fuel economy is critical to glass melting efficiency. To decrease heat loss, today's furnaces are heavily insulated; the superstructure areas of the tank being the most highly insulated zones. These practices reduce the thermal gradient through the refractory, raising the overall temperature through the wall. The lower melting bonding phase may then soften and allow the structure to deform. Insulation applied over existing bonded refractories already saturated with alkali vapors from a previous campaign can be particularly prone to this problem. The glass-free alumina fused cast refractories, however, work effectively when highly insulated.

Furnace zone factors

Operating conditions differ from one furnace zone to another, and are important factors in refractory selection.

• Charging end. Doghouse opening arches, sill blocks, first and second port arches, breast walls and tuckstones are subjected to the most severe furnace environments, with temperatures up to 2850°F. Cold batch being pushed into the furnace is blown about, impinging and melting on the superstructure. Refractories for this area require corrosion resistance, heat resistance, and abrasion and thermal shock resistance. Alumina-zirconia-silica fused cast refractories are generally selected, preferably 36% zirconia material. Depending upon the severity of batch dusting, the beta alumina refractory may sometimes be used in port necks.

• Third, fourth and fifth ports. Port entrance arches, port necks, jambs, breast walls and tuckstones in these areas are not directly subjected to the abrasive-corrosive action of flame-borne batch. However, these areas are still subjected to alkali vapors at high temperatures and are also heavily insulated. The beta alumina fused cast refractory is ideal for these conditions.

• Bridge wall covers, shadow walls and front end walls. These furnace areas are not subjected to extremely high temperatures or batch dusting. However, alkali-laden atmospheres can still result in chemical alteration of refractories. Any exudation or rundown will almost certainly result in glass defects, particularly if zirconia-class refractories are in place. High aluminas, beta alumina and, in the case of end fired furnaces, the alpha-beta alumina are the best choices.

• Refiner superstructure. Forehearth entrance covers and forehearth and canal covers are sometimes subjected to condensation and reflux action from alkali vapors. Again, soda-saturated beta and alpha-beta alumina fused cast materials are favored.

How Furnace Operating Conditions Affect Refractories Selection

Furnace Zone	Applicable Refractory
Charging end	AZS, beta-alumina
Third, fourth and fifth ports	Beta-alumina
Bridge wall covers, shadow walls and front walls	Aluminas
Refiner superstructure	Aluminas

How Tank Life Factors Affect Refractories Selection

Problem	Applicable Refractory
Batch carryover	AZS
Thermal shock	Beta-alumina and 36% zirconia AZS
Alkali vapors	Aluminas
Glassy phase exudation	Aluminas
Insulation	Aluminas

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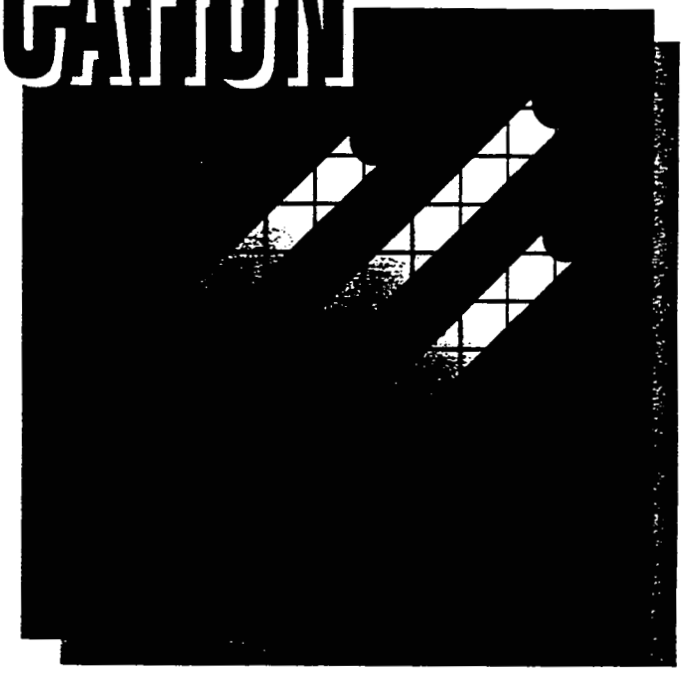
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REFERENCE 8

MONOFRAX APPLICATION BRIEF



'Exudation' And Corrosion Behavior Of Fusion Cast AZS Refractories

GRP
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Reference 8-1

'EXUDATION' AND CORROSION BEHAVIOR OF FUSION CAST AZS REFRACTORIES

SM Winder, KR Selkregg, A Gupta and D Walrod
(Monofrax Refractories)

ABSTRACT

Fused cast Alumina - Zirconia - Silica (AZS) refractories are used in the construction of glass melting furnaces. but are associated with defects in the glass due to exudation and corrosion phenomena. Exudation of a viscous liquid phase onto the refractory surface is predominantly caused by oxidation of the AZS at high temperatures. Oxidized AZS exudes less than reduced AZS, and the presence of open porosity may serve to moderate the degree of exudation experienced. Exudation volume is independent of the Na_2O content of the AZS.

First push exudation causes defect generation during first heating of AZS refractories in a newly installed glass melting furnace. However, this exudation is a short term phenomenon under isothermal conditions, and any further liquid generation on the AZS surface is due to corrosion by the glass melt or vapor. The ASTM exudation test C-1223-92 may be used to qualitatively compare first push exudation volumes from refractories subjected to the same test. It does not take into account the corrosive environment of a glass melting furnace and cannot be used to compare propensity for long term defect generation.

The current manufacturing process at Monofrax Refractories produces oxidized AZS, by introduction of molecular oxygen to the melt during fusion. This product is as close to ideal fully oxidized fully dense material as current production technology will allow, and exhibits low exudation volumes.

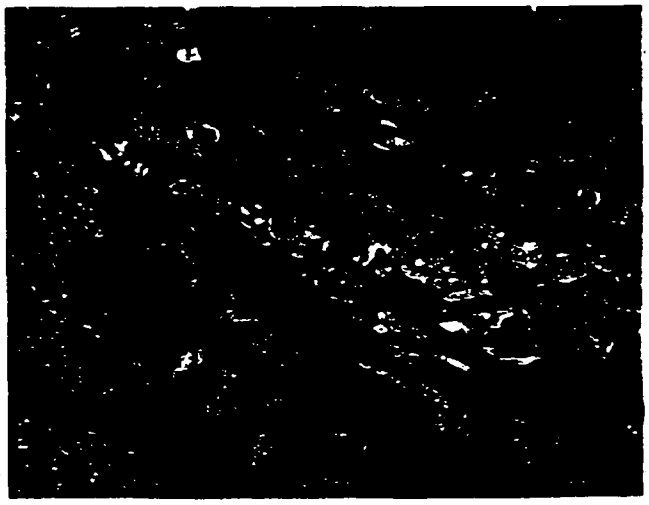
1. INTRODUCTION

Fused cast Alumina - Zirconia - Silica (AZS) refractories are used in glass contact as well as superstructure application in a wide variety of glass melting furnaces. The fusion casting process involves electric arc melting of the AZS. raw materials at ~1840°C, pouring the molten liquid into a sand mold, and then cooling the casting slowly over a period of 1-2 weeks.

During cooling, the AZS refractory develops a unique microstructure consisting primarily of three phases, as shown in Figure 1. Zirconia (the bright phase) exists as primary dendrites and alumina (the light gray phase) exists as hexagonal plates - usually containing small rods of co-precipitated zirconia. These crystals are surrounded by a sodium-aluminosilicate glassy matrix, which accounts for

Figure 1.
Typical AZS Microstructure

Zirconia dendrites (bright contrasting phase) Alumina plates (with co-precipitated Zirconia rods) Glassy matrix (~20wt%)



Monofrax CS3 (15828-15-11) *bar = 50 micrometers*

000279

~35 vol% of the refractory and provides structural integrity. The distribution and size of these different phases present in AZS varies significantly throughout the volume of a cast block, as a natural consequence of the solidification process.

When AZS blocks are placed in service in glass melting furnaces, first exposure to heat and ambient atmosphere occurs during initial heat up of the furnace. At high temperatures a phenomenon referred to as 'exudation' occurs, when a portion of the glass phase in AZS refractory exudes out onto the surface of the casting. This first push exudation phenomenon has been generally associated with various types of defects in the host glass, such as stones, cords or knots. Further exudation is then assumed to maintain defect generation - even over months or years of continuous isothermal furnace operation. This assumed link between exudation and long-term defect generation is often made on the strength of chemical analysis, which shows that both the AZS glass phase and defects in the host glass are found to contain alumina and zirconia.

To the extent that AZS exudation may be responsible for generation of defects, the glass industry prefers an AZS refractory with low exudation performance in an exudation test. High exudation volume has been correlated with partially reduced refractory material, although some glass producers have also incorrectly speculated an association between high exudation volume and higher Na_2O concentration in the AZS glass phase.

This paper will focus on the driving forces for first push exudation as experienced in isothermal application of AZS. The relationship between chemistry of AZS glassy phase and exudation will also be discussed, and a distinction between exudation and corrosion phenomena will address long-term generation of alumina and zirconia rich defects in glass. Consideration will also be given to the method of determining exudation volume.

2. THE FIRST PUSH EXUDATION PHENOMENON

AZS glassy phase exudation has been previously reviewed [1],[2], including discussion of sustained isothermal heat treatment and temperature cycling. In this section, the current ASTM test to simulate exudation will be reviewed and new experimental data will be presented to allow discussion of the driving force for first push exudation.

2.1. Driving Forces:

A range of driving forces for AZS exudation phenomena have previously been considered[1]:

Internal gas pressure derived from:
- Oxidation of reduced species
- Redox of polyvalent impurities (Fe, Ti, Cr)
- 'Reboil' of dissolved gases in the glass

'Vitrostatic pressure' due to:
- ZrO_2 phase transformation
- Other stresses in the AZS casting

Of the phases present in AZS, both the glassy matrix and the crystalline zirconia can exist in partially reduced form. In addition, any unmolten nitrate or carbonate batch entrained in the pourstream and any carbon contamination from the graphite electrodes used in arc melting, can all contribute to a list of possibly present reduced species. Reaction with the AZS melt in the mold and the presence of convection enhance dispersion of these species throughout the volume of the AZS block prior to solidification. After solidification and removal of such a partially reduced solid AZS block from its mold and insulating bed, any subsequent exposure to heat and oxygen causes generation of internal gas pressure due to oxidation reaction. *This gas pressure is potentially a major driving force for first push exudation, but must eventually be depleted when the block achieves a fully oxidized state.*

Other studies[2] have shown that temperature oscillations as low as 50°C or

100°C around the ASTM test temperature can cause an increase in exudation volume. This effect has been ascribed to redox reactions of polyvalent impurities (Fe, Ti, Cr) present in the AZS glassy phase. The polyvalent species release oxygen upon heating and accept oxygen upon cooling[10] so that each temperature cycle causes exchange of oxygen between the environment and the AZS. This cyclical pumping of oxygen then causes exudation during heating, due to generation of gas pressure within the AZS. This investigation concentrated on results from isothermal exudation testing, thus temperature cycling is not considered in this paper. *However, it is possible that redox exchange between any polyvalent glass ions and the environment could contribute a component of first push exudation during first exposure of the AZS to heat and ambient oxygen.*

'Reboil' is a term usually associated with foaming of silicate glass melts due to evolution of SO_2. This occurs upon changing redox state of the melt and SO_3 solubility in the melt is decreased[4]. *Such reboil of dissolved gases in the AZS in situ glass phase may be possible during heating, as the redox state changes upon exposure to ambient oxygen, and could contribute to exudation.* Since this effect is dependent upon oxidation reaction, it may be considered another form of 'oxidation of reduced species'.

Upon heating through the temperature range ~750°C - 900°C, zirconia undergoes a phase transformation, from monoclinic to tetragonal form, which is accompanied by a net volume contraction of ~3 - 5%. This transformation is reversible, with hysteresis, upon cooling - so that a net volume expansion occurs in the temperature range ~1175°C - 1000°C[3]. Considering Monofrax CS3 AZS, with ~33% zirconia which is all above the critical size for transformation, heating through the critical range is expected to cause ~1.3% volume shrinkage. *This effect may lead to cavitation phenomena and flow of glassy phase, but it is not expected to contribute to first push exudation during heating of AZS to isothermal service temperatures.* However, during cooling from the ASTM exudation test (see Section 2.2.), the reverse zirconia phase transformation and associated volume expansion may be considered to cause exudation that would not have been experienced in isothermal service.

Other stresses are expected to exist within any AZS block due to thermal expansion mismatch between the various phases present. However, the glassy phase accommodates considerable stress relief, by viscous flow, above its glass transition temperature (T_g ~610°C). *Any stresses developed below the T_g must be balanced within the block volume and easily relieved upon re-heating.* In contrast, any test core removed from the block may represent an unbalanced volume element under net stress. Annealing of this stress upon heating may cause some viscous flow of the glassy phase, as further discussed in Section 2.4.2.

2.2. The ASTM Exudation Test:

The ASTM test method, C-1223-92[5] is available to the glass and refractory industries to simulate exudation performance of fusion cast AZS refractories, and is generally considered useful as a basis for selection of these materials. In the test, a set of 4 inch (102mm) long 1-inch diameter AZS cores is seated in platinum foil cups and heated in 12 hours, under air in an electrically powered furnace, to a temperature of 1510°C (2750°F).

In order to simulate exudation at constant temperature, the cores are then soaked at 1510°C for 4 hours until the furnace power is cut and the specimens cool to room temperature. It is commonly accepted that over 90% of the glassy exudation volume generated during this test has occurred during heat up on the first push - before the samples reach isothermal equilibrium. This has been determined by heating cores to maximum test temperature and then immediately

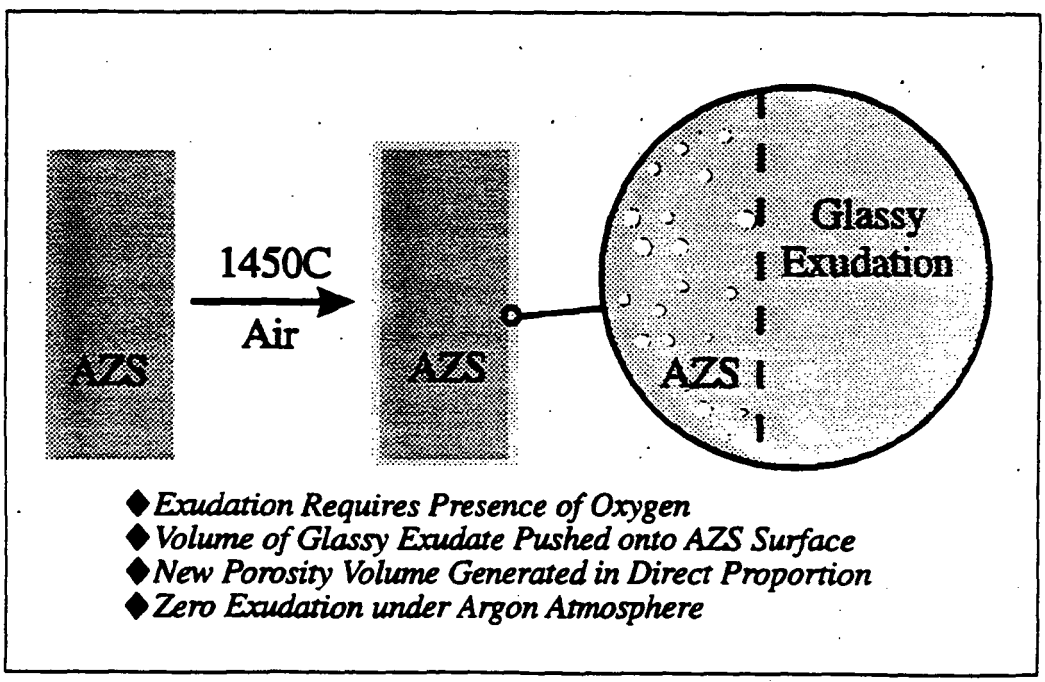
cooling them (without subjecting them to the isothermal soak), and measuring exudation volume. This test has been criticised, since it is uncertain whether the entirety of the measured exudation volume was all present on the AZS surface immediately prior to cooling. A component of that volume may have been contributed during cooling, eg. driven by volume expansion at the zirconia phase transformation. This argument is attended to in Section 2.4.1.

A variation of this test includes cooling to room temperature and re-heating the AZS samples in two more cycles. The second cycle usually generates a similar volume of exudation as the first cycle, but the third cycle causes a smaller increase in exudation. From experience, 5 or 6 cycles are usually required to yield a total exudation volume of 3 times that generated on the first cycle.

These tests may be used to compare performance of different materials tested together in the same furnace, but particular care must be taken in selection of samples, due to the gross inhomogeneity that is natural in fusion cast AZS refractories. Differences in crystal size and phase distribution occur as a function of position within any cast AZS block, as a function of depth below the cast skin. This is due to different cooling rates experienced at the solidification front, and also gravity separation of the primary zirconia dendrites - which crystallize in free liquid ahead of the co-precipitated alumina-zirconia crystalline network. Useful criteria to aid in correct sample selection are provided in the ASTM document.

Subsequent to either of the isothermal or temperature cycle tests, optical light microscopy allows observation of a significant amount of newly generated porosity within the volume of the refractory core. In fact, the amount of new porosity has been observed to be directly proportional to the exudation volume[1]. The formation of this new porosity is commonly accepted as evidence that exudation is driven by gas pressure generated inside the casting due to reaction during the test.. Figure 2 is a schematic model of the exudation phenomenon as obtained in the ASTM test.

Figure 2. Exudation Schematic



2.3. Measurement Of Exudation Volume:
The commonly followed procedure for assessing volume of glassy exudate generated during an ASTM test utilizes water immersion technique to allow comparison of total sample volume before and after testing. However, AZS in the as-produced state is highly permeable, and open porosity, including any cracks or other flaws, is easily penetrated by boiling water when following the normal practice of this method. This fact is easily demonstrated by immersion of an AZS core in ink at room temperature, the ink completely penetrates the entire core in a few hours. A very different result is obtained upon repeating the ink experiment after an exudation trial, when very little penetration of the sample is observed. The act of heat treatment apparently seals most of the open porosity, preventing infiltration.

This effect leads to an overestimation of exudation volume when using water immersion technique, since the water does not penetrate the samples' open porosity following exudation. The exudation volume is expressed as the total sample volume after the exudation test minus the total sample volume before the exudation test. Thus the error is equal to the difference in volume of open porosity penetrated during the first and second assessments, and this can be a large proportion of the totally expressed volume change including exudation!

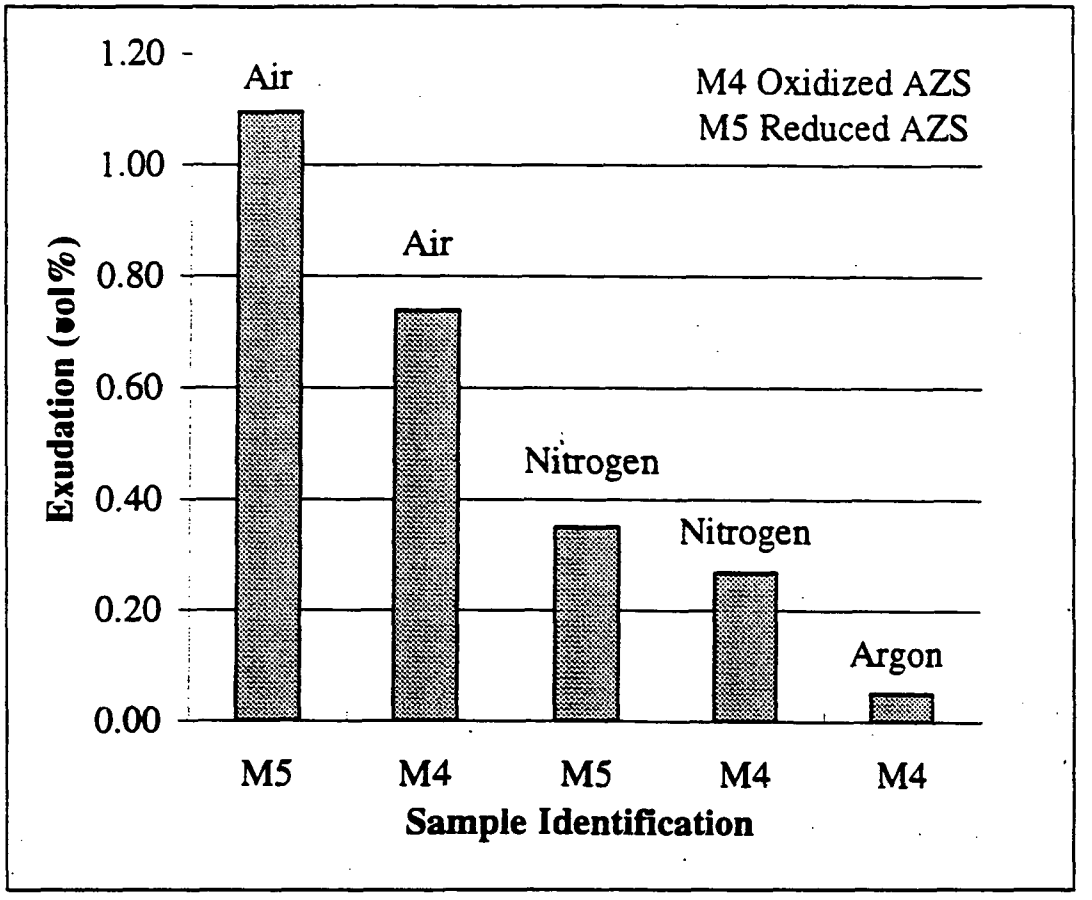
A preferred method of assessing total sample volume change is by immersion of the AZS core in a non-wetting liquid, such as mercury. When properly performed, this test allows correct determination of exudation volume - even for relatively porous samples.

2.4. Experimental Investigation Of First Push Exudation Driving Force:
Results from the Monofrax exudation test program are presented and discussed in this section.

2.4.1. Effect Of Ambient Oxygen On Exudation Volume:
In order to test the concept of *oxidation reaction* as a driving force for first push exudation, AZS samples were heat treated under air (ASTM procedure) and also under 'inert' atmosphere in a sealed tube furnace. Two experimental AZS blocks were selected for comparison, M5 was a partially reduced material of gray color, while M4 was a more oxidized material of buff color. The degree of oxidation displayed by the AZS product may be controlled during the fusion process by changing arc length from the electrodes to the melt surface, and / or by reacting molecular oxygen with the AZS melt. Glass phase composition was obtained from both AZS materials, by microanalysis, and found to be indistinguishable with respect to cation content (see Table 1).

'Inert' atmosphere tests were performed under Nitrogen resulting in about one third of the exudation measured in air, see Figure 3. A test on M4 under Argon resulted in only 0.05% exudation which corresponds to ~7% of the volume exuded under air. In addition, mercury immersion was used to assess air and nitrogen treated samples, whereas the argon treated sample was assessed using water immersion. Thus, even the small exudation volume recorded for the argon treated sample could be ascribed to an artifact of the water technique. This occurrence of very low (or zero) exudation due to testing under argon implies that ('vitrostatic') stress relaxation is not a major driving force for exudation in M4. Thus, the widely held belief (considered in Section 2.2.), that ~90% of total exudation occurs during heating to maximum temperature, is probably acceptable.

Figure 3. Exudation From Experimental CS3 AZS Under Different Atmospheres



This data demonstrates that the reaction to produce gas pressure in AZS is significantly decreased under nitrogen atmosphere (minor oxygen impurity present) and almost completely halted under argon atmosphere (very low oxygen). This is prime evidence that a major component of the exudation driving force is a consequence of *oxidation* reaction within the AZS sample, even in buff colored 'oxidized' AZS samples like M4. The M5 (gray reduced) sample gave significantly more exudation volume than M4. This is in agreement with usual observation from testing AZS refractories which exhibit a wide range of oxidation state, as further discussed in Section 2.4.4.

2.4.2. Exudation As A Function Of Thermal History And Sample Location: Fused-cast AZS is naturally inhomogeneous in structure / chemistry and density / porosity as a function of position within a given block. In order to test the effects of *pre-oxidation treatment, and sample location* on first push exudation volume, results were compared from pretreated and standard samples.

Four cores were drilled from different parts of a flat cast CS3 AZS block (M1822) that had been rejected due to the presence of porous zones. This block was intently chosen in order to obtain samples with a wide range of structure and porosity. Each cylindrical core (25mm diameter, 90mm length) was sliced longitudinally through its axis to provide two comparable halves with similar gradients in structure / chemistry and density / porosity as a function of length (depth below block surface). Each half was further diced into 3 segments representing the AZS structure at different depths (0-30mm, 30-60mm and 60-90mm) below the block surface. See Figure 4.

Segments from one half of each core were used as standard untreated benchmark samples and segments from the second half were heat treated at 1055°C/138hr. This (low temperature) pre-treatment was performed in order to allow oxidation of the core segments without causing exudation. Following the heat treatment, the core segments were inspected visually and no evidence of exudation was observed.

Pre-treated and standard samples were subjected to the ASTM time / temperature profile for assessment of first push exudation. Exudation volume was assessed using mercury immersion, and results are presented as Figure 5. Note that for each core the relationship between exudation volume and depth is different. This is not surprising, considering the macro-scale inhomogeneity within AZS blocks, as previously discussed. However, the form of this relationship is generally mirrored by standard samples and pre-treated samples from a given core, lending validity to the data.

In every case, the core segments subjected to the pre-treatment gave significantly more exudation volume than the corresponding standard sample. This is opposite to the instinctively expected trend, since pre-oxidation of the AZS during this heat treatment might have been expected to lower the driving force for exudation in a subsequently performed test.

Figure 4. Typical ZrO_2 Profiles in Flat Cast CS3 AZS.

Core sampling detail for pre-oxidation study.

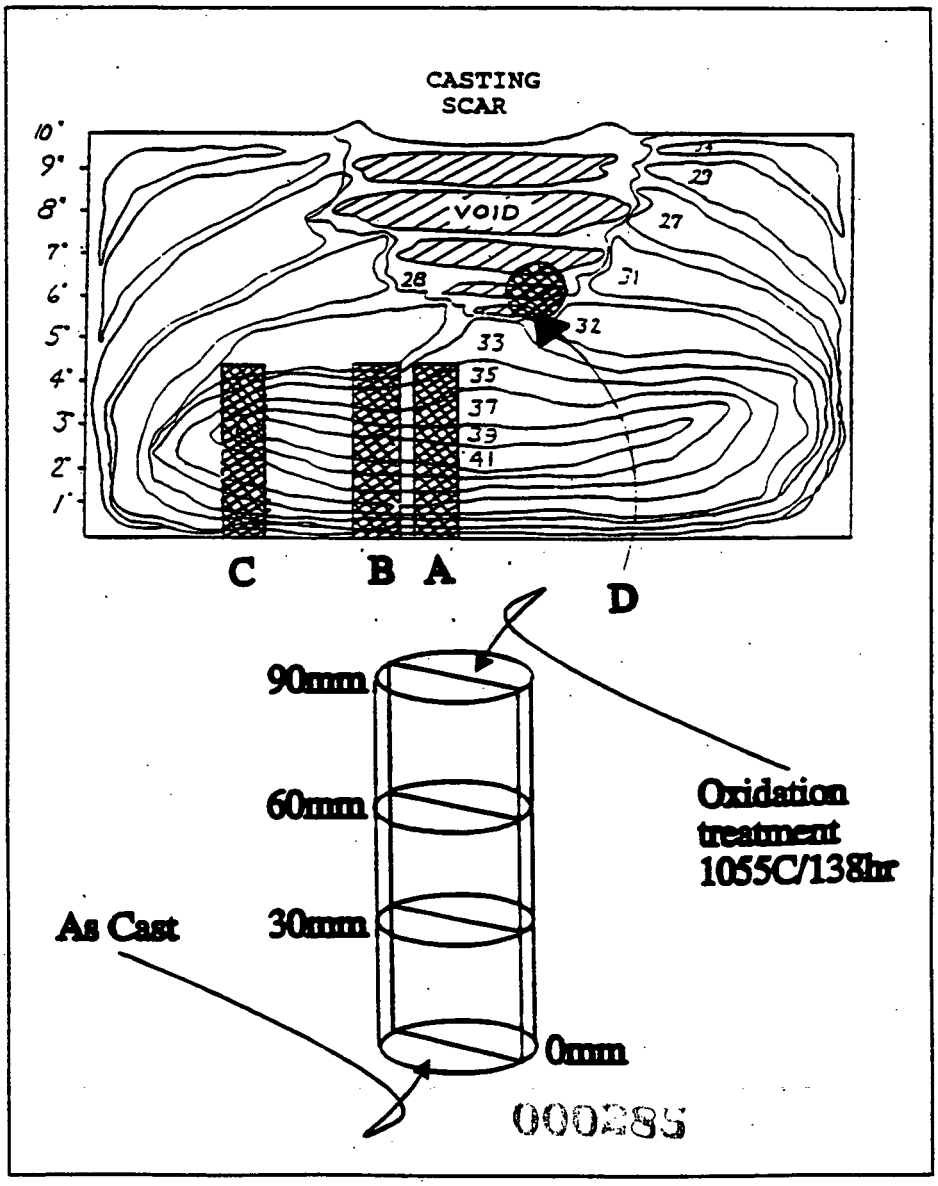
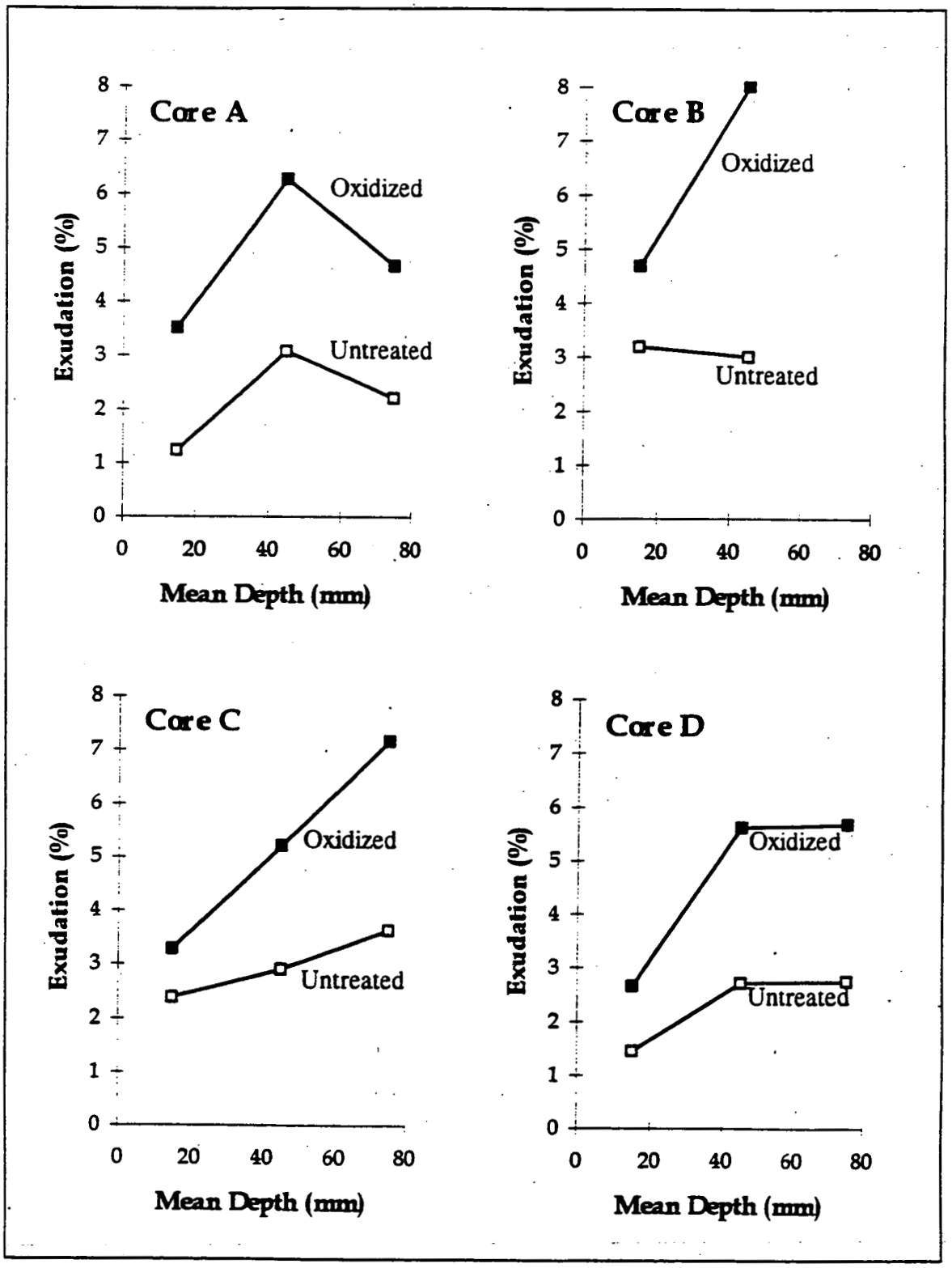


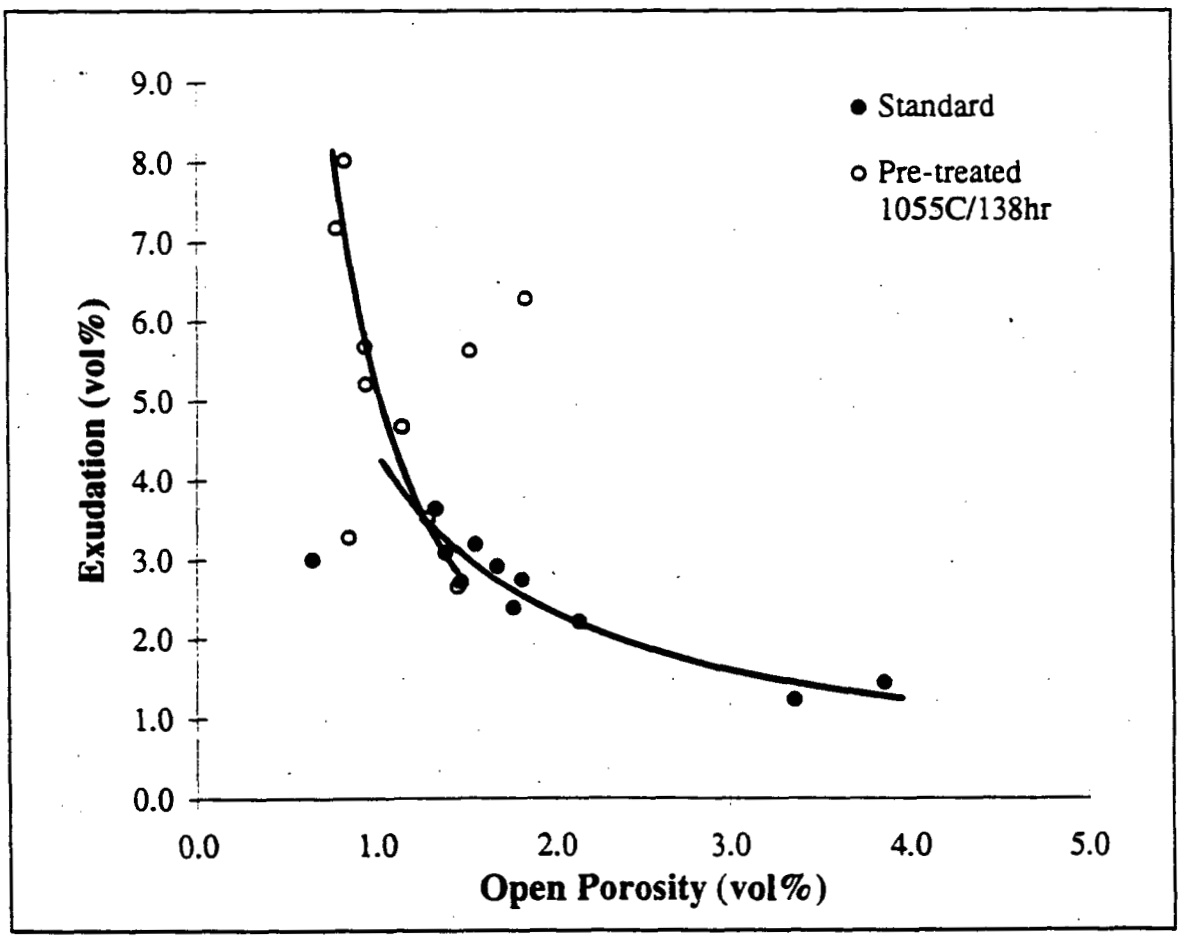
Figure 5. Exudation as a Function of Depth and Thermal History in Cores A, B, C and D



Open porosity volumes of each standard and pre-treated segment were calculated by comparing data from water immersion density measurements (water intrudes through open porosity) to data from mercury immersion density measurements (non-wetting). Figure 6 is a plot of the relationship between open porosity in the samples prior to exudation and the amount of exuded glassy phase. Shaded circles represent data for standard

This effect is easily understood by considering the AZS core as a volume element removed from the constraint of a cast block. Every AZS block includes low level long-range residual stresses which set in below the glass transition temperature during the cooling process. Generation of any high level stress at higher temperatures, eg. at the zirconia phase transformation, is defeated by viscous flow of the glassy phase in the block. When the unconstrained core

Figure 6. Exudation vs. Open Porosity



untreated core segments and open circles represent data for core segments subjected to pre-treatment. Note that all of the open circles are in the portion of the plot representing lower volumes of open porosity and higher volumes of exudation. It seems that annealing of the glass phase during the 1055°C/138hr pre-treatment caused sealing of a large portion of the previously open porosity.

is re-heated to or above the glass transition temperature for long times, relief of the now unbalanced stress field in the free core is expected to be accommodated by slow viscous flow of the glassy phase. This flow can seal off pores and flaws that were previously open to the environment.

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From Figure 6, it can be seen that decreasing the volume of open porosity lower than ~1.5% leads to increasingly unacceptable volumes of exudation from samples of the M1822 flat cast AZS block. Thus, the presence of more than ~1.5% open porosity (as determined by the described method) acts to decrease first push exudation volumes in this refractory. It is likely that this open porosity allows venting of gas pressure from the AZS during heating towards maximum temperature, and this action serves to decrease the driving force for exudation.

From the foregoing discussion, some conclusions can be drawn regarding behavior of AZS samples during the ASTM exudation test:

Firstly, if more than 90% of the first push exudation occurs in the temperature range 1055°C to 1510°C, any open porosity surviving through the short time interval spent in that temperature range must be important in allowing gas pressure relief, and thereby moderating the driving force for exudation.

Secondly, the actions of annealing and exudation eventually serve to almost completely seal the open porosity, so that a lower driving force would be necessary to exude a similar quantity of glass on subsequent heating cycles. However, the experimental observation of decreasing exudation volumes[1],[2] with increasing cycle count or time at constant temperature, imply that the driving force is being exhausted with each ensuing temperature cycle and time.

Thirdly, this action in sealing off open porosity would also be expected to decrease the in-diffusion rate of oxygen from the environment, thereby limiting the rate of reaction for continuing production of gas pressure and exudation.

Fourthly, since the ASTM test core is 4 inches (102mm) long, different amounts of first push exudation are expected to be produced from different zones along the core length. The exudation volume obtained in this test is therefore a composite average of exudation from zones between 0 and 4 inches into the block. In service, not all of these zones would necessarily be exposed to oxidation on first heat up of an AZS block, and oxygen access may initially be contained to a limited depth below the block surface.

A glass producer may therefore experience a different amount of first push exudation than expected from ASTM test results, particularly if the furnace follows a first heat schedule different to that used in the ASTM test. The test is therefore not expected to represent a quantitative prediction of first push exudation as experienced in a real furnace heat up schedule. In the absence of any other effects, the test may be expected to allow reasonable prediction of long term exudation behavior from a given block. However, absence of other effects is an unrealistic assumption - as demonstrated in Section 3 of this paper. Despite these arguments, it is still considered that the ASTM test may allow a qualitative comparison of exudation behavior exhibited by different AZS refractories in the same test - so long as extreme care is taken in sample selection, and uniformity of furnace hot zone temperature is assured during the test.

2.4.3. Effect Of AZS Oxidation State On Exudation Volume:

The data presented above confirms that a major component of first push exudation is a consequence of oxidation reaction in the AZS - there is no oxidation reaction or significant exudation under argon atmosphere. Any open porosity serves to moderate the degree of exudation caused by the product gas pressure. However, even a fully dense sample would not exude significantly in the complete absence of this driving force, so that the ideal AZS material would be fully oxidized and could be fully dense.

A reduced AZS material is expected to exude more than an oxidized AZS material, assuming other factors (porosity, sample location, etc.) to be constant. This is the usual experience observed

Figure 7(a). Open Porosity In AZS

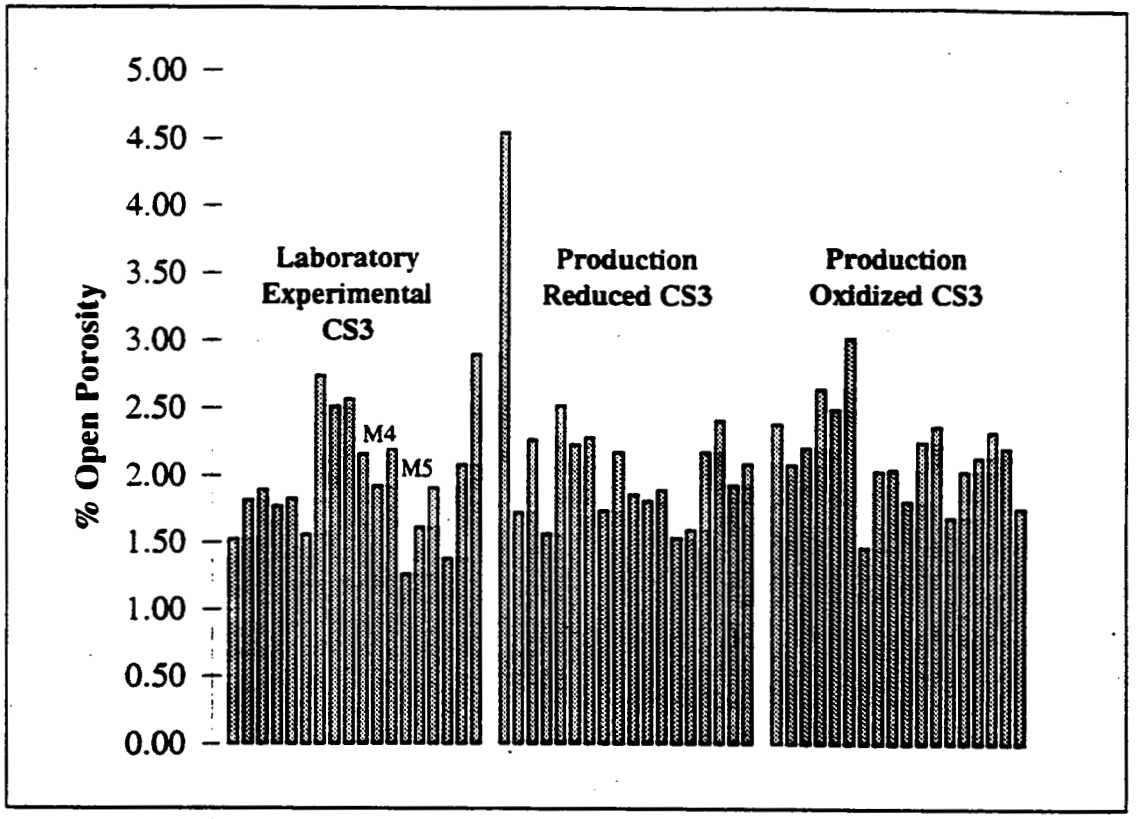
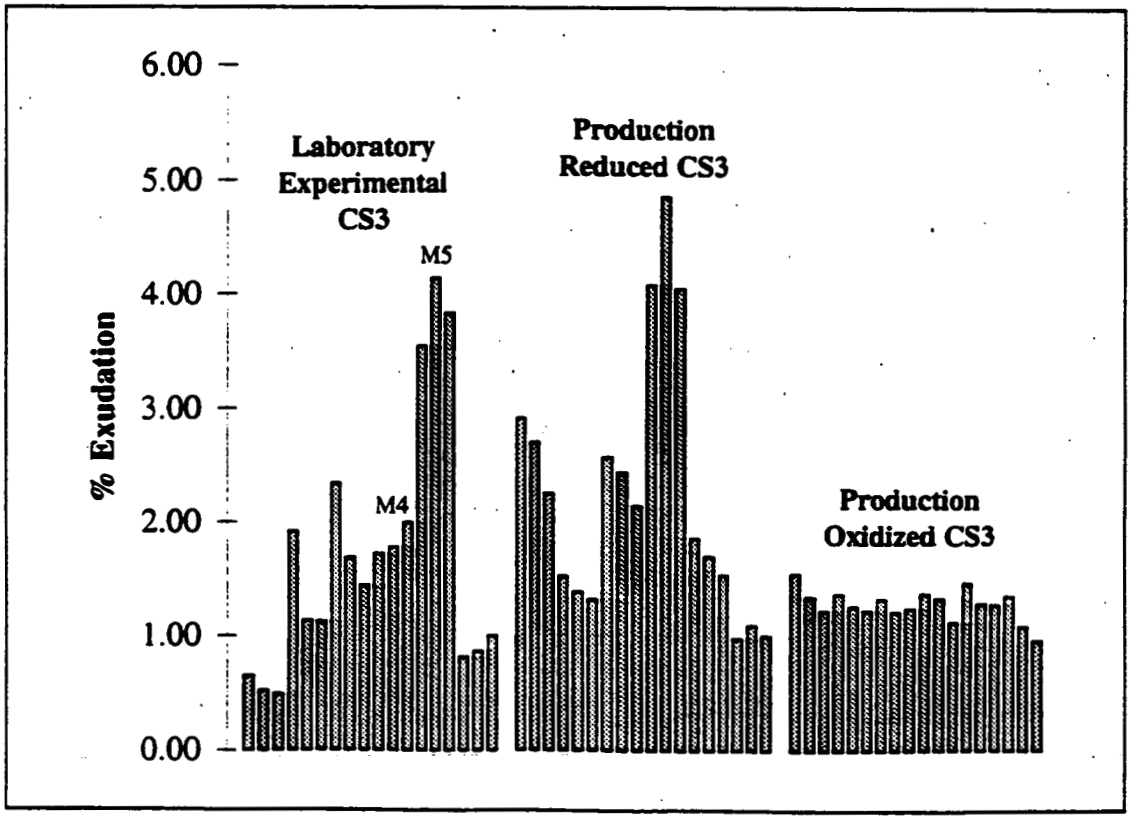


Figure 7(b). Exudation In AZS



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from the ASTM test as discussed above. In order to further test this proposition, a series of six reduced CS3 AZS blocks and six oxidized CS3 AZS blocks were prepared in the production facility at Falconer, by varying process conditions during fusion. Three core samples were carefully drilled, from equivalent locations in each block, for exudation testing using the ASTM (isothermal) technique.

Prior to exudation testing, pre-test open porosity volumes of reduced and oxidized cores were measured by difference between water and mercury immersion, as described previously. Data are presented as Figure 7(a), as the second and third set of bar charts respectively. Each consecutive bar represents a result obtained from one of the three cores taken from each of the six reduced and six oxidized cores, so that there are 18 bars in each set. Exudation data obtained from the same cores by water immersion (to allow comparison with other studies) are similarly presented as Figure 7(b). The data presented as the first set of bars in each chart are discussed in Section 2.5.

By comparing data in the second and third set of bars in Figure 7(a), it may be deduced that both reduced and oxidized cores display a similar range of open pore volumes. Total porosity was also found to be at similar levels in each series of cores. In addition, microstructures were observed to be similar in cores of each type, and microanalysis of the glass phases gave similar results to those described previously for M5 and M4 (see also Table 1). The only observable difference between the cores was color, with oxidized cores appearing uniformly buff and reduced material exhibiting a variable degree of gray tinge.

Despite the physical and chemical similarities between the reduced and oxidized cores, a striking difference can be seen in their exudation behavior - see Figure 7(b). Oxidized material yields lower exudation volume, with much less scatter around the mean, than reduced material. The current manufacturing process at Monofrax Refractories is adjusted to produce oxidized AZS, by introduction of molecular oxygen to the melt during the fusion process.

Table 1. AZS In-Situ Glass Phase Chemistry And Exudation

AZS	AZS In-Situ Glass Phase Analysis (wt%), Combined SIMS/EDS									Exudation	Porosity
	SiO2	Al2O3	Na2O	ZrO2	B2O3	TiO2	Fe2O3	CaO	Y2O3	(vol%)*	(vol%)**
M1	66.08	22.76	8.50	2.56	0.00	0.01	0.04	0.05	0.00	0.5	1.7
M2	72.10	18.45	6.26	1.85	0.01	0.24	0.33	0.45	0.29	1.4	1.7
M3	71.50	19.00	6.24	2.19	0.01	0.30	0.28	0.23	0.26	1.8	2.6
M4	68.04	20.46	6.19	3.65	0.49	0.56	0.21	0.14	0.27	1.8	2.1
M5	67.98	20.77	6.59	3.16	0.47	0.46	0.17	0.15	0.26	3.8	1.6
M6	53.81	28.50	15.14	1.59	0.50	0.17	0.03	0.13	0.12	0.9	2.1

Glass Oxidation State Undetermined

* Mean Water Immersion Exudation (ASTM) from previously untreated cores

** Mean Open Porosity measured before exudation testing

The product therefore exhibits the low exudation volumes represented in the third set of bars in Figures 7(a) and (b). This AZS refractory is as close to the ideal fully oxidized fully dense material as current production technology will allow.

2.5. AZS Glass Phase Chemistry And Exudation:

A number of AZS refractories were produced in the laboratory scale arc furnace at Monofrax. A series of six materials, M1 through M6, with varying composition and oxidation state was selected for study. All compositions were comparable to Monofrax CS3 in terms of their zirconia and alumina contents, but the composition or redox state of the glassy matrix phase in each material varied. Glass phase compositions were determined for each material by microanalysis and are presented in Table 1. The glass compositions are also plotted in projection on the Na_2O-Al_2O_3-SiO_2 phase diagram[5], presented as Figure 8, to facilitate comparison.

Three cores were taken from each experimental block, as previously described for the comparison of reduced and oxidized AZS refractories manufactured in the production facility. Open porosity and exudation volumes (water immersion) were obtained and are presented in Figures 7(a) and (b), in the first set of bars. Mean exudation data (of 3 cores) for each refractory is also presented in Table 1.

M1 is a high purity oxidized AZS composition with extremely low concentration of polyvalent ions in the glass phase. This refractory is snow white in color and exhibits the lowest exudation volume when exposed to the ASTM test. The Na_2O content of the glass phase is relatively high in comparison to most of the other compositions, and this is accompanied by an increased level of alumina.

M2 and M3 are both oxidized AZS materials made without intentionally added B_2O_3, but containing similar levels of Fe_2O_3 and TiO_2. M3 contains slightly more open porosity than M2. Exudation volumes are very similar.

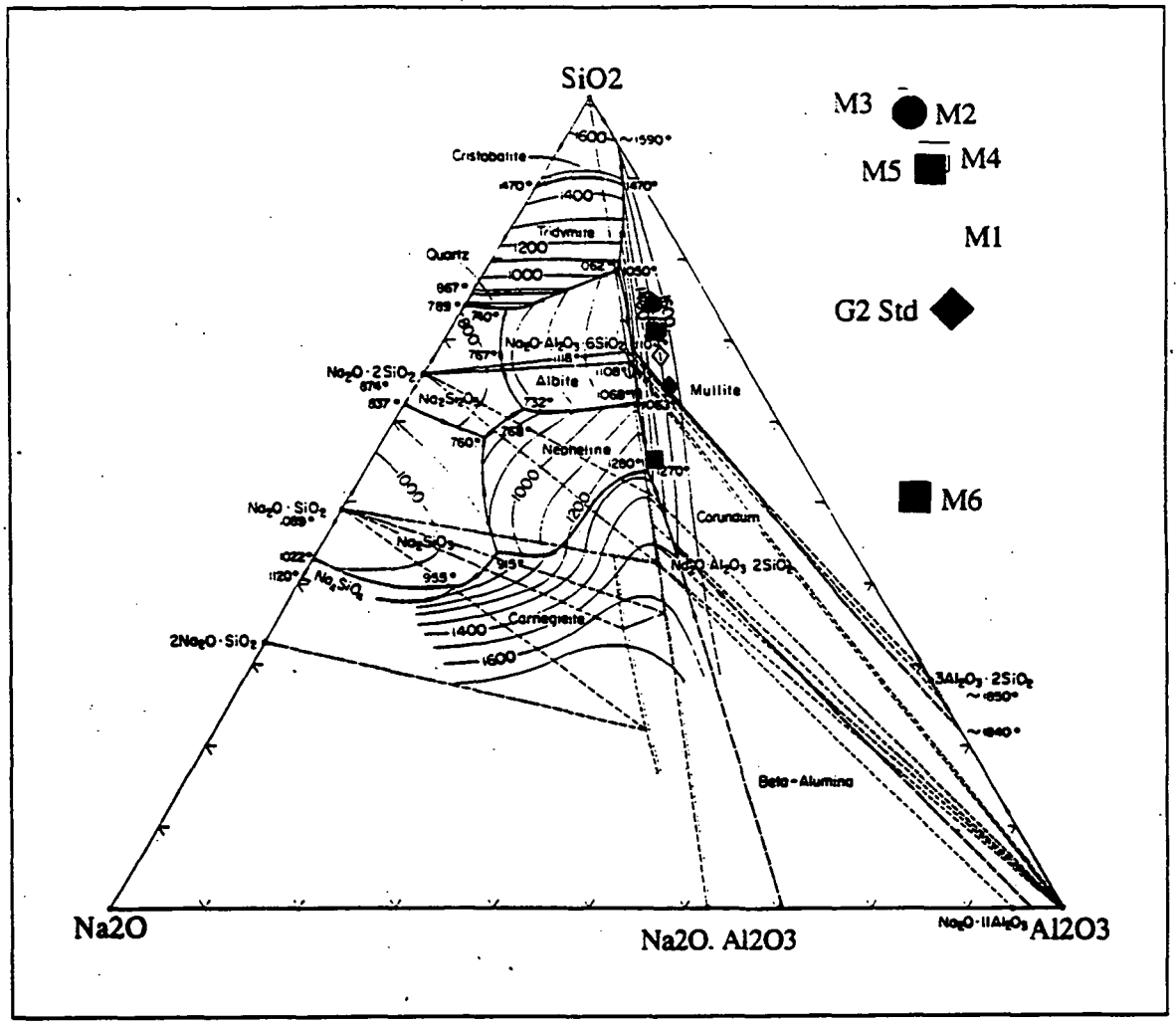
M4 and M5 are both the normal Monofrax CS3 composition, as previously discussed. M5 was intentionally produced in the reduced state in order to compare exudation behavior with that from the normal oxidized product. Note that M4 and M5 glass phase compositions are essentially identical, but can be differentiated from M2 and M3 due to their B_2O_3 content, Fe/Ti ratios and generally lower CaO content. The oxidized material (M4) yields a similar volume of exudate as M2 and M3, but the reduced material (M5) gives considerably more exudation in the same test.

M6 is a special oxidized composition, produced with ~3 times more soda in the batch than normal CS3 AZS. This material also exhibited a large range in open porosity as a function of depth. The impurity oxides have been diluted to low levels, due to the large volume of glassy phase generated in the presence of this unusually high alkali content. Despite the large Na_2O content and consequential increased volume of glass, this material produces a low exudation volume when submitted to the ASTM test. Note again that the high Na_2O concentration in this glass is accompanied by an enhanced presence of dissolved Al_2O_3.

It is currently believed by certain parts of the glass industry, and some refractory manufacturers, that alkali content of AZS materials is an important parameter in determining exudation volume. However, the data presented here demonstrate clearly that addition of extra Na_2O to the AZS batch does not lead to increased exudation in the ASTM test. This behavior is easily understood by considering both the driving force for exudation and also considering the structural network theory of glass formation[7].

Within the range of normally applied compositions, addition of extra Na_2O to the AZS batch does not change the redox state of the final product block - and therefore does not affect the driving force for exudation. In the extreme example of M6, a high volume of open

Figure 8. AZS In-Situ Glass Phase Chemistry Microanalysis Nov. 93.



Data from Table 1 plotted in projection on Figure 501 from Phase Diagrams for Ceramists[6]. Note that all glass compositions lie at a similar temperature on the liquidus and form a line parallel to SiO_2 - $Na_2O.Al_2O_3$. Any addition of Na_2O to the AZS batch must reside in the glassy matrix of the final product, increasing the solubility limit for alumina. Associated $Na_2O.Al_2O_3$ units complete a structural role similar to that of SiO_2 tetrahedra in the glass network. Thus, charge balance, together with degree of "internal coherence" of the glass network structure, is automatically compensated for by the increased presence of alumina -which always attains saturation concentration. This maintains a relatively constant set of glass phase properties, despite variation in chemistry, so that viscosity and T_g do not vary substantially from one AZS composition to another.

Reference 8-15

porosity in some segments may even serve to moderate the driving force for exudation by relieving gas pressure.

Addition of extra Na_2O to the AZS batch results in higher concentration of Na_2O in the glassy matrix phase of the product refractory. However, the extra alkali does not enter the glass structure alone as a single modifying cation, but allows an increased solubility for alumina which accompanies it into the network structure in 1:1 molar ratio. This $Na_2O.Al_2O_3$ interaction does not cause an increase in non-bridging oxygen that would occur due to addition of Na_2O in the absence of Al_2O_3. Thus, the degree of 'internal coherence' of the glassy network is maintained and properties are virtually unaffected. This means that speculation of low viscosity AZS glassy phases, rich in Na_2O, is incorrect. This can be proven by assuming Stevels model[7] to calculate the average number of bridging oxygen per tetrahedra in the glass structure, which is essentially unchanged for the range of glass compositions exhibited by M1 through M6.

Only refractory M5 was intentionally produced in the reduced state, and all other materials were oxidized within the limits of production capability. However, the high purity M1 material produced considerably less exudation than the other materials. This may be seen as evidence that polyvalent redox reactions do contribute to first push exudation, but more work is required to prove this beyond reasonable doubt.

Logic developed from the data and discussion presented in Sections 2.4. and 2.5. leads us to a governing principle employed in manufacturing Monofrax AZS to produce low first push exudation volumes:

The size of driving force due to oxidation reaction(s) is determinant and other factors (porosity, etc.) may serve to adjust the volume of exudation experienced. Na_2O content of CS3 AZS in-situ glass phase has no effect on exudation volume within reasonable limits. The objective in manufacturing optimized material should then be to remove the driving force for exudation, by production of oxidized material.

While very high purity raw materials (M1) may further optimize exudation performance, the current market for fused-cast refractories seems unwilling to pay the cost of manufacture of such premium product. Upon exposure to corrosive atmosphere in glass melting service, even high purity AZS may produce defects due to liquid drips and runoff - but this is not due to exudation, as will be discussed in the next section.

3. AZS VAPOR PHASE CORROSION IN GLASS MELTING SERVICE

During service in glass melting furnaces, AZS refractory is subjected to attack by components of the glass melt, either by direct contact with the liquid melt or through vapor phase transport of volatile species. Corrosion of AZS via the liquid phase has been extensively treated in the literature [4],[8],[9] and will be only briefly reviewed here. Corrosion of AZS and other refractories via the vapor phase has previously been given less attention, but with the introduction of oxyfuel combustion technology this topic has now become a major concern for glass producers and refractory manufacturers. Monofrax Refractories is currently pursuing an extensive study of refractory vapor phase corrosion under oxyfuel and airfuel combustion atmospheres. Regardless of the path that corrosive species take, via liquid or vapor phase, the presence of any open porosity or open flaws in the AZS will only enhance their rate of introduction.

At and below the melt line, attack via the liquid phase leads to corrosion of AZS by diffusive transport of species across the interface. Components of the glass melt, usually alkali and alkaline earth species, rapidly diffuse into the AZS surface through the glassy matrix and any flaws. The presence of these structural modifiers tends to cause dissolution of the AZS alumina phase, which then dif-

fuses out into the infinite sink of the glass melt. The AZS zirconia phase, being less soluble in the liquid at the interfacial transition zone, tends to remain undissolved and can eventually be washed into the glass melt by local currents to cause defects. Thus, the corrosive interface progresses into the AZS block, at a faster rate where currents due to convection and glass - pull are highest.

Above the melt line, alkali and other cations. can be taken from the surface of the glass melt into the vapor phase, by formation of volatile hydroxide and other species - such as NaOH, KOH, BOH, PbO, SbO, Na_2SO_4, etc. In certain parts of the furnace there is also opportunity for direct contact of gas-borne batch dust with the refractory. These corrosive species again diffuse into the AZS surface decreasing the liquidus temperature and liberating alumina into the AZS matrix. However, in this case, there is no sink in direct contact with the alumina so it remains on the AZS surface concentrated in solution in the large liquid pool containing large amounts of zirconia crystals, created due to vapor phase corrosion. Eventually, enough liquid can be created to cause drips and runoff which may become defects in the glass melt. Some glass compositions are more easily able to digest these defects than others, and longer residence times can aid in this digestion.

Given the different conditions of corrosive genesis of defects created above and below the glass melt line, it is often possible to distinguish the source of these defects by using microanalysis to compare their Al_2O_3 and ZrO_2 contents. Since corrosion is an ongoing process throughout the life of a furnace, the glass producer prefers the most corrosion resistant refractory in order to extend furnace life and decrease defect generation.

AZS subjected to service above the melt line in glass melting furnaces may therefore generate liquid phase drips and runoff due to vapor phase corrosion by interaction with volatiles from the glass melt. This long term liquid source of defects has been mistaken for exudation in some instances. However, its origin is very different and its occurrence cannot be forecast from the results of exudation trials such as the ASTM test. On cooling to room temperature, a sample of this liquid phase corrosion product will usually become opaque or white in color, due to the presence of crystalline phases throughout its volume. However, once the liquid drips into glass melt, it can lose alumina and exchange other species by diffusion, so that the defect generated in this manner may remain completely glassy upon cooling.

True exudation, as defined by ASTM type testing, occurs in the absence of corrosive species and creates a transparent glassy surface layer on the AZS when cooled to room temperature. This effect is usually experienced as a short term phenomenon, it cannot continue as a defect source over months or years in an isothermal environment, since its driving force must be exhausted with time. It has been shown that exudation due to temperature cycling of AZS containing polyvalent species may continue for longer times[2] than exudation under isothermal conditions. However, under real glass melting conditions this effect must also be combined with vapor phase corrosion as a source of defects.

4. CONCLUSIONS

First push exudation is predominantly caused by oxidation of the AZS at high temperatures. Gas pressure, generated during this oxidation, pushes a proportion of the AZS glassy matrix phase onto the refractory surface. Oxidized AZS exudes less than reduced AZS, and the presence of open porosity may serve to moderate the degree of exudation experienced. Exudation volume is independent of the Na_2O content in the AZS compositions studied.

First push exudation causes defect generation during first heating of AZS refractories placed in service in a glass melting furnace. However, this exudation

is a short term phenomenon under isothermal conditions, and any further liquid generation on the AZS surface is due to corrosive interaction with the glass melt. Above the glass melt. long term liquid generation on AZS surfaces due to vapor phase corrosion may lead to continuing production of defects. Increased presence of open porosity in the AZS is expected to exacerbate the degree of corrosion and thereby increase liquid formation.

The ASTM exudation test C-1223-92 may be used to qualitatively compare first push exudation volumes from refractories subjected to the same test. It does not take into account the corrosive environment of a glass melting furnace and cannot be used to compare propensity for long term liquid generation.

The current manufacturing process at Monofrax Refractories is adjusted to produce oxidized AZS, by introduction of molecular oxygen to the melt during the fusion process. The product therefore exhibits the low exudation volumes represented in the third set of bars in Figures 7(a) and (b). This AZS refractory is as close to the ideal fully oxidized fully dense material as current production technology will allow.

In furnaces subjected to temperature oscillations as low as 50°C[2], polyvalent ion redox exchange may extend exudation for longer time periods. However, a corrosion component must also be present in generating liquid on the AZS surface. Polyvalent ion redox exchange may contribute to first push exudation, even from oxidized AZS, but further work is necessary to prove this. High purity AZS, with low polyvalent ion content, gives the lowest exudation volumes but is not economical to produce.

ACKNOWLEDGMENT

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REFERENCES

[1]. Walrod D, 'How Furnace Operation Affects AZS Glass Phase Exudation', Glass Industry (July 1990).
[2] Dunki M, 'Studies On The Glassy And Reaction Phases Given Off By Fused Cast AZS Blocks And Their Effects On Glass Quality', Glastech. Ber., **62**, 11 (1989), 389-94
[3]. Stevens R, 'Zirconia And Zirconia Ceramics', 2nd Edition (1986), Publication 113 by Magnesium Elektron Ltd.
[4]. Trier W, 'Glass Furnaces Design Construction And Operation', English Translation by Lowenstein KL, Society Of Glass Technology, England (1987).
[5]. ASTM Standards, **15.01**, (1995).
[6]. Levin EM, Robbins CR and McMurdie HF, Figure 501, Phase Diagrams for Ceramists, **1.**, American Ceramics Society Inc. (1964).
[7]. Kingery WD, Bowen HK and Uhlmann DR, 'Introduction To Ceramics' 2nd Edition, Wiley, (1976).
[8]. Leaskey LA and McHale AE, 'Micro And Macro Aspects Of Refractory Corrosion', Ceram. Trans. 4 (Adv. Reract. Technol.), (1991), 453-63
[9]. Manfredo LJ and McNally RN, 'The Corrosion Resistance of High ZrO_2 Fusion - Cast AZS Glass Refractories In Soda Lime Glass', J. Mats. Sci., **19** (1984), 1272-76
[10]. Schreiber HD, 'Redox Processes In Glass Forming Melts' J. Non Cryst. Solids **84** (1986)129-141

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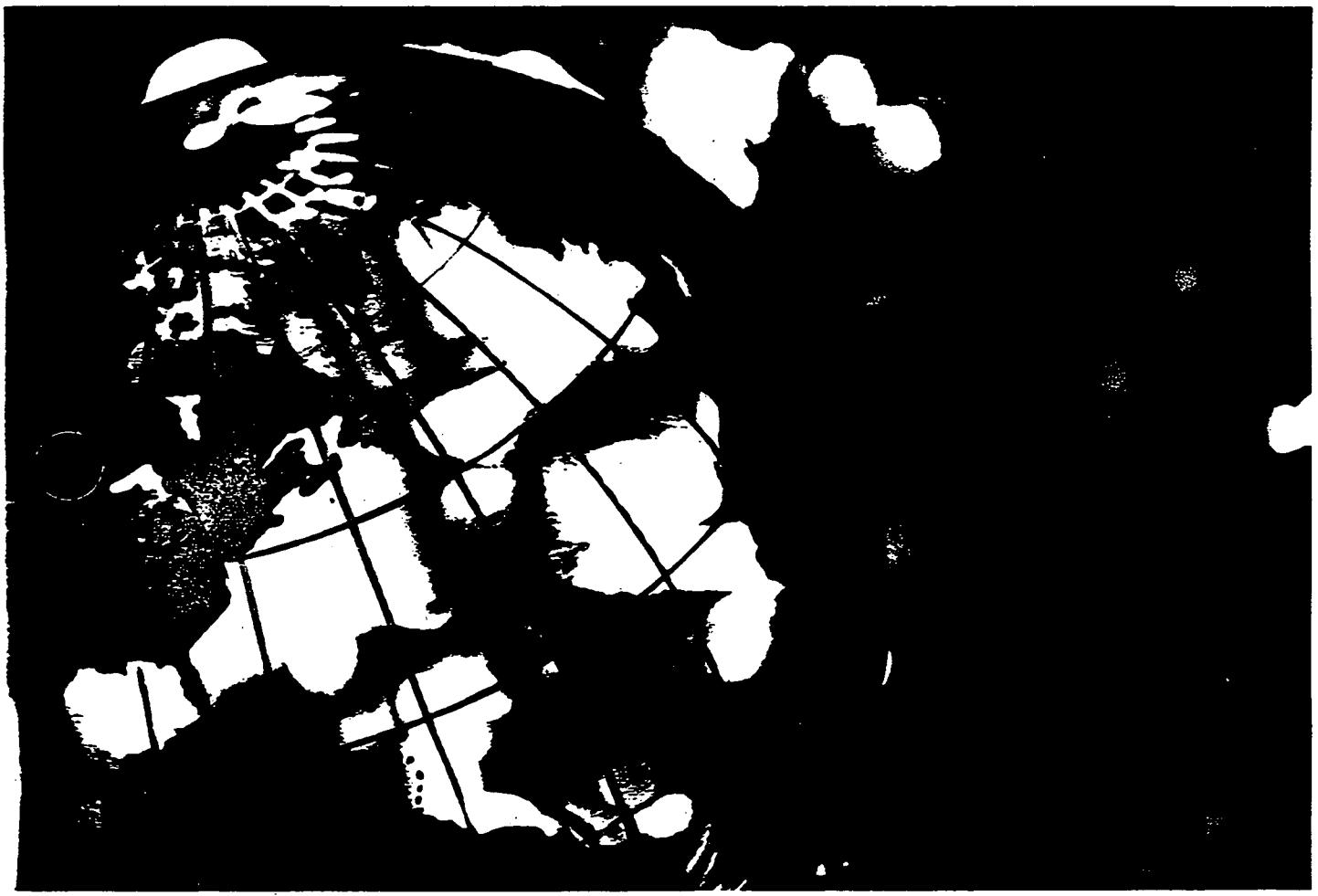
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REFERENCE 9

MONOFRAX FUSED CAST REFRACTORIES



'Exudation' and Corrosion Behavior of Fusion Cast AZS Refractories

000298

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Reference 9-1

'EXUDATION' AND CORROSION BEHAVIOR OF FUSION CAST AZS REFRACTORIES

SM Winder and KR Selkregg (Carborundum Technology Center).
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ABSTRACT

Fused cast Alumina - Zirconia - Silica (AZS) refractories are used in the construction of glass melting furnaces, but are associated with defects in the glass due to exudation and corrosion phenomena. Exudation of a viscous liquid phase onto the refractory surface is predominantly caused by oxidation of the AZS at high temperatures. Oxidized AZS exudes less than reduced AZS, and the presence of open porosity may serve to moderate the degree of exudation experienced. Exudation volume is independent of the Na_2O content of the AZS.

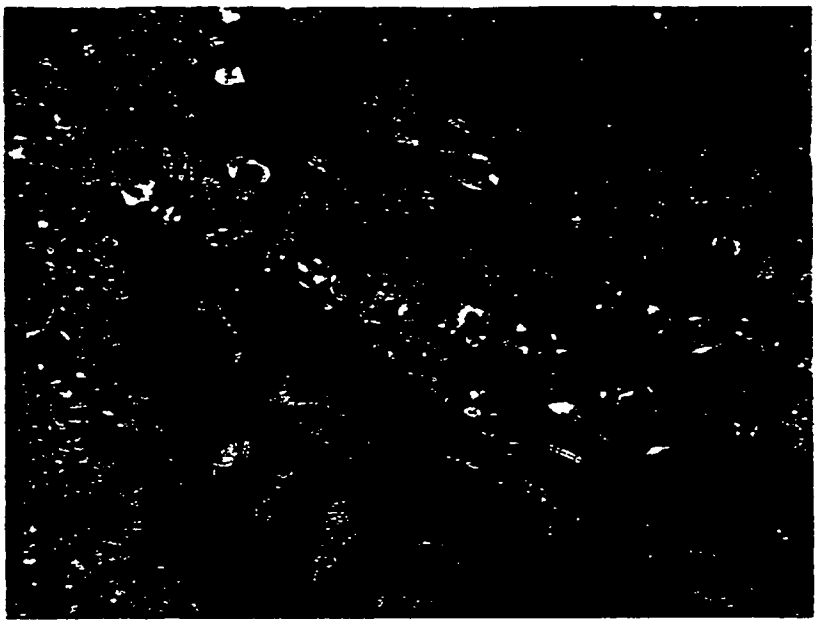
First push exudation causes defect generation during first heating of AZS refractories in a newly installed glass melting furnace. However, this exudation is a short term phenomenon under isothermal conditions, and any further liquid generation on the AZS surface is due to corrosion by the glass melt or vapor. The ASTM exudation test C-1223-92 may be used to qualitatively compare first push exudation volumes from refractories subjected to the same test. It does not take into account the corrosive environment of a glass melting furnace and cannot be used to compare propensity for long term defect generation.

The current manufacturing process at Monofrax Refractories Division produces oxidized AZS, by introduction of molecular oxygen to the melt during fusion. This product is as close to ideal fully oxidized fully dense material as current production technology will allow, and exhibits low exudation volumes.

1. INTRODUCTION

Fused cast Alumina - Zirconia - Silica (AZS) refractories are used in glass contact as well as superstructure application in a wide variety of glass melting furnaces. The fusion casting process involves electric arc melting of the AZS raw materials at ~1840°C, pouring the molten liquid into a sand mold, and then cooling the casting slowly over a period of 1-2 weeks.

Figure 1. Typical AZS Microstructure



Monofrax CS3 (15828-15-11) bar = 50 micrometers

Zirconia dendrites (bright contrasting phase)
Alumina plates (with co-precipitated Zirconia rods)
Glassy matrix (~20wt%)

During cooling, the AZS refractory develops a unique microstructure consisting primarily of three phases, as shown in Figure 1. **Zirconia** (the bright phase) exists as primary dendrites and **alumina** (the light gray phase) exists as hexagonal plates - usually containing small rods of co-precipitated zirconia. These crystals are surrounded by a **sodium-aluminosilicate glassy matrix,** which accounts for ~35 vol% of the refractory and provides structural integrity. The distribution and size of these different phases present in AZS varies significantly throughout the volume of a cast block, as a natural consequence of the solidification process.

When AZS blocks are placed in service in glass melting furnaces, first exposure to heat and ambient atmosphere occurs during initial heat up of the furnace. At high temperatures a phenomenon referred to as '**exudation**' occurs, when a portion of the glass phase in AZS refractory exudes out onto the surface of the casting. This first push exudation phenomenon has been generally associated with various types of defects in the host glass, such as stones, cords or knots.

Reference 9-3

Further exudation is then assumed to maintain defect generation - even over months or years of continuous isothermal furnace operation. This assumed link between exudation and long-term defect generation is often made on the strength of chemical analysis, which shows that both the AZS glass phase and defects in the host glass are found to contain alumina and zirconia.

To the extent that AZS exudation may be responsible for generation of defects, the glass industry prefers an AZS refractory with low exudation performance in an exudation test. High exudation volume has been correlated with partially reduced refractory material, although some glass producers have also incorrectly speculated an association between high exudation volume and higher Na_2O concentration in the AZS glass phase.

This paper will focus on the driving forces for first push exudation as experienced in isothermal application of AZS. The relationship between chemistry of AZS glassy phase and exudation will also be discussed, and a distinction between exudation and corrosion phenomena will address long-term generation of alumina and zirconia rich defects in glass. Consideration will also be given to the method of determining exudation volume.

2. THE FIRST PUSH EXUDATION PHENOMENON

AZS glassy phase exudation has been previously reviewed [1],[2], including discussion of sustained isothermal heat treatment and temperature cycling. In this section, the current ASTM test to simulate exudation will be reviewed and new experimental data will be presented to allow discussion of the driving force for first push exudation.

2.1. Driving Forces:- A range of driving forces for AZS exudation phenomena have previously been considered[1]:-

Internal gas pressure derived from:-	Oxidation of reduced species
	Redox of polyvalent impurities (Fe, Ti, Cr)
	'Reboil' of dissolved gases in the glass
'Vitrostatic pressure' due to:-	ZrO_2 phase transformation
	Other stresses in the AZS casting

Of the phases present in AZS, both the glassy matrix and the crystalline zirconia can exist in partially reduced form. In addition, any unmolten nitrate or carbonate batch entrained in the pourstream and any carbon contamination from the graphite electrodes used in arc melting, can all contribute to a list of possibly

present reduced species. Reaction with the AZS melt in the mold and the presence of convection enhance dispersion of these species throughout the volume of the AZS block prior to solidification. After solidification and removal of such a partially reduced solid AZS block from its mold and insulating bed, any subsequent exposure to heat and oxygen causes generation of internal gas pressure due to oxidation reaction. *This gas pressure is potentially a major driving force for first push exudation, but must eventually be depleted when the block achieves a fully oxidized state.*

Other studies[2] have shown that temperature oscillations as low as 50°C or 100°C around the ASTM test temperature can cause an increase in exudation volume. This effect has been ascribed to redox reactions of polyvalent impurities (Fe, Ti, Cr) present in the AZS glassy phase. The polyvalent species release oxygen upon heating and accept oxygen upon cooling[10] so that each temperature cycle causes exchange of oxygen between the environment and the AZS. This cyclical pumping of oxygen then causes exudation during heating, due to generation of gas pressure within the AZS. This investigation concentrated on results from 'isothermal' exudation testing, thus temperature cycling is not considered in this paper. However, *it is possible that redox exchange between any polyvalent glass ions and the environment could contribute a component of first push exudation during first exposure of the AZS to heat and ambient oxygen.*

'Reboil' is a term usually associated with foaming of silicate glass melts due to evolution of SO_2. This occurs upon changing redox state of the melt and SO_3 solubility in the melt is decreased[4]. *Such reboil of dissolved gases in the AZS in situ glass phase may be possible during heating, as the redox state changes upon exposure to ambient oxygen, and could contribute to exudation.* Since this effect is dependent upon oxidation reaction, it may be considered another form of 'oxidation of reduced species'.

Upon heating through the temperature range ~750°C - 900°C, zirconia undergoes a phase transformation, from monoclinic to tetragonal form, which is accompanied by a net volume contraction of ~3 - 5%. This transformation is reversible, with hysteresis, upon cooling - so that a net volume expansion occurs in the temperature range ~1175°C - 1000°C[3]. Considering Monofrax CS3 AZS, with ~33% zirconia which is all above the critical size for transformation, heating through the critical range is expected to cause ~1.3% volume shrinkage. *This effect may lead to cavitation phenomena and flow of glassy phase, but it is not expected to contribute to first push exudation during heating of AZS to isothermal service temperatures.* However, during cooling from the ASTM exudation test (see Section 2.2.), the reverse zirconia phase transformation and associated

volume expansion may be considered to cause exudation that would not have been experienced in isothermal service.

Other stresses are expected to exist within any AZS block due to thermal expansion mismatch between the various phases present. However, the glassy phase accommodates considerable stress relief, by viscous flow, above its glass transition temperature (T_g ~610°C). *Any stresses developed below the T_g must be balanced within the block volume and easily relieved upon re-heating.* In contrast, any test core removed from the block may represent an unbalanced volume element under net stress. Annealing of this stress upon heating may cause some viscous flow of the glassy phase, as further discussed in Section 2.4.2.

2.2. The ASTM Exudation Test:- The ASTM test method, C-1223-92[5] is available to the glass and refractory industries to simulate exudation performance of fusion cast AZS refractories, and is generally considered useful as a basis for selection of these materials. In the test, a set of 4 inch (102mm) long 1 inch diameter AZS cores is seated in platinum foil cups and heated in 12 hours, under air in an electrically powered furnace, to a temperature of 1510°C (2750°F).

In order to simulate exudation at constant temperature, the cores are then soaked at 1510°C for 4 hours until the furnace power is cut and the specimens cool to room temperature. It is commonly accepted that over 90% of the glassy exudation volume generated during this test has occurred during heat up on the first push - before the samples reach isothermal equilibrium. This has been determined by heating cores to maximum test temperature and then immediately cooling them (without subjecting them to the isothermal soak), and measuring exudation volume. This test has been criticised, since it is uncertain whether the entirety of the measured exudation volume was all present on the AZS surface immediately prior to cooling. A component of that volume may have been contributed during cooling, eg. driven by volume expansion at the zirconia phase transformation. This argument is attended to in Section 2.4.1.

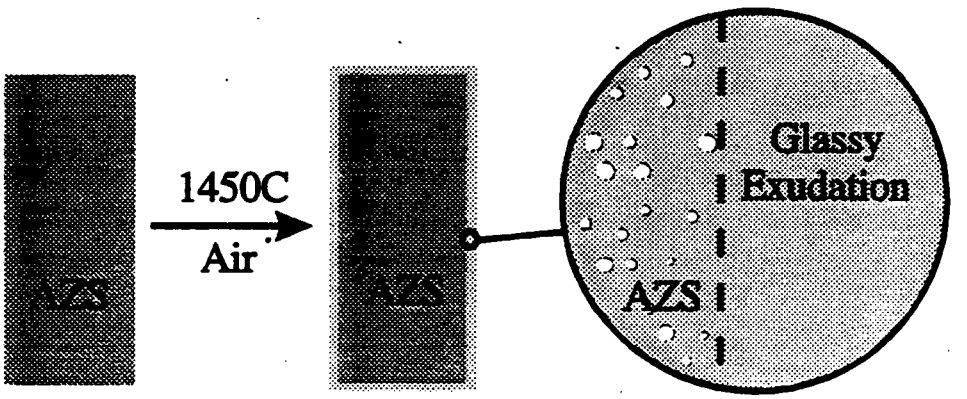
A variation of this test includes cooling to room temperature and re-heating the AZS samples in two more cycles. The second cycle usually generates a similar volume of exudation as the first cycle, but the third cycle causes a smaller increase in exudation. From experience, 5 or 6 cycles are usually required to yield a total exudation volume of 3 times that generated on the first cycle.

These tests may be used to compare performance of different materials tested together in the same furnace, but particular care must be taken in selection of samples, due to the gross inhomogeneity that is natural in fusion cast AZS

refractories. Differences in crystal size and phase distribution occur as a function of position within any cast AZS block, as a function of depth below the cast skin. This is due to different cooling rates experienced at the solidification front. and also gravity separation of the primary zirconia dendrites - which crystallize in free liquid ahead of the co-precipitated alumina-zirconia crystalline network. Useful criteria to aid in correct sample selection are provided in the ASTM document.

Subsequent to either of the isothermal or temperature cycle tests, optical light microscopy allows observation of a significant amount of newly generated porosity within the volume of the refractory core. In fact, the amount of new porosity has been observed to be directly proportional to the exudation volume[1]. The formation of this new porosity is commonly accepted as evidence that exudation is driven by gas pressure generated inside the casting due to reaction during the test.. Figure 2 is a schematic model of the exudation phenomenon as obtained in the ASTM test.

Figure 2. Exudation Schematic



- *Exudation Requires Presence of Oxygen*
- *Volume of Glassy Exudate Pushed onto AZS Surface*
- *New Porosity Volume Generated in Direct Proportion*
- *Zero Exudation under Argon Atmosphere*

2.3. Measurement Of Exudation Volume:- The commonly followed procedure for assessing volume of glassy exudate generated during an ASTM test utilizes water immersion technique to allow comparison of total sample volume before and after testing. However, AZS in the as-produced state is highly permeable,

Reference 9-7

and 'open porosity', including any cracks or other flaws, is easily penetrated by boiling water when following the normal practice of this method. This fact is easily demonstrated by immersion of an AZS core in ink at room temperature, the ink completely penetrates the entire core in a few hours. A very different result is obtained upon repeating the ink experiment after an exudation trial, when very little penetration of the sample is observed. The act of heat treatment apparently seals most of the open porosity, preventing infiltration.

This effect leads to an overestimation of exudation volume when using water immersion technique, since the water does not penetrate the samples' open porosity following exudation. The exudation volume is expressed as the total sample volume after the exudation test minus the total sample volume before the exudation test. Thus the error is equal to the difference in volume of open porosity penetrated during the first and second assessments, and this can be a large proportion of the totally expressed volume change including exudation!

A preferred method of assessing total sample volume change is by immersion of the AZS core in a non-wetting liquid, such as mercury. When properly performed, this test allows correct determination of exudation volume - even for relatively **porous samples.**

2.4. Experimental Investigation Of First Push Exudation Driving Force:-

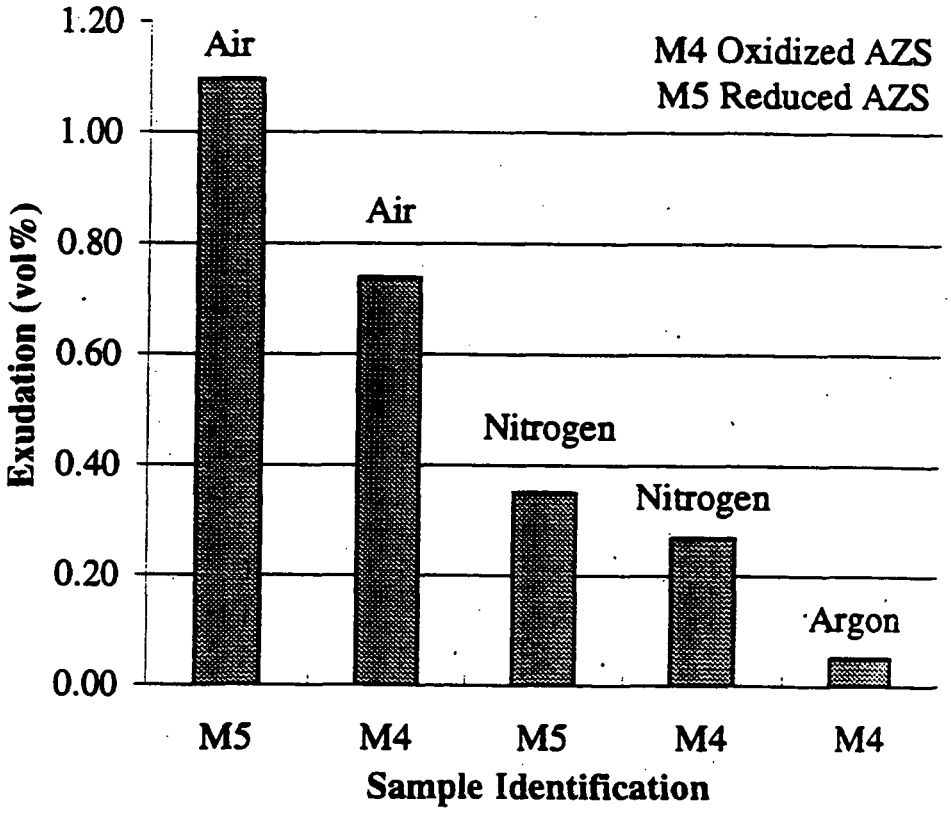
Results from the Monofrax exudation test program are presented and discussed in this section.

2.4.1. Effect Of Ambient Oxygen On Exudation Volume:- In order to test the concept of **oxidation reaction** as a driving force for first push exudation, AZS samples were heat treated under air (ASTM procedure) and also under 'inert' atmosphere in a sealed tube furnace. Two experimental AZS blocks were selected for comparison, M5 was a partially reduced material of gray color, while M4 was a more oxidized material of buff color. The degree of oxidation displayed by the AZS product may be controlled during the fusion process by changing arc length from the electrodes to the melt surface, and / or by reacting molecular oxygen with the AZS melt. Glass phase composition was obtained from both AZS materials, by microanalysis, and found to be indistinguishable with respect to cation content (see Table 1.).

'Inert' atmosphere tests were performed under Nitrogen resulting in about one third of the exudation measured in air, see Figure 3. A test on M4 under Argon resulted in only 0.05% exudation which corresponds to ~7% of the volume

exuded under air. In addition, mercury immersion was used to assess air and nitrogen treated samples, whereas the argon treated sample was assessed using water immersion. Thus, even the small exudation volume recorded for the argon treated sample could be ascribed to an artifact of the water technique. This occurrence of very low (or zero) exudation due to testing under argon implies that ('vitrostatic') stress relaxation is not a major driving force for exudation in M4. Thus, the widely held belief (considered in Section 2.2.), that ~90% of total exudation occurs during heating to maximum temperature, is probably acceptable.

Figure 3. Exudation From Experimental CS3 AZS Under Different Atmospheres



This data demonstrates that the reaction to produce gas pressure in AZS is significantly decreased under nitrogen atmosphere (minor oxygen impurity present) and almost completely halted under argon atmosphere (very low oxygen). This is prime evidence that a major component of the exudation driving force is a consequence of **oxidation** reaction within the AZS sample, even in buff colored 'oxidized' AZS samples like M4. The M5 (gray reduced) sample gave

significantly more exudation volume than M4. This is in agreement with usual observation from testing AZS refractories which exhibit a wide range of oxidation state, as further discussed in Section 2.4.4.

2.4.2. Exudation As A Function Of Thermal History And Sample Location:- Fused-cast AZS is naturally inhomogeneous in structure / chemistry and density / porosity as a function of position within a given block. In order to test the effects of **pre-oxidation treatment, and sample location** on first push exudation volume, results were compared from pre-treated and standard samples.

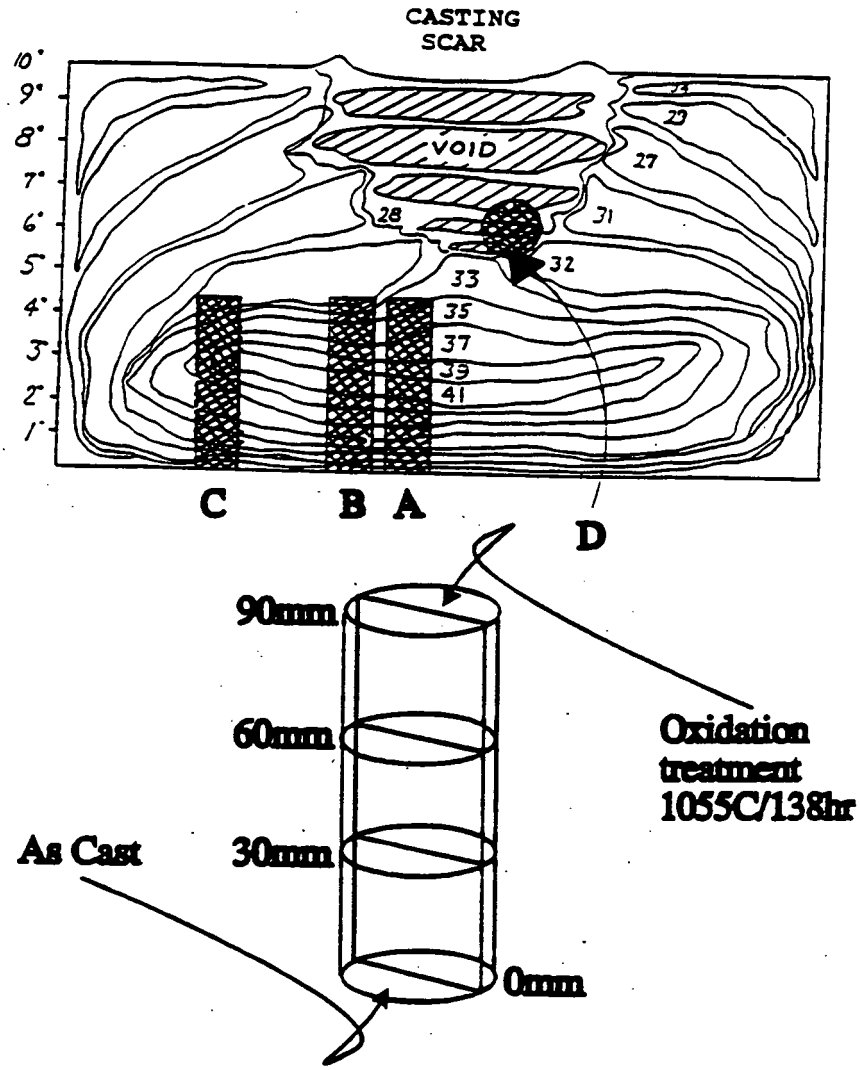
Four cores were drilled from different parts of a flat cast CS3 AZS block (M1822) that had been rejected due to the presence of porous zones. This block was intently chosen in order to obtain samples with a wide range of structure and porosity. Each cylindrical core (25mm diameter, 90mm length) was sliced longitudinally through its axis to provide two comparable halves with similar gradients in structure / chemistry and density / porosity as a function of length (depth below block surface). Each half was further diced into 3 segments representing the AZS structure at different depths (0-30mm, 30-60mm and 60-90mm) below the block surface. See Figure 4.

Segments from one half of each core were used as 'standard' untreated benchmark samples and segments from the second half were heat treated at 1055°C/138hr. This (low temperature) pre-treatment was performed in order to allow oxidation of the core segments without causing exudation. Following the heat treatment, the core segments were inspected visually and no evidence of exudation was observed.

Pre-treated and standard samples were subjected to the ASTM time / temperature profile for assessment of first push exudation. Exudation volume was assessed using mercury immersion, and results are presented as Figure 5. Note that for each core the relationship between exudation volume and depth is different. This is not surprising, considering the macro-scale inhomogeneity within AZS blocks, as previously discussed. However, the form of this relationship is generally mirrored by standard samples and pre-treated samples from a given core, lending validity to the data.

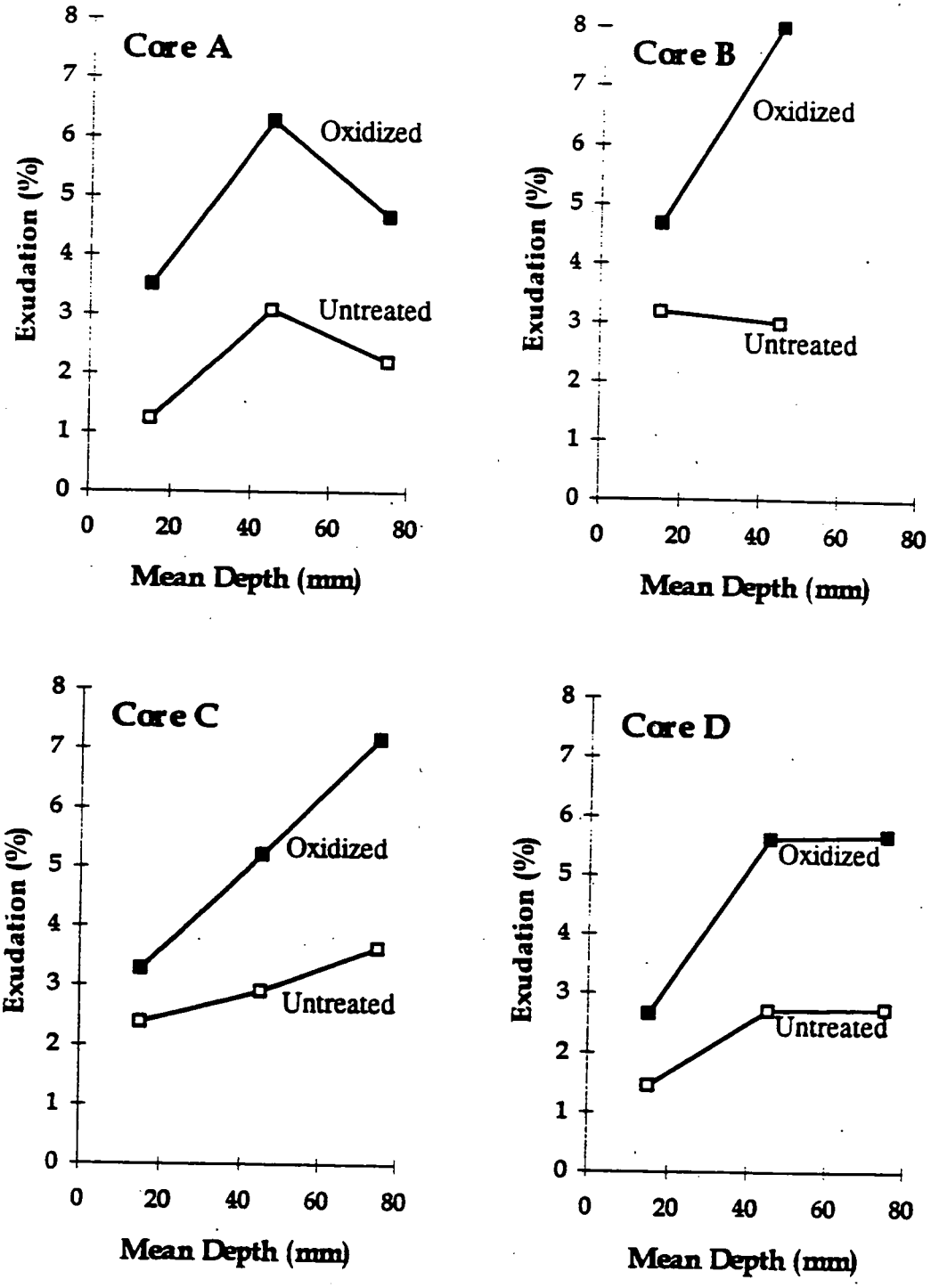
In every case, the core segments subjected to the pre-treatment gave significantly more exudation volume than the corresponding standard sample. This is opposite to the instinctively expected trend, since pre-oxidation of the AZS during this heat treatment might have been expected to lower the driving force for exudation in a subsequently performed test.

Figure 4. Typical ZrO_2 Profiles in Flat Cast CS3 AZS
Core sampling detail for pre oxidation study



Reference 9-11

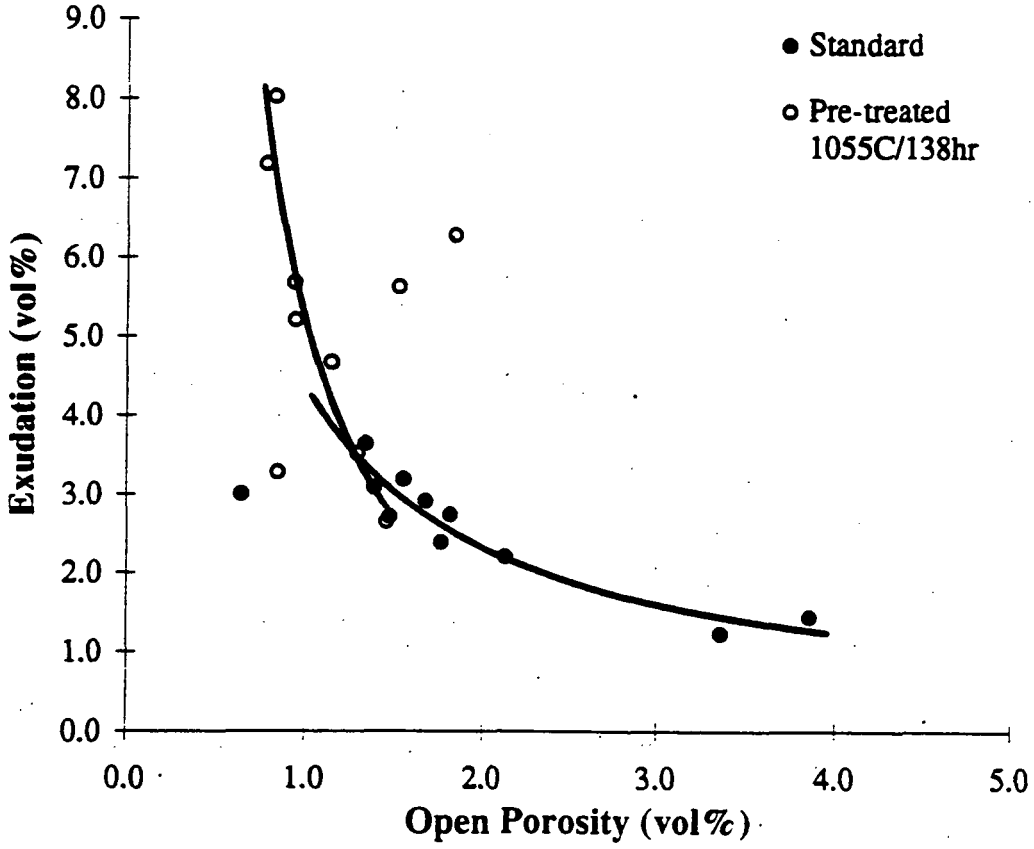
Figure 5. Exudation as a Function of Depth and Thermal History in Cores A, B, C and D



Reference 9-12

Open porosity volumes of each standard and pre-treated segment were calculated by comparing data from water immersion density measurements (water intrudes through open porosity) to data from mercury immersion density measurements (non-wetting). Figure 6. is a plot of the relationship between open porosity in the samples prior to exudation and the amount of exuded glassy phase. Shaded circles represent data for standard untreated core segments and open circles represent data for core segments subjected to pre-treatment. Note that all of the open circles are in the portion of the plot representing lower volumes of open porosity and higher volumes of exudation. It seems that annealing of the glass phase during the long-term pre-treatment caused sealing of a large portion of the usually open porosity.

Figure 6. Exudation vs Open Porosity



This effect is easily understood by considering the AZS core as a volume element removed from the constraint of a cast block. Every AZS block includes low level long-range residual stresses which set in below the glass transition temperature during the cooling process. Generation of any high level stress at higher

Reference 9-13

temperatures, eg. at the zirconia phase transformation, is defeated by viscous flow of the glassy phase in the block. When the unconstrained core is re-heated to or above the glass transition temperature for long times, relief of the now unbalanced stress field in the free core is expected to be accommodated by slow viscous flow of the glassy phase. This flow can seal off pores and flaws that were previously open to the environment.

From Figure 6, it can be seen that decreasing the volume of open porosity lower than ~1.5% leads to increasingly unacceptable volumes of exudation from samples of the M1822 flat cast AZS block. Thus, the presence of more than ~1.5% open porosity (as determined by the described method) acts to decrease first push exudation volumes in this refractory. It is likely that this open porosity allows venting of gas pressure from the AZS during heating towards maximum temperature, and this action serves to decrease the driving force for exudation.

From the foregoing discussion, some conclusions can be drawn regarding behavior of AZS samples during the ASTM exudation test:-

Firstly, if more than 90% of the first push exudation occurs in the temperature range 1055°C to 1510°C, any open porosity surviving through the short time interval spent in that temperature range must be important in allowing gas pressure relief, and thereby moderating the driving force for exudation.

Secondly, the actions of annealing and exudation eventually serve to almost completely seal the open porosity, so that a lower driving force would be necessary to exude a similar quantity of glass on subsequent heating cycles. However, the experimental observation of decreasing exudation volumes[1],[2] with increasing cycle count or time at constant temperature, imply that the driving force is being exhausted with each ensuing temperature cycle and time.

Thirdly, this action in sealing off open porosity would also be expected to decrease the in-diffusion rate of oxygen from the environment, thereby limiting the rate of reaction for continuing production of gas pressure and exudation.

Fourthly, since the ASTM test core is 4 inches (102mm) long, different amounts of first push exudation are expected to be produced from different zones along the core length. The exudation volume obtained in this test is therefore a composite average of exudation from zones between 0 and 4 inches into the block. In service, not all of these zones would necessarily be exposed to oxidation on first heat up of an AZS block, and oxygen access may initially be contained to a limited depth below the block surface.

A glass producer may therefore experience a different amount of first push exudation than expected from ASTM test results, particularly if the furnace follows a first heat schedule different to that used in the ASTM test. The test is therefore not expected to represent a quantitative prediction of first push exudation as experienced in a real furnace heat up schedule. In the absence of any other effects, the test may be expected to allow reasonable prediction of long term exudation behavior from a given block. However, absence of other effects is an unrealistic assumption - as demonstrated in Section 3 of this paper. Despite these arguments, it is still considered that the ASTM test may allow a qualitative comparison of exudation behavior exhibited by different AZS refractories in the same test - so long as extreme care is taken in sample selection, and uniformity of furnace hot zone temperature is assured during the test.

2.4.3. Effect Of AZS Oxidation State On Exudation Volume:- The data presented above confirms that a major component of first push exudation is a consequence of oxidation reaction in the AZS - there is no oxidation reaction or significant exudation under argon atmosphere. Any open porosity serves to moderate the degree of exudation caused by the product gas pressure. However, even a fully dense sample would not exude significantly in the complete absence of this driving force, so that the ideal AZS material would be fully oxidized and could be fully dense.

A reduced AZS material is expected to exude more than an oxidized AZS material, assuming other factors (porosity, sample location, etc.) to be constant. This is the usual experience observed from the ASTM test as discussed above. In order to further test this proposition, a series of six reduced CS3 AZS blocks and six oxidized CS3 AZS blocks were prepared in the production facility at Falconer, by varying process conditions during fusion. Three core samples were carefully drilled, from equivalent locations in each block, for exudation testing using the ASTM (isothermal) technique.

Prior to exudation testing, pre-test open porosity volumes of reduced and oxidized cores were measured by difference between water and mercury immersion, as described previously. Data are presented as Figure 7(a), as the second and third set of bar charts respectively. Each consecutive bar represents a result obtained from one of the three cores taken from each of the six reduced and six oxidized cores, so that there are 18 bars in each set. Exudation data obtained from the same cores by water immersion (to allow comparison with other studies) are similarly presented as Figure 7(b). The data presented as the first set of bars in each chart are discussed in Section 2.5.

Figure 7(a). Open Porosity in AZS

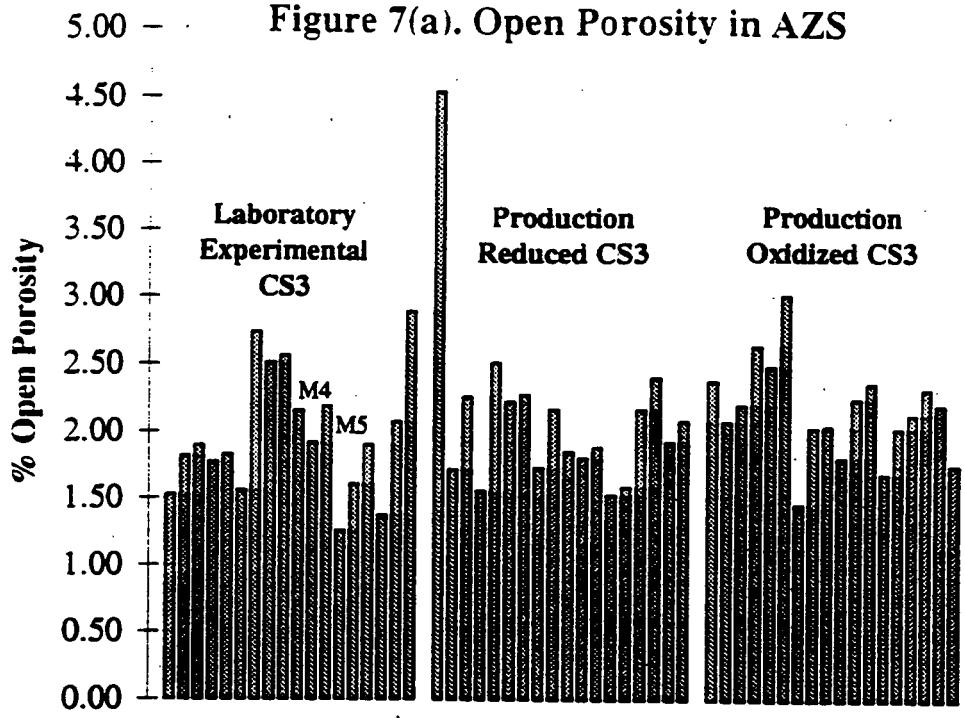
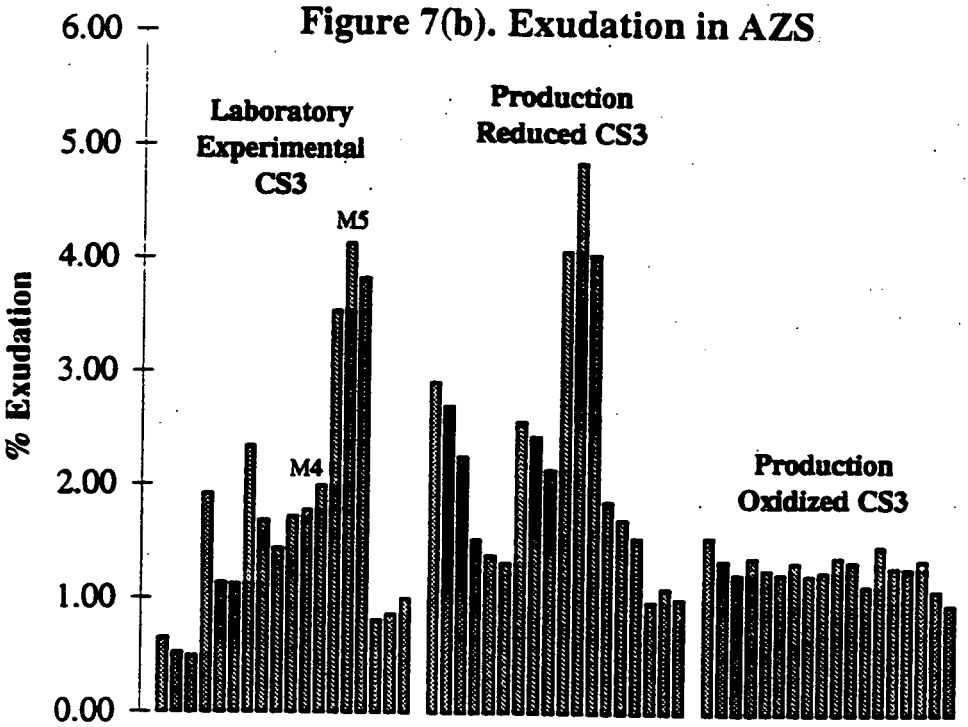


Figure 7(b). Exudation in AZS



Reference 9-16

By comparing data in the second and third set of bars in Figure 7(a), it may be deduced that both reduced and oxidized cores display a similar range of open pore volumes. Total porosity was also found to be at similar levels in each series of cores. In addition, microstructures were observed to be similar in cores of each type, and microanalysis of the glass phases gave similar results to those described previously for M5 and M4 (see also Table 1.). The only observable difference between the cores was color, with oxidized cores appearing uniformly buff and reduced material exhibiting a variable degree of gray tinge.

Despite the physical and chemical similarities between the reduced and oxidized cores, a striking difference can be seen in their exudation behavior - see Figure 7(b). Oxidized material yields lower exudation volume, with much less scatter around the mean, than reduced material. The current manufacturing process at Monofrax Refractories Division is adjusted to produce oxidized AZS, by introduction of molecular oxygen to the melt during the fusion process. The product therefore exhibits the low exudation volumes represented in the third set of bars in Figures 7(a) and (b). This AZS refractory is as close to the ideal fully oxidized fully dense material as current production technology will allow.

2.5. AZS Glass Phase Chemistry And Exudation:- A number of AZS refractories were produced in the laboratory scale arc furnace at Monofrax. A series of six materials, M1 through M6, with varying composition and oxidation state was selected for study. All compositions were comparable to Monofrax CS3 in terms of their zirconia and alumina contents, but the composition or redox state of the glassy matrix phase in each material varied. Glass phase compositions were determined for each material by microanalysis and are presented in Table 1. The glass compositions are also plotted in projection on the Na_2O-Al_2O_3-SiO_2 phase diagram[5], presented as Figure 8, to facilitate comparison.

Three cores were taken from each experimental block, as previously described for the comparison of reduced and oxidized AZS refractories manufactured in the production facility. Open porosity and exudation volumes (water immersion) were obtained and are presented in Figures 7(a) and (b), in the first set of bars. Mean exudation data (of 3 cores) for each refractory is also presented in Table 1.

M1 is a high purity oxidized AZS composition with extremely low concentration of polyvalent ions in the glass phase. This refractory is snow white in color and exhibits the lowest exudation volume when exposed to the ASTM test. The Na_2O content of the glass phase is relatively high in comparison to most of the other compositions, and this is accompanied by an increased level of alumina.

Table 1. AZS In-Situ Glass Phase Chemistry and Exudation

AZS	AZS In-Situ Glass Phase Analysis (wt%), Combined SIMS/EDS									Exudation (vol%)*	Porosity (vol%)**
	SiO2	Al2O3	Na2O	ZrO2	B2O3	TiO2	Fe2O3	CaO	Y2O3		
M1	66.08	22.76	8.50	2.56	0.00	0.01	0.04	0.05	0.00	0.5	1.7
M2	72.10	18.45	6.26	1.85	0.01	0.24	0.33	0.45	0.29	1.4	1.7
M3	71.50	19.00	6.24	2.19	0.01	0.30	0.28	0.23	0.26	1.8	2.6
M4	68.04	20.46	6.19	3.65	0.49	0.56	0.21	0.14	0.27	1.8	2.1
M5	67.98	20.77	6.59	3.16	0.47	0.46	0.17	0.15	0.26	3.8	1.6
M6	53.81	28.50	15.14	1.59	0.50	0.17	0.03	0.13	0.12	0.9	2.1

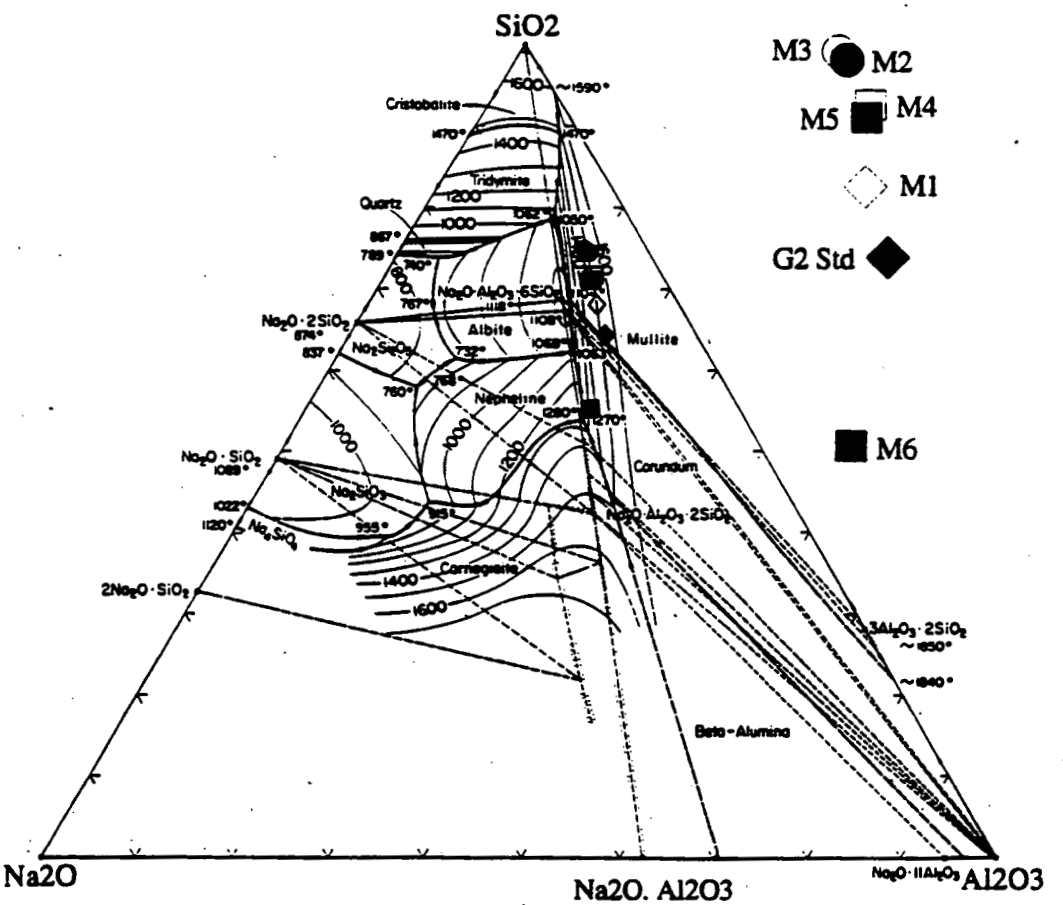
Glass Oxidation State Undetermined

* Mean Water Immersion Exudation (ASTM) from previously untreated cores

** Mean Open Porosity measured before exudation testing

000315

Figure 8. AZS In-Situ Glass Phase Chemistry Microanalysis Nov. 93.



Data from Table 1. plotted in projection on Figure 501 from Phase Diagrams for Ceramists[6]. Note that all glass compositions lie at a similar temperature on the liquidus and form a line parallel to SiO_2 - $Na_2O.Al_2O_3$. Any addition of Na_2O to the AZS batch must reside in the glassy matrix of the final product, increasing the solubility limit for alumina. Associated $Na_2O.Al_2O_3$ units complete a structural role similar to that of SiO_2 tetrahedra in the glass network. Thus, charge balance, together with degree of 'internal coherence' of the glass network structure, is automatically compensated for by the increased presence of alumina -which always attains saturation concentration. This maintains a relatively constant set of glass phase properties, despite variation in chemistry, so that viscosity and T_g do not vary substantially from one AZS composition to another.

Reference 9-19

M2 and M3 are both oxidized AZS materials made without intentionally added B_2O_3, but containing similar levels of Fe_2O_3 and TiO_2. M3 contains slightly more open porosity than M2. Exudation volumes are very similar.

M4 and M5 are both the normal Monofrax CS3 composition, as previously discussed. M5 was intentionally produced in the reduced state in order to compare exudation behavior with that from the normal oxidized product. Note that M4 and M5 glass phase compositions are essentially identical, but can be differentiated from M2 and M3 due to their B_2O_3 content, Fe/Ti ratios and generally lower CaO content. The oxidized material (M4) yields a similar volume of exudate as M2 and M3, but the reduced material (M5) gives considerably more exudation in the same test.

M6 is a special oxidized composition, produced with ~3 times more soda in the batch than normal CS3 AZS. This material also exhibited a large range in open porosity as a function of depth. The impurity oxides have been diluted to low levels, due to the large volume of glassy phase generated in the presence of this unusually high alkali content. Despite the large Na_2O content and consequential increased volume of glass, this material produces a low exudation volume when submitted to the ASTM test. Note again that the high Na_2O concentration in this glass is accompanied by an enhanced presence of dissolved Al_2O_3.

It is currently believed by certain parts of the glass industry, and some refractory manufacturers, that alkali content of AZS materials is an important parameter in determining exudation volume. However, the data presented here demonstrate clearly that addition of extra Na_2O to the AZS batch does not lead to increased exudation in the ASTM test. This behavior is easily understood by considering both the driving force for exudation and also considering the structural network theory of glass formation[7].

Within the range of normally applied compositions, addition of extra Na_2O to the AZS batch does not change the redox state of the final product block - and therefore does not affect the driving force for exudation. In the extreme example of M6, a high volume of open porosity in some segments may even serve to moderate the driving force for exudation by relieving gas pressure.

Addition of extra Na_2O to the AZS batch results in higher concentration of Na_2O in the glassy matrix phase of the product refractory. However, the extra alkali does not enter the glass structure alone as a single modifying cation, but allows an increased solubility for alumina which accompanies it into the network structure in 1:1 molar ratio. This $Na_2O.Al_2O_3$ interaction does not cause an increase in non-

bridging oxygen that would occur due to addition of Na_2O in the absence of Al_2O_3. Thus, the degree of 'internal coherence' of the glassy network is maintained and properties are virtually unaffected. This means that speculation of low viscosity AZS glassy phases, rich in Na_2O, is incorrect. This can be proven by assuming Stevels' model [7] to calculate the average number of bridging oxygen per tetrahedra in the glass structure, which is essentially unchanged for the range of glass compositions exhibited by M1 through M6.

Only refractory M5 was intentionally produced in the reduced state, and all other materials were oxidized within the limits of production capability. However, the high purity M1 material produced considerably less exudation than the other materials. This may be seen as evidence that polyvalent redox reactions do contribute to first push exudation, but more work is required to prove this beyond reasonable doubt.

Logic developed from the data and discussion presented in Sections 2.4. and 2.5. leads us to a governing principle employed in manufacturing Monofrax AZS to produce low first push exudation volumes:-
The size of driving force due to oxidation reaction(s) is determinant and other factors (porosity, etc.) may serve to adjust the volume of exudation experienced. Na_2O content of CS3 AZS in-situ glass phase has no effect on exudation volume within reasonable limits. The objective in manufacturing optimized material should then be to remove the driving force for exudation, by production of oxidized material.

While very high purity raw materials (M1) may further optimize exudation performance, the current market for fused-cast refractories seems unwilling to pay the cost of manufacture of such premium product. Upon exposure to corrosive atmosphere in glass melting service, even high purity AZS may produce defects due to liquid drips and runoff - but this is not due to exudation, as will be discussed in the next section.

3. AZS VAPOR PHASE CORROSION IN GLASS MELTING SERVICE

During service in glass melting furnaces, AZS refractory is subjected to attack by components of the glass melt, either by direct contact with the liquid melt or through vapor phase transport of volatile species. Corrosion of AZS via the liquid phase has been extensively treated in the literature [4],[8],[9] and will be only briefly reviewed here. Corrosion of AZS and other refractories via the vapor phase has previously been given less attention, but with the introduction of

oxyfuel combustion technology this topic has now become a major concern for glass producers and refractory manufacturers. Monofrax Refractories Division is currently pursuing an extensive study of refractory vapor phase corrosion under oxyfuel and airfuel combustion atmospheres. Regardless of the path that corrosive species take, via liquid or vapor phase, the presence of any open porosity or open flaws in the AZS will only enhance their rate of introduction.

At and below the melt line, attack via the liquid phase leads to corrosion of AZS by diffusive transport of species across the interface. Components of the glass melt, usually alkali and alkaline earth species, rapidly diffuse into the AZS surface through the glassy matrix and any flaws. The presence of these structural modifiers tends to cause dissolution of the AZS alumina phase, which then diffuses out into the infinite sink of the glass melt. The AZS zirconia phase, being less soluble in the liquid at the interfacial transition zone, tends to remain undissolved and can eventually be washed into the glass melt by local currents to cause defects. Thus, the corrosive interface progresses into the AZS block, at a faster rate where currents due to convection and glass - pull are highest.

Above the melt line, alkali and other cations. can be taken from the surface of the glass melt into the vapor phase, by formation of volatile hydroxide and other species - such as NaOH, KOH, BOH, PbO, SbO, Na_2SO_4, etc. In certain parts of the furnace there is also opportunity for direct contact of gas - borne batch dust with the refractory. These corrosive species again diffuse into the AZS surface decreasing the liquidus temperature and liberating alumina into the AZS matrix. However, in this case, there is no directly contacting sink for the alumina so it remains on the AZS surface concentrated in solution in the large liquid pool containing large amounts of zirconia crystals, created due to vapor phase corrosion. Eventually, enough liquid can be created to cause drips and runoff which may become defects in the glass melt. Some glass compositions are more easily able to digest these defects than others, and longer residence times can aid in this digestion.

Given the different conditions of corrosive genesis of defects created above and below the glass melt line, it is often possible to distinguish the source of these defects by using microanalysis to compare their Al_2O_3 and ZrO_2 contents. Since corrosion is an ongoing process throughout the life of a furnace, the glass producer prefers the most corrosion resistant refractory in order to extend furnace life and decrease defect generation.

AZS subjected to service above the melt line in glass melting furnaces may therefore generate liquid phase drips and runoff due to vapor phase corrosion by

interaction with volatiles from the glass melt. This long term liquid source of defects has been mistaken for exudation in some instances. However, its origin is very different and its occurrence cannot be forecast from the results of exudation trials such as the ASTM test. On cooling to room temperature, a sample of this liquid phase corrosion product will usually become opaque or white in color, due to the presence of crystalline phases throughout its volume. However, once the liquid drips into glass melt, it can lose alumina and exchange other species by diffusion, so that the defect generated in this manner may remain completely glassy upon cooling.

True exudation, as defined by ASTM type testing, occurs in the absence of corrosive species and creates a transparent glassy surface layer on the AZS when cooled to room temperature. This effect is usually experienced as a short term phenomenon, it cannot continue as a defect source over months or years in an isothermal environment, since its driving force must be exhausted with time. It has been shown that exudation due to temperature cycling of AZS containing polyvalent species may continue for longer times[2] than exudation under isothermal conditions. However, under real glass melting conditions this effect must also be combined with vapor phase corrosion as a source of defects.

4. CONCLUSIONS

First push exudation is predominantly caused by oxidation of the AZS at high temperatures. Gas pressure, generated during this oxidation, pushes a proportion of the AZS glassy matrix phase onto the refractory surface. Oxidized AZS exudes less than reduced AZS, and the presence of open porosity may serve to moderate the degree of exudation experienced. Exudation volume is independent of the Na_2O content in the AZS compositions studied.

First push exudation causes defect generation during first heating of AZS refractories placed in service in a glass melting furnace. However, this exudation is a short term phenomenon under isothermal conditions, and any further liquid generation on the AZS surface is due to corrosive interaction with the glass melt. Above the glass melt, long term liquid generation on AZS surfaces due to vapor phase corrosion may lead to continuing production of defects. Increased presence of open porosity in the AZS is expected to exacerbate the degree of corrosion and thereby increase liquid formation.

The ASTM exudation test C-1223-92 may be used to qualitatively compare first push exudation volumes from refractories subjected to the same test. It does not

take into account the corrosive environment of a glass melting furnace and cannot be used to compare propensity for long term liquid generation.

The current manufacturing process at Monofrax Refractories Division is adjusted to produce oxidized AZS, by introduction of molecular oxygen to the melt during the fusion process. The product therefore exhibits the low exudation volumes represented in the third set of bars in Figures 7(a) and (b). This AZS refractory is as close to the ideal fully oxidized fully dense material as current production technology will allow.

In furnaces subjected to temperature oscillations as low as 50°C[2], polyvalent ion redox exchange may extend exudation for longer time periods. However, a corrosion component must also be present in generating liquid on the AZS surface. Polyvalent ion redox exchange may contribute to first push exudation, even from oxidized AZS, but further work is necessary to prove this. High purity AZS, with low polyvalent ion content, gives the lowest exudation volumes but is not economical to produce.

ACKNOWLEDGMENT

The authors would like to thank S Gould and JH Gibson for their important contributions to the work presented in this paper.

REFERENCES

[1]. Walrod D, 'How Furnace Operation Affects AZS Glass Phase Exudation', Glass Industry (July 1990).
[2] Dunkl M, 'Studies On The Glassy And Reaction Phases Given Off By Fused Cast AZS Blocks And Their Effects On Glass Quality', Glastech. Ber., **62**, 11 (1989), 389 - 94
[3]. Stevens R, 'Zirconia And Zirconia Ceramics', 2nd Edition (1986), Publication 113 by Magnesium Elektron Ltd.
[4]. Trier W, 'Glass Furnaces Design Construction And Operation', English Translation by Lowenstein KL, Society Of Glass Technology, England (1987).
[5]. ASTM Standards, **15.01**, (1995).
[6]. Levin EM, Robbins CR and McMurdie HF, Figure 501, Phase Diagrams for Ceramists, **1.**, American Ceramics Society Inc. (1964).
[7]. Kingery WD, Bowen HK and Uhlmann DR, 'Introduction To Ceramics' 2nd Edition, Wiley, (1976).

[8]. Leaskey LA and McHale AE, 'Micro And Macro Aspects Of Refractory Corrosion', Ceram. Trans. 4 (Adv. Reract. Technol.), (1991), 453 - 63
[9]. Manfredo LJ and McNally RN, 'The Corrosion Resistance of High ZrO_2 Fusion - Cast AZS Glass Refractories In Soda Lime Glass', J. Mats. Sci., **19** (1984), 1272 - 76
[10]. Schreiber HD, 'Redox Processes In Glass Forming Melts'J. Non Cryst. Solids **84** (1986)129-141

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CARBORUNDUM

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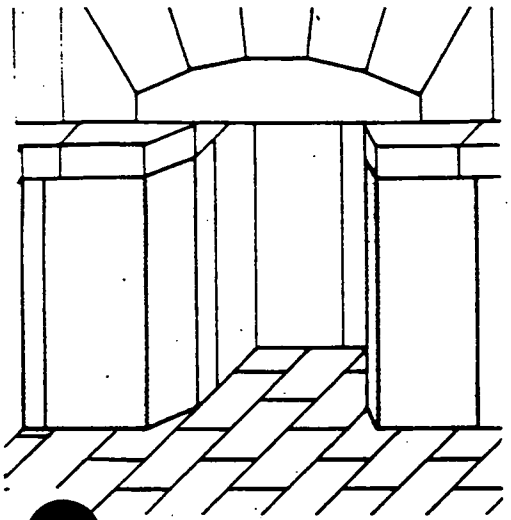
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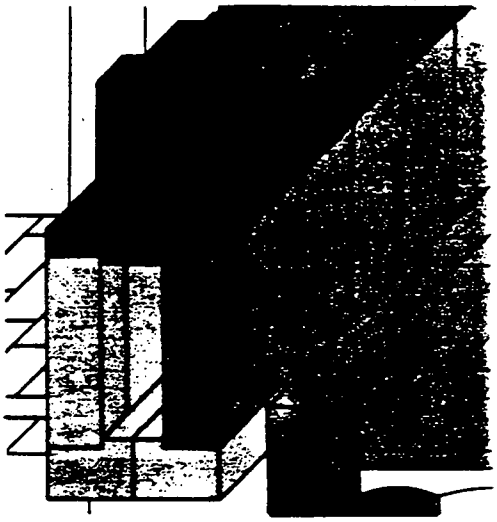
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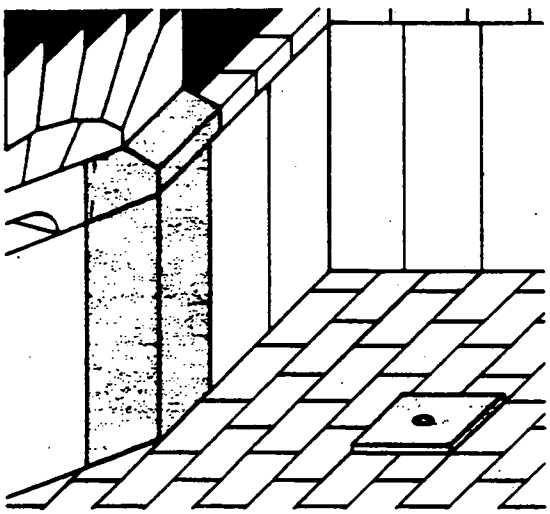
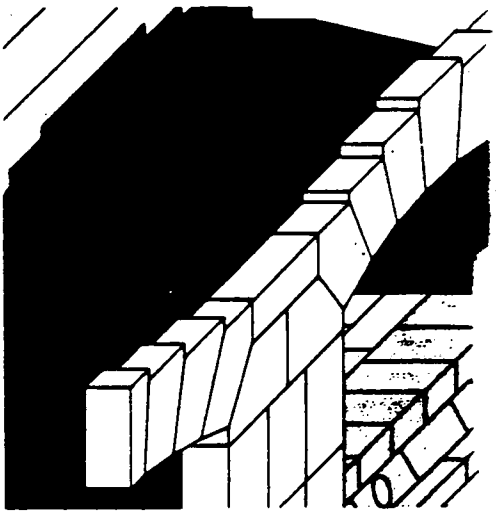
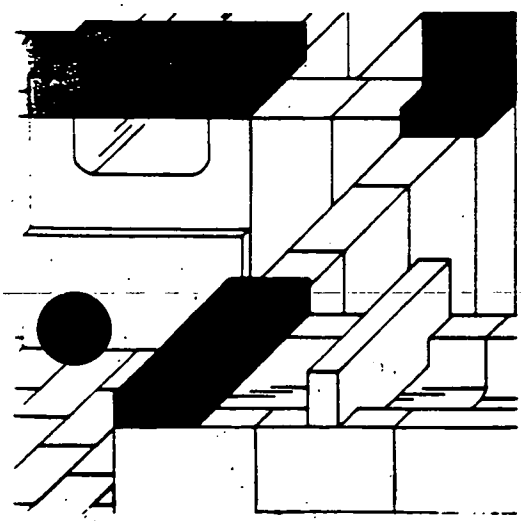
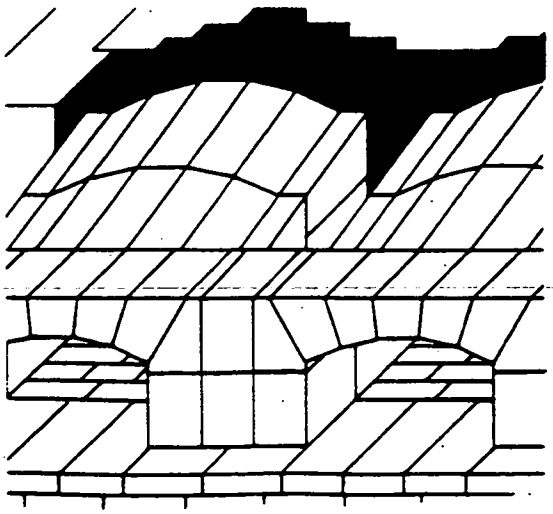
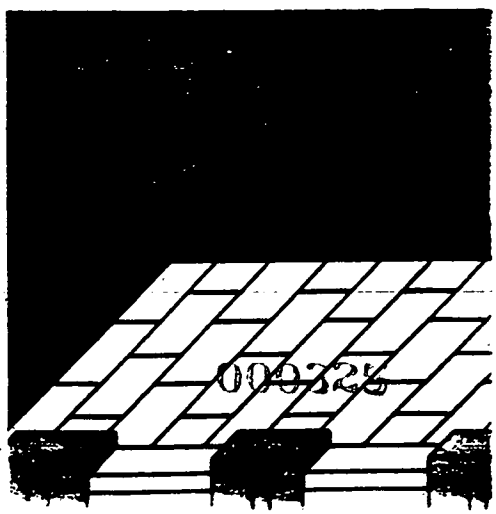
Monofrax® Refractory Systems for Glass Furnaces

Carborundum Products

CARBORUNDUM





000326

Glass Furnace Refractories

The Carborundum Company manufactures Monofrax® fused cast refractories which improve glass quality and extend tank life. Each, by virtue of strength and structure, plays a specific role in withstanding the varied and harsh conditions present in modern glass tanks. No single refractory has the properties to meet all the varying conditions in a glass furnace.

The Monfrax product line, the world's broadest, includes:
—Types A, M, and H Monofrax aluminas which offer high purity, and low seed, cord, and stone evolution.
—Conditioned S-3, S-4, and S-5 Monofrax alumina-zirconia-silica (AZS) mixes which are exceptionally erosion and corrosion resistant at high temperatures.
—Types E, K-3 and B Monofrax chrome-bearing refractories resist the most severe erosion and corrosion in throats, hot spots, sidewalls, and other problem areas in fiberglass furnaces. These products may cause glass coloration.

We recommend specific Monofrax refractories, based on their unique properties for glass furnace melters, refiners, and superstructures. With the appropriate choice of Monofrax composition and casting technique coupled with proper insulation, these refractories extend tank campaigns, improve pack percentage and lower fuel and maintenance costs.

Other Products and Services
The Carborundum Company also provides solutions to other glass plant operating problems. For example:
—Fiberfax® ceramic fiber products insulate glass tanks and protect refractories from thermal shock during hot repairs.

Our engineers can frequently simplify refractory designs and eliminate costly and unnecessary deviations from rectangular blocks.

Petrology

To apply fused cast refractories intelligently, an understanding of their microstructure is important.

Corrosion resistant zirconia is the prime ingredient in AZS mixes. Zirconia crystals, set in alumina and a silica glass, are as important to the performance of AZS refractories as diamonds, set in metal, are to the service life of diamond saws. The glass phase is the key in AZS products because it softens in service to accommodate the erratic thermal expansion and contraction behavior of crystalline zirconia, which is 33% to 40% of AZS fusions. In normal glass furnace thermal cycling, zirconia's behavior stresses a glass-free structure. However, AZS' glass phase is the weakest part of the refractory's structure in furnace service. It dissolves first, followed by alumina and finally zirconia.



Type S-3 33% Zirconia (50X)
A micro section of Monofrax CS-3 refractory, photographed with reflected light, shows laths of corundum (Al_2O_3) mottled with included zirconia crystallites (white) in a field of darker gray representing the siliceous glass matrix. White chains locate the intersection of branching zirconia dendrites with the plane of the section. The non-crystalline siliceous phase makes up the remainder of the refractory.



Type S-4 36% Zirconia (50X)
A micro section of Monofrax CS-4 refractory, photographed with reflected light, shows alumina crystalized in elongated, generally oriented forms with fine, well dispersed included crystallites (white) of zirconia. Larger, white rounded granules of zirconia are scattered through the microstructure. Alumina and zirconia are crystallized in a glassy matrix, represented as the darker gray areas.



Type S-5 40% Zirconia (50X)
A micro section of Monofrax CS-5 refractory, photographed with reflected light, shows a composite crystal development of alumina (Al_2O_3) with included zirconia (ZrO_2). The light gray regular forms mottled with fine zirconia crystallites (white) represent this composite phase in the photo. The section also shows a high concentration of zirconia as white rounded granules generously scattered through the body. These crystalline phases, solidified from the melt, are fixed in a siliceous non-crystalline matrix (dark gray areas).

000327

Reference 10-3

Alumina and chrome bearing refractories are essentially glass-free, totally crystalline materials. The lath-like beta alumina crystals in type M interlock with the chunky alpha alumina crystals in a dense structure. Sodium beta alumina, most stable in alkali atmospheres, slowly reverts to alpha alumina in contact with molten batch carryover. In K-3, extremely corrosion resistant chromic oxide is uniformly distributed in both the R_2O_3 and spinel phases. Type E, with typically 78% chromia, provides excellent corrosion resistance to insulating fiberglasses. Type B, with higher chromia and lower accessory oxide contents, is excellent with glasses having high alkaline or high alkaline/earth oxides.



Type H-Beta Alumina (50X)
Beta alumina, crystallized from an electric furnace melt of soda and alumina, forms 100% of this unique glass-free fusion.



Type M Alpha-Beta Alumina (50X)
Essentially glass-free, this fusion is a structural intergrowth of 45% alpha alumina (Al_2O_3) and 55% lath-like beta alumina ($Na_2O{\cdot}11Al_2O_3$).



Type E-78% Chromia (50X)
Major crystalline phase (Mg, Fe) (Cr, Al, Fe)O_4 comprises approximately 50% of the microstructure. It envelopes 45% R_2O_3 phase (Cr, Al, Fe)$_2O_3$ and 5% glassy matrix.



Type K-3 Chrome-Alumina (50X)
Solid solution crystals of chrome-alumina comprise 60 to 65% of this refractory, glass 3% to 4%, and a complex magnesia-chrome-alumina spinel the remainder.



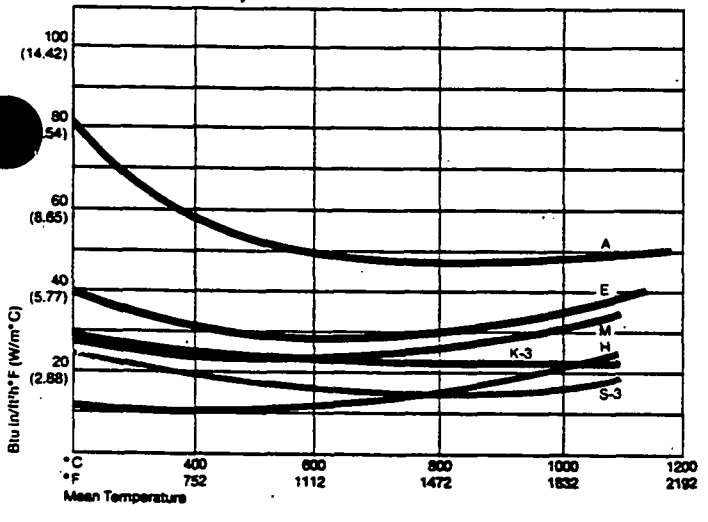
Type B-83% Chromia (50X)
Major crystalline phase is typically 75% (Cr, Al)$_2O_3$ solid solution, 20% (Cr, Al)O_4 phase and 3% interstitial glass.

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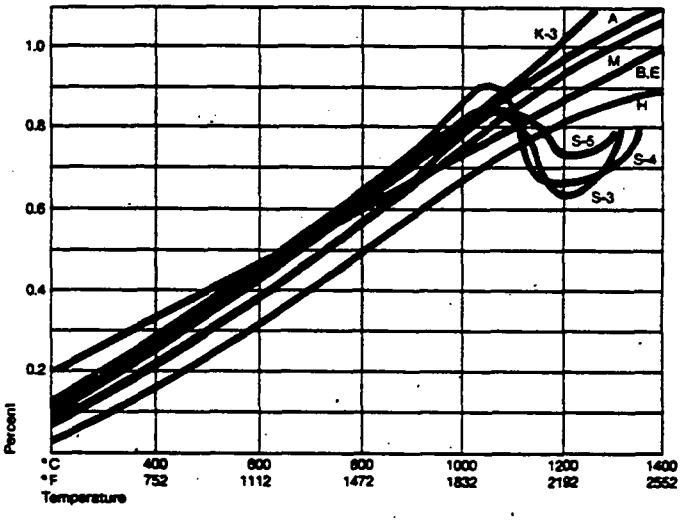
Properties

Monofrax Refractory Type	Typical Chemical Composition (%) Al_2O_3	ZrO_2	Cr_2O_3	SiO_2	Alkali and Accessory Oxides	Typical Physical Properties Bulk Density lb/ft³(g/cm³)	Apparent Porosity (%)
S-3, Conditioned	49.7	33.0	—	15.3	2.0	231 (3.70)	1.0
S-4, Conditioned	47.8	36.4	—	14.0	1.8	236 (3.78)	1.0
S-5, Conditioned	45.8	39.5	—	12.9	1.8	246 (3.94)	1.0
A	98.2	—	—	0.7	1.1	235 (3.76)	2.0
M	94.5	—	—	0.8	4.7	212 (3.40)	2.0
H	93.3	—	—	0.3	6.4	195 (3.12)	4.0
K-3	58.6	—	27.1	1.6	12.7	239 (3.83)	4.0
E	6.5	—	77.7	1.7	14.1	260 (4.17)	5.0
B	13.7	—	82.2	0.8	3.3	285 (4.56)	5.0

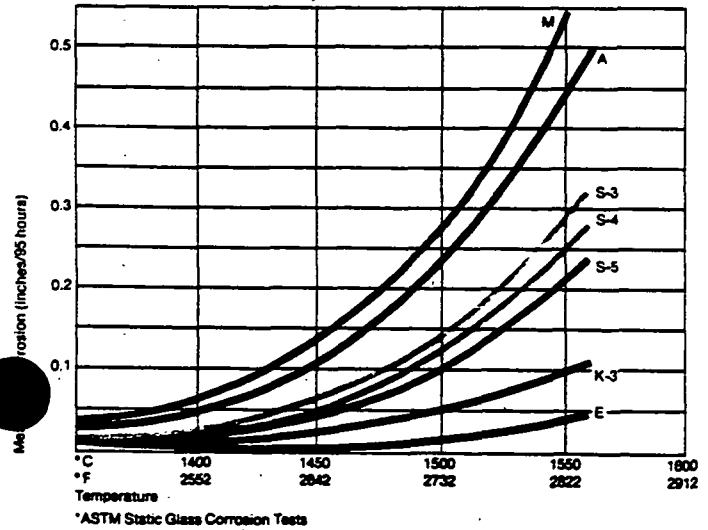
Thermal Conductivity



Thermal Expansion

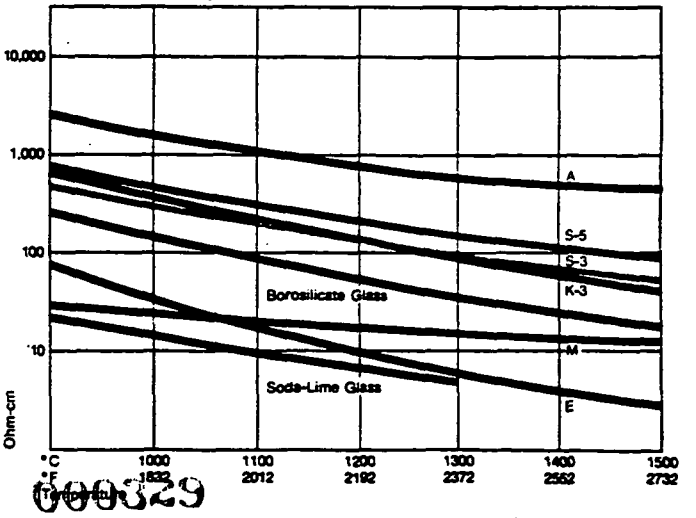


Metal Line Corrosion vs. Temperature for Soda Lime Glass



*ASTM Static Glass Corrosion Tests

Electrical Resistivity vs. Temperature



000329

Reference 10-5

Casting Techniques

As regular fused cast refractories solidify in molds, they develop shrinkage cavities. In most furnace applications these 9-11% voids seldom present problems. However, we have spent years of intensive work to displace, reduce, and eliminate them. As a result, Monofrax refractories can be produced as required in tilt, reduced void, end cast, or end poured, inhibited cavity (EPIC) castings.

Tilt Cast

Blocks are poured and annealed in a way that locates the casting void at the high end of the block, comparable to the bubble in a spirit level. In a glass furnace sidewall, the void is placed away from the maximum erosion zone. Blocks are appropriately stenciled to avoid misuse.

Reduced Void Cast

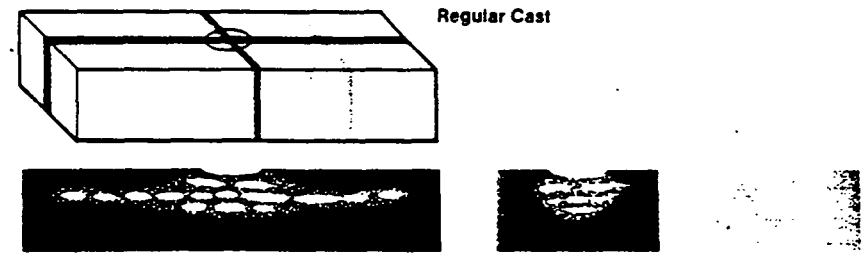
Through repour techniques, up to 80% of the normal void is filled before block annealing occurs, and the amount of useful refractory is increased because of the improved, fine-grain crystalline structure. Reduced void refractories are widely used for throat, doghouse, and electrode blocks, and in maximum erosion zones of sidewalls and bridgewalls.

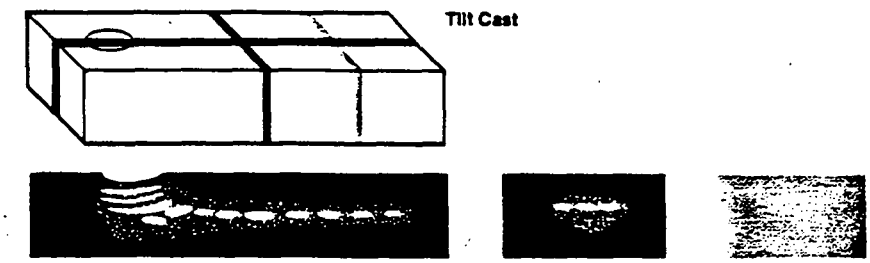
End Cast

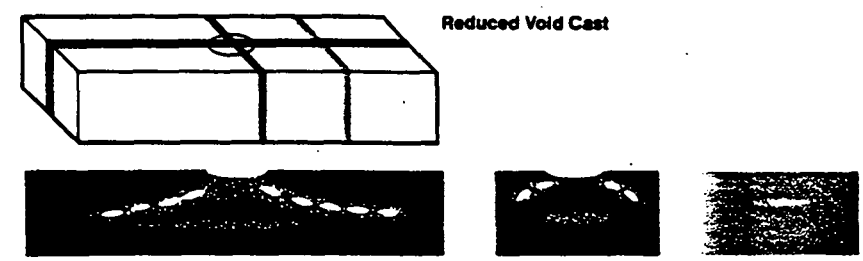
This technique provides economical sidewall castings. The upper portion of the blocks are extremely dense and essentially free of porosity. This zirconia-rich zone provides very high corrosion resistance, equivalent to EPIC-3 quality, and ideal protection against the classic metal line cut.

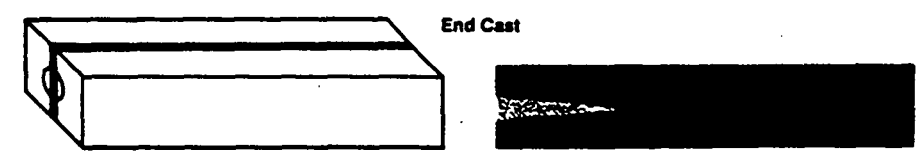
EPIC-3 Cast

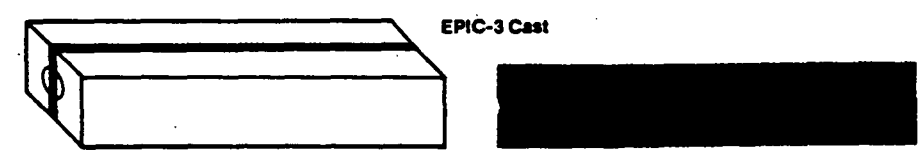
The EPIC-3 casting technique is an end-cast process utilizing carefully timed, multiple pourings into oversized headers. When the refractory is cool, the header is removed with a diamond saw. The EPIC-3 technique produces void-free castings with unsurpassed crystalline uniformity. EPIC-3 castings are recommended for the most critical throat and doghouse blocks, port floors, electric furnace sidewalls, and electrode bubbler blocks. EPIC-3 has replaced the diamond cut lug (DCL) process, except for paving tiles.











000330

Because of their high temperature corrosion resistance in the melter end, Monofrax S refractory mixes should be used for sidewalls, pavers, bubblers, doghouses, electrode blocks and bridgewall tank throats. Corrosion resistance increases as zirconia content becomes higher from Conditioned S-3 to S-5; S-4 and S-5 are respectively 25% and 35% more resistant to soda lime glass corrosion than S-3.

Monofrax S-4 refractories, with an intermediate 36% zirconia content, have the highest thermal shock resistance of all AZS materials. This is extremely significant in electrode and doghouse assembly selection.

Because of their exceptional corrosion resistance, K-3 refractories are widely used in oil, gas, and electric furnaces which melt fiberglass insulation. They are also used in metal line hot spots in flat glass tanks.

Other Melter Refractories

Monofrax A alpha alumina blocks are generally used only for special glasses, e.g., nucleated glasses where zirconia is an unacceptable contaminant. Monofrax E chrome-spinel refractories, available only in void-free EPIC-3 blocks, are used in throats of fiberglass insulation (wool) tanks to equal the exceptional service life provided in sidewalls by Monofrax K-3 EPIC-3 blocks.

Paving

In melter pavings, large DCL tiles reduce glass and tramp metal attacks on joints. Conditioned S-3 DCL tiles are the usual choice.

Laying Cement

Upward drilling under pavers is sharply reduced when Monofrax S zirconia-alumina laying cement is used as bedding and vertical joint mortar. K-3 chrome-alumina cement provides a safety barrier between subcourses of zircon and clay in insulated melter bottoms. In paving studies, Monofrax K cement effectively blocks penetration of soda-lime glass and metal.

K chrome-alumina cement and K-3 RAM with its chrome-alumina fused cast grog are used in wool fiberglass melter pavings. They provide the greatest resistance to glass penetration and tramp metal when their chrome coloration potential can be tolerated.

Throats

Conditioned S-5 EPIC-3 cover blocks provide extended, trouble-free throat service. EPIC-3 void-free blocks have exceptional service life because of their greater crystalline uniformity, fine-grained structure and absence of casting surface shell top than regular cast blocks.

K-3 chrome-alumina refractories have exceptional glass corrosion resistance. They are used effectively in throats, melting opal, amber, and green glasses where limited amounts of this 27% chromic oxide refractory assure minimal risk of glass color contamination.

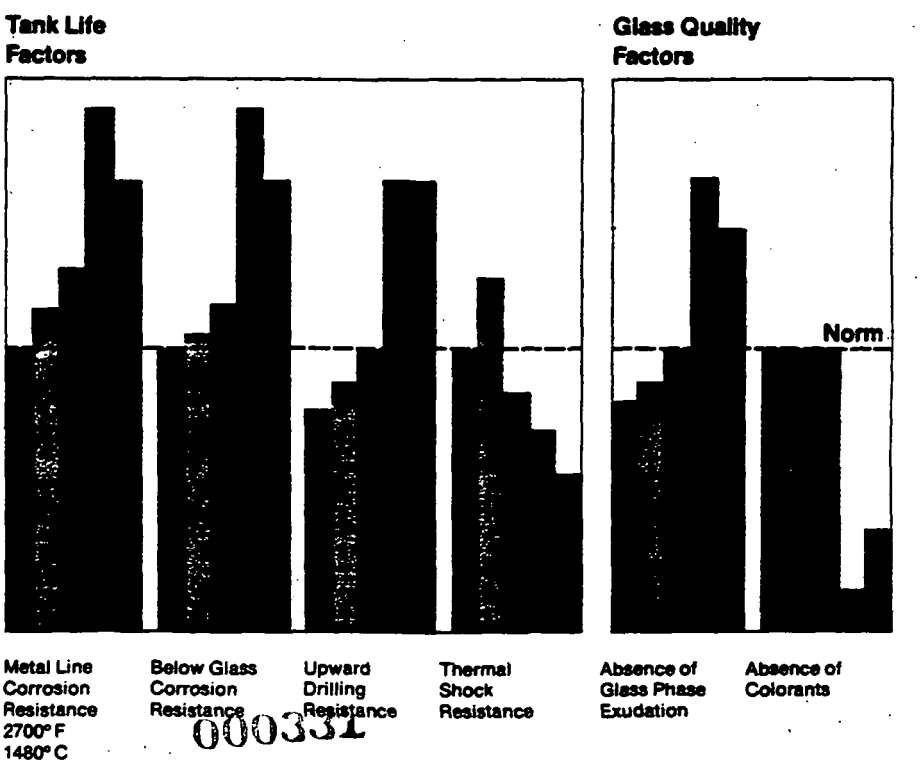
Monofrax E 78% chromic oxide and Monofrax B 82% chromic oxide have the highest corrosion resistances of any fused cast refractory. Because it increases throat life, Monofrax E extends electric furnace campaigns many months.

Premium Blocks

Reduced void, tilt cast, end cast or end cast void-free EPIC-3 cast blocks should be used in throats, doghouse corners, electrode blocks, bubbler blocks and severe wear areas. Selective use of these special structures assures longer, more uniform lining wear throughout the melter, maximizing return on investment with longer campaigns.

Criteria for Refractory Selection

The profile charts compare Monofrax compositions and show how they relate to the key factors of tank life and glass quality in the melter, refiner, and superstructure. Each refractory is critically compared against traditional industry norms for various furnace areas. It is critical to know and use these composition characteristics to choose the ideal refractory, because an all-purpose refractory does not exist.



000331

Reference 10-7

Refiner

- S-3 Conditioned AZS
- S-4 Conditioned AZS
- S-5 Conditioned AZS
- E Chrome Spinel
- K-3 Chrome-Alumina
- M Alpha-Beta Alumina

Refractory Selection:
Glass quality and improved percentage of pack dictate the refractory choice for refiner applications. Any reduction in seed, cord, and stone evolution translates directly into significant improvement in acceptable ware. Even a slight improvement in glass yield significantly increases the profitability of the tank campaign.

The profile chart compares the major factors which affect glass quality. The glass phase content of the S-3 AZS material is 18% by weight. Because of lower specific gravity the glass phase is 25% by volume. In corrosive conditions, the glass phase is attacked first and has the most harmful effect on glass quality.

Generally, Monofrax M refractory is best for glass contact applications in soda-lime refining ends. It equals the refiner corrosion resistance of AZS products at 2500° F (1370° C), and is better at lower refining end temperatures commonly found in float glass canal assemblies and TV glass channels. Conditioned S AZS refractories are suitable for the special operating conditions encountered with borosilicate and some lead glasses.

Soda-Lime and Television Glasses
Crystalline Monofrax M alpha-beta alumina, without zirconia and glass phase, has the lowest seed and blister potential of all glass contact refractories. Thus, it is used internationally in soda-lime refiners and channels. It is also used almost universally in feeder channels for television glass, as refiner paving in flat glass tanks, and as canal sections for float glass.

Monofrax M blocks surpass AZS materials in resistance to horizontal joint attack in refiners.

The higher corrosion resistance of Conditioned S-4 and S-3 provides greater performance balance when glass contact temperatures in the riser sections exceed 2500° F (1370° C) and, typically, in the first few hot end channels of electric tanks. Low carbon and metallic impurities in Conditioned AZS refractories lower the defect potential in refining areas.

Type M is widely used for refiner grounding electrode blocks. Conditioned S-4 and S-5, with higher electrical resistivity and low seeding and glass exudation potential, are ideal for powered refiner electrode blocks.

Monofrax M refractories for feeder entrances and channels are supplied with diamond-trued mating ends at no extra cost. Joint corrosion potential in forehearths is greatly minimized. With diamond-ground joints between feeder entrances and support blocks, upward drilling at horizontal joints is reduced. If the feeder entrance block is slightly recessed from the support block, upward drilling is further minimized.

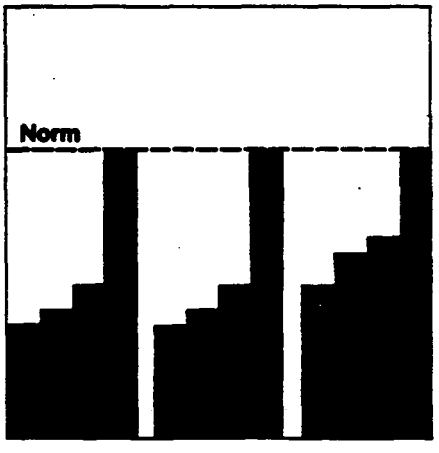
Borosilicate Glasses
Because of their higher operating temperatures, Conditioned S refractories are recommended in borosilicate refiner feeder channels.

Feeder entrances of Conditioned S-5 give added resistance to upward drilling because the glass phase is only 13% versus 18% for common AZS materials. Conditioned S-4 is generally used in feeder bowls.

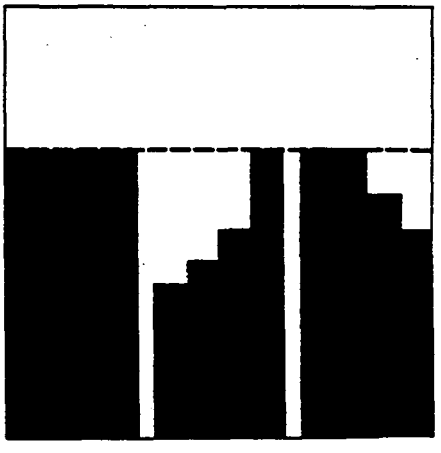
Type K-3 and E chrome fused cast refractories are widely used for glass contact in wool fiberglass forehearths because their corrosion resistances are several times greater than AZS fused cast. Type E chrome-spinel is much superior to bonded chrome refractories in thermal shock resistance while equivalent or better in corrosion resistance.

Laying Cement
Monofrax HMS laying cement is recommended as a bedding and vertical joint mortar for type M refiner pavings. For added service, it is common to clean and pack worn joints in type M sidewalls with HMS cement. This high alumina, airset cement is exceptionally pure and highly resistant to glass corrosion.

Glass Quality Factors



Tank Life Factors



000332

Doghouse End
Combinations of Conditioned S-3, S-4, and S-5 AZS mixes protect against batch carryover, flame erosion, and entrained dust impingement. These refractories have the required abrasion, alkali vapor, thermal shock, and batch carryover resistances. Conditioned S refractories also have low glass exudation at high temperatures.

Conditioned S-4 and S-5 void-free EPIC-3 blocks provide maximum service life in critical port entrance areas.

Port Necks
Conditioned S-4 and type H beta alumina materials deliver the best refractory balance in port necks where very high thermal shock resistance and dryness are most important. Low glass phase exudation is also important in port necks because clean, non-dripping materials minimize buildup of batch dust and condensing alkalis. A dry refractory is even more desirable when insulation is used. Insulation lowers thermal gradients and raises the temperature in glass-phase-containing refractories; this accelerates flow of glass to the surface. High alkali resistance is also desirable.

Type S-4, a 36% zirconia product, contains only 14% siliceous glass phase. It provides the greatest thermal shock resistance of any AZS composition and is recommended where batch carryover is excessive.

Glass-free Monofrax H refractory has the highest tolerance against thermal shock of any fused cast refractory, and is superior to many bonded refractories. It is soda-saturated alumina, and is remarkably dry and stable in hot alkali temperatures. Because of its 100% crystalline composition, Type H can be easily insulated without runout.

Refiner End
In the refiner end, alkali vapor and condensate attack accelerate rundown and drip; stones and cords can be formed. All-crystalline, zirconia-free M and H aluminas supply the high temperature inertness required in this section of a glass tank superstructure. Heavily insulated type M and H aluminas protect against glass phase exudation and drip generated by AZS refractories. Zirconia stones and cords encountered with AZS fused cast and bonded refractories are avoided. Heavily insulated AZS products allow more glassy phase, up to 25% by volume, to become liquid and mobile.

Monofrax M and H refractories remain dry at high temperatures and in concentrated alkali vapors. This is an important consideration in frontwalls of glass tubing tanks, flat glass canal entrance arches, and feeder entrance lintels of television glass furnaces and container glass furnaces.

In superstructure locations, Monofrax H refractories frequently last for two campaigns.

Monofrax M alpha-beta alumina blocks are superior to type H in solid and block bridgewall superstructures of end-fired tanks because of their batch carryover, abrasion and alkali resistance, and almost total absence of glass phase.

Laying Cement
Zirconia-free Monofrax HMS airset laying cement, ideal for superstructure applications, has been used worldwide for more than 20 years. This high alumina mortar is made from high quality raw materials.

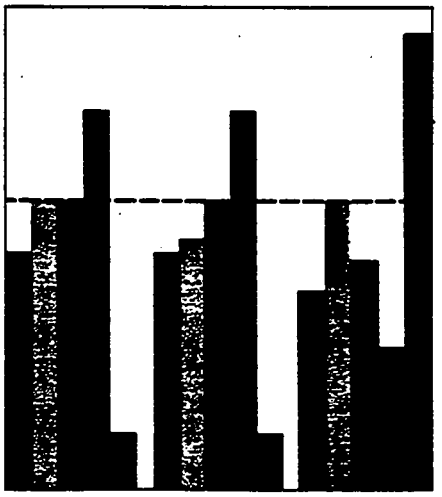
High alumina Monofrax RAM cement is recommended for rammed port bottoms where batch carryover is minimal. It has lower fired shrinkage than type HMS.

Oil Firing
Superstructure performance is affected by the use of fuel oils, especially those high in sulphur and vanadium. A study[1] of vanadium pentoxide attack on superstructures suggests new alternative uses for Monofrax M versus AZS types to solve this problem.

Rugged, glass-free and zirconia-free type M alpha-beta alumina has lower thermal shock resistance than AZS products. However, this can be offset by appropriate insulation which lowers thermal gradients through the Monofrax M blocks. In addition, chunky block design of Monofrax M refractory minimizes thermal stress.

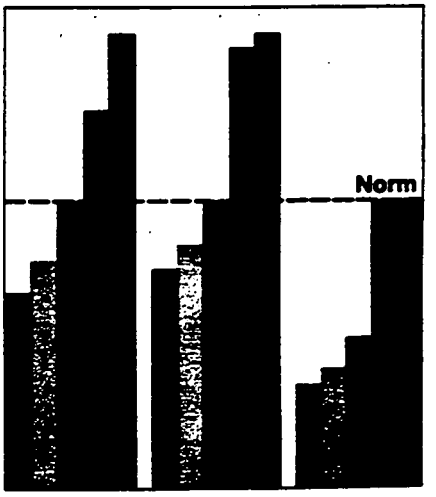
Types M and H will withstand substructure temperatures to 3200° F (1760° C) and can be heavily insulated without subsidence under load.

Tank Life Factors



Batch Carryover Resistance | Abrasion Resistance 2800° F 1550° C | Thermal Shock Resistance

Glass Quality Factors



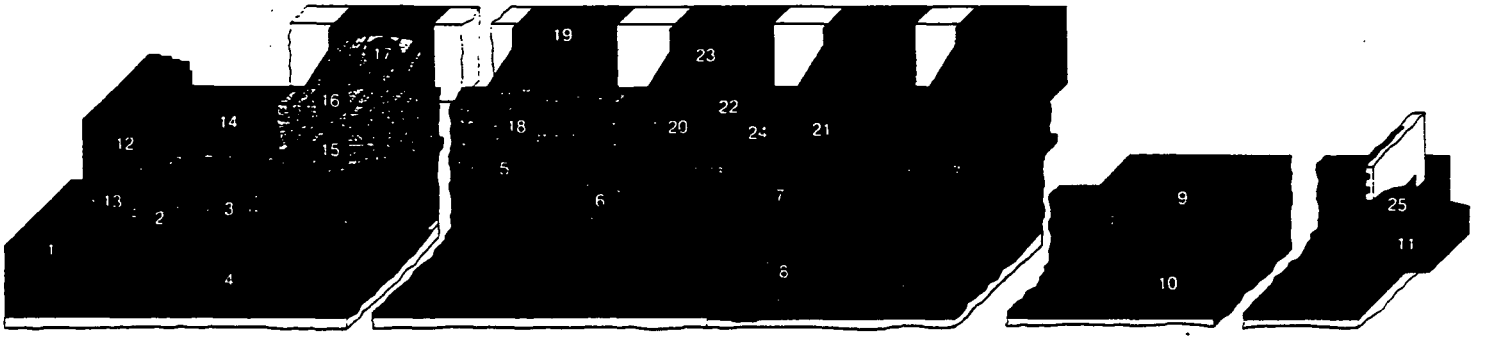
Alkali Resistance | Absence of Glass Phase Exudation | Effectiveness With Insulation

[1]Roy W. Brown and Karl H. Sandmeyer, "Sodium Vanadate's Effect of Superstructure Refractories", *The Glass Industry Magazine*, (November 1978) p. 16, (December 1978) p. 15.

Flat Glass Furnace

Soda-Lime Glass

- S-3 Conditioned AZS
- S-4 Conditioned AZS
- S-5 Conditioned AZS
- H Beta Alumina
- M Alpha-Beta Alumina
- K-3 Chrome-Alumina
- E Chrome Spinel
- Monofrax B



		Furnace Area	Recommended Monofrax Refractory Type	Recommended Casting Technique
Melter	①	Doghouse	Conditioned S-4	Tilt Cast or EPIC-3
	②	Doghouse Corners	Conditioned S-5	EPIC-3
	③	Sidewalls up to Hot Spot	Conditioned S-3, S-4	Regular Cast
	④	Pavers	Conditioned S-3	DCL Tile
	⑤	Side Electrode Blocks	Conditioned S-4	EPIC-3
	⑥	Hot Spot	Conditioned S-4 or S-5	End Cast
		Hot Spot Alternative	K-3	EPIC-3
	⑦	Sidewalls beyond Hot Spot	Conditioned S-3	Regular Cast
	⑧	Bubblers	Conditioned S-5	EPIC-3
Refiner	⑨	Sidewalls	M	Regular Cast
	⑩	Pavers	M	DCL Tile or Regular Cast
	⑪	Float Canals	M	Ground EPIC-3
		Drawbacks, Shutoffs*	M	Regular Cast
Superstructure up to Hot Spot	⑫	Backwall	M	Regular Cast
		Backwall Alternative	Conditioned S-3	Regular Cast
	⑬	Doghouse Arch	Conditioned S-4	Regular Cast
	⑭	Breastwalls	M	Regular Cast
		Breastwalls Alternative	Conditioned S-4	Regular Cast
	⑮	Burners	Conditioned S-4	EPIC-3
	⑯	Port Arches/Jambs	Conditioned S-4 or S-5	Reduced Void
	⑰	Port Necks (1st and 2nd)	Conditioned S-4	Regular Cast
	⑱	Plate Blocks, Port Floors	Conditioned S-4 or S-5	EPIC-3
	⑲	Regenerator Entrances	H	Regular Cast
Superstructure beyond Hot Spot	⑳ ㉑	Port Floors	Conditioned S-3, H	Regular Cast
	㉒	Port Arches	H	Regular Cast
	㉓	Port Necks	H	Regular Cast
	㉔	Breastwalls	H	Regular Cast
	㉕	Front Wall Arch, Veneer	H	Regular Cast

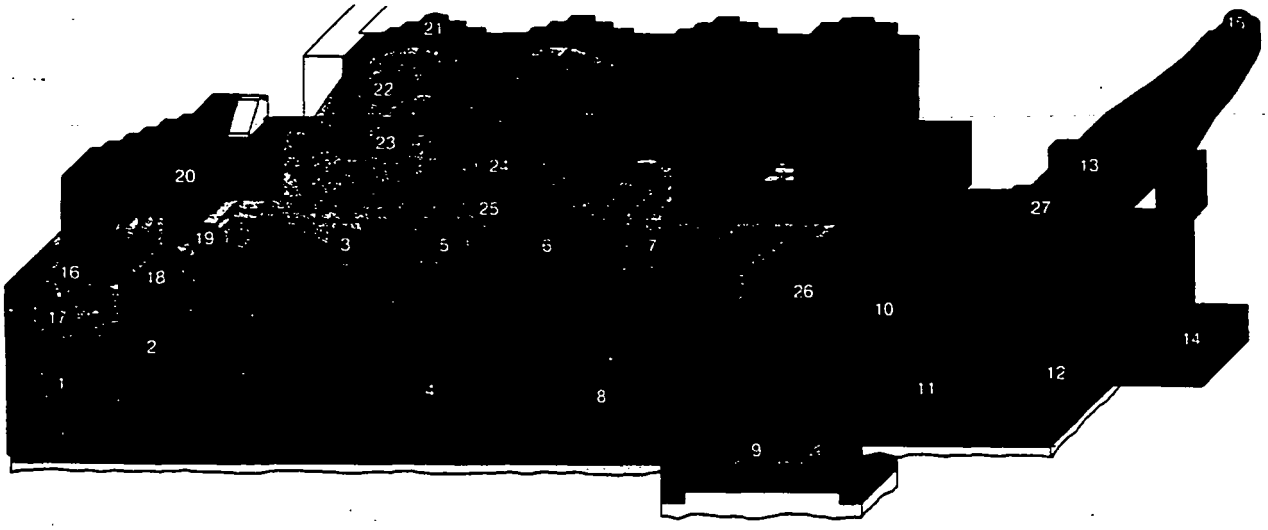
*Not pictured

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000334

Side Fired Furnace

Soda-Lime Glass



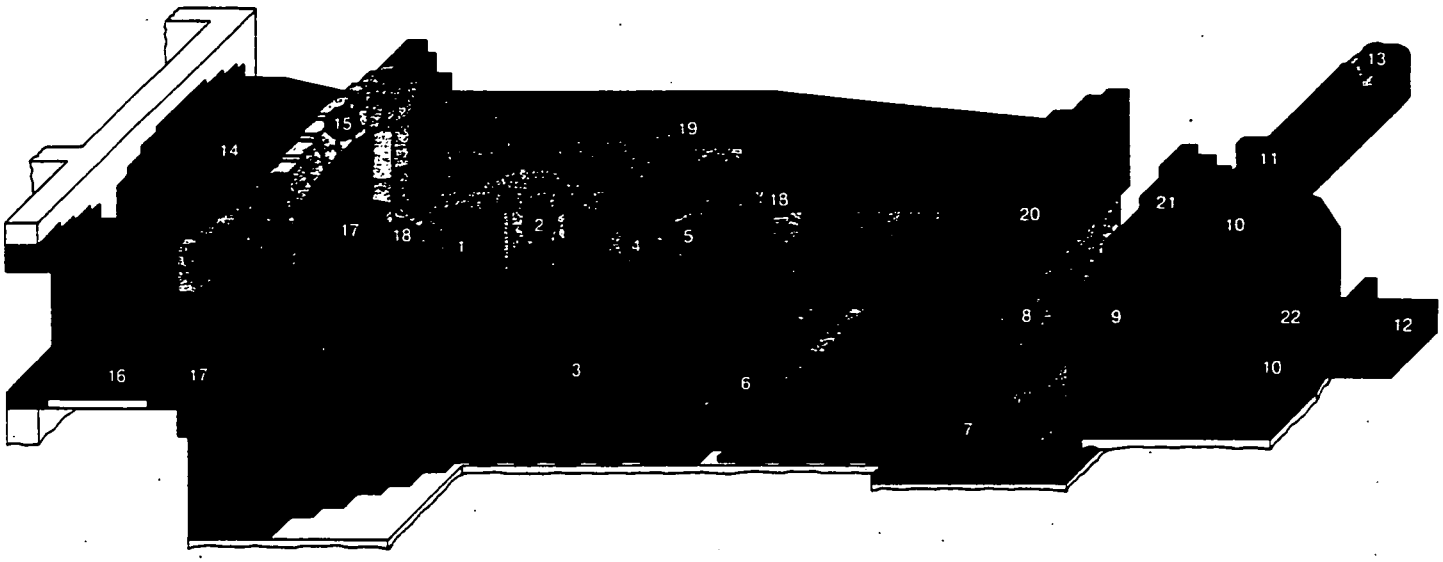
	Furnace Area	Recommended Monofrax Refractory Type	Recommended Casting Technique
Melter	① Doghouse Sidewalls	Conditioned S-4	Reduced Void, End Cast or EPIC-3
	② Doghouse Corners	Conditioned S-5	EPIC-3
	③ Side Electrode Blocks	Conditioned S-4	EPIC-3
	④ Bottom Electrode Blocks	Conditioned S-5	EPIC-3
	⑤ Sidewalls up to Hot Spot	Conditioned S-3	End Cast, Tilt Cast or Regular Cast
	⑥ Hot Spot	Conditioned S-3	End Cast, Tilt Cast or Regular Cast
	⑦ Sidewalls beyond Hot Spot	Conditioned S-3	End Cast or Regular Cast
	⑧ Pavers	Conditioned S-3	DCL Tile
	⑨ Throat Assembly	Conditioned S-5, S-4	EPIC-3
	Throat Alternative	K-3	EPIC-3
	Bridgewall*	Conditioned S-3	Regular Cast
Refiner	⑩ Sidewalls/Bridgewall	M	Regular Cast
	⑪ Pavers	M	DCL Tile or Regular Cast
	⑫ Feeder Entrances	M	Regular Cast
	⑬ Skimmers	M	Regular Cast
	⑭ Feeder Channels	M	Regular Cast
	⑮ Feeder Bowls	Conditioned S-4	Regular Cast
Superstructure up to Hot Spot	⑯ Doghouse Arch	Conditioned S-3	Regular Cast
	⑰ Feeder Blocks	Conditioned S-4	Reduced Void
	⑱ Doghouse Corners	Conditioned S-5	Reduced Void
	⑲ Tuck Stones	Conditioned S-4	Regular Cast
	⑳ Rear Gable Wall	M	Regular Cast
	Rear Wall Alternative	Conditioned S-3	Regular Cast
	㉑ Regenerator Entrances	H	Regular Cast
	㉒ Port Necks	Conditioned S-4	Regular Cast
	㉓ Port Arches/Jambs	Conditioned S-4, S-3	Reduced Void
	㉔ Port Neck Pavers	Conditioned S-3	DCL Tile
	㉕ Port Sills	Conditioned S-4 or S-5	EPIC-3
Superstructure beyond Hot Spot	㉖ Bridge Covers/Shadow Walls	H, M	Regular Cast
	㉗ Feeder Entrance Covers	H	Regular Cast
	Other Areas	H	Regular Cast

*Not pictured

End Fired Furnace

Soda-Lime Glass

- S-3 Conditioned AZS
- S-4 Conditioned AZS
- S-5 Conditioned AZS
- H Beta Alumina
- M Alpha-Beta Alumina
- K-3 Chrome-Alumina
- E Chrome Spinel
- Monofrax B



		Furnace Area	Recommended Monofrax Refractory Type	Recommended Casting Technique
Melter	①	Doghouse Corners	Conditioned S-5	EPIC-3
	②	Doghouse Sidewalls	Conditioned S-4	Reduced Void
	③	Pavers	Conditioned S-3	DCL Tile
	④	Sidewalls/Backwall	Conditioned S-3	End Cast, Regular Cast or Tilt Cast
	⑤	Side Electrode Blocks	Conditioned S-4	EPIC-3
	⑥	Bubblers	Conditioned S-5	EPIC-3
	⑦	Throat Assembly	Conditioned S-5, S-4	EPIC-3
		Throat Alternative	K-3	EPIC-3
	⑧	Bridgewall	Conditioned S-3	Tilt Cast or End Cast
Refiner	⑨	Sidewalls/Bridgewall	M	Regular Cast
	⑩	Feeder Entrances	M	Regular Cast
	⑪	Skimmers	M	Regular Cast
	⑫	Feeder Channels	M	Regular Cast
	⑬	Feeder Bowls	Conditioned S-4	Regular Cast
Superstructure	⑭	Port Necks	H	Regular Cast
	⑮	Port Arches/Jambs	Conditioned S-4	Regular Cast
	⑯	Port Neck Pavers	Conditioned S-3	DCL Tile
	⑰	Port Sills	Conditioned S-5 or S-4	EPIC-3
	⑱	Tuck Stones	Conditioned S-4, S-3	Regular Cast
	⑲	Breastwalls	Conditioned S-3, M	Regular Cast
	⑳	Solid Shadow Walls	M	Regular Cast
	㉑	Refiner Superstructure	H	Regular Cast
	㉒	Feeder Entrance Covers	H	Regular Cast
		Bridge Covers*	M	Regular Cast

*Not pictured

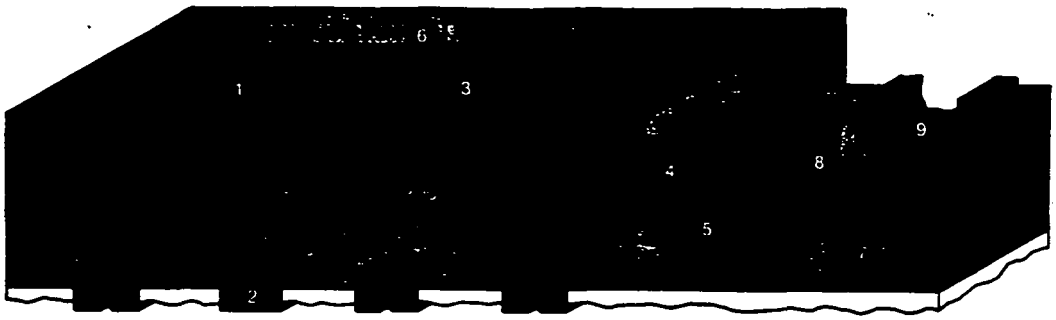
000336

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Reference 10-12

Electric Furnace

Wool Fiberglass Tank



		Furnace Area	Recommended Monofrax Refractory Type	Recommended Casting Technique
Melter	①	Sidewalls	K-3	EPIC-3
		Sidewalls Alternative	E	EPIC-3
	②	Bottom Electrode Blocks	Conditioned S-5	EPIC-3
	③	Side Electrode Blocks	K-3	EPIC-3
		Side Electrode Blocks Alternative	E	EPIC-3
	④	Throat Assembly	K-3	EPIC-3
	⑤	Throat Cover	E	EPIC-3
	⑥	Cap Course	Conditioned S-3	DCL Tile
Riser (Refiner)	⑦	Paving	Conditioned S-3	DCL Tile
	⑧	Sidewalls/Bridgewall	Conditioned S-3, S-5	Tilt Cast, EPIC-3, End Cast
	⑨	Forehearth	K-3	Regular Cast or EPIC-3

Because of increasingly stringent furnace emmission standards, more and more insulating fiberglasses are being melted electrically. There has also been a significant switch from AZS refractories to K-3 chrome-alumina because of its dramatically increased corrosion resistance with these glasses. Chromic oxide coloration is a minimal consideration here.

Type K-3 is generally supplied in EPIC-3 void-free castings. EPIC-3 castings, with their uniform crystalline structure, are easier to properly insulate and have improved thermal shock resistance. Rugged Type E chrome-spinel, with its 78% Cr_2O_3 content, is the recommended fused cast refractory for throats and especially corrosive fiberglasses.

Electric Furnace

Other Glasses

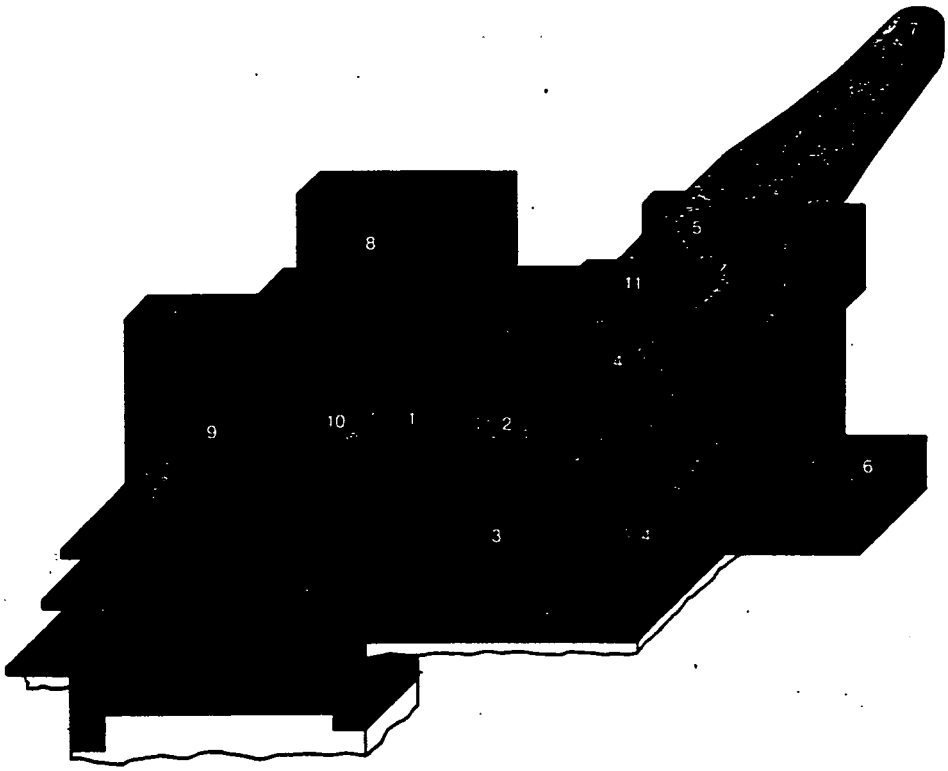


		Furnace Area	Recommended Monofrax Refractory Type	Recommended Casting Technique
Melter	①	Sidewalls	Conditioned S-5	EPIC-3
	②	Side Electrode Blocks	Conditioned S-5	EPIC-3
	③	Pavers	Conditioned S-3	DCL Tile
	④	Bottom Electrode Blocks	Conditioned S-4	EPIC-3
		Electrode Alternative	Conditioned S-5	EPIC-3
	⑤	Throat Assembly	Conditioned S-5, S-4	EPIC-3
Refiner	⑥	Sidewalls/Bridgewall	Conditioned S-4	Regular Cast
	⑦	Feeder — Soda Lime	Conditioned S-3, M	Regular Cast
	⑦	Feeder — Borosilicate	Conditioned S-3	Regular Cast

000337

Borosilicate Glass Refiner

- S-3 Conditioned AZS
- S-4 Conditioned AZS
- S-5 Conditioned AZS
- H Beta Alumina
- M Alpha-Beta Alumina
- K-3 Chrome-Alumina
- E Chrome Spinel
- Monofrax B



		Furnace Area	Recommended Monofrax Refractory Type	Recommended Casting Technique
Refiner	(1)	Sidewalls	Conditioned S-3	Regular Cast
	(2)	Electrode Blocks	Conditioned S-4	EPIC-3
	(3)	Pavers	Conditioned S-3	Regular Cast
	(4)	Feeder Entrances	Conditioned S-4	Regular Cast
	(5)	Skimmers	Conditioned S-3	Regular Cast
	(6)	Feeder Channels	Conditioned S-3	Regular Cast
	(7)	Feeder Bowls	Conditioned S-4	Regular Cast
Superstructure	(8)	Breastwalls	H	Regular Cast
	(9)	Shadow Walls	M	Regular Cast
	(10)	Bridge Covers	M	Regular Cast
	(11)	Feeder Entrance Covers	H	Regular Cast

Borosilicate glasses are usually melted at higher temperatures than soda-lime and other glasses. Melter refractory selection is generally the same as with other glasses. However, the higher zirconia contents of Conventional S-5 and S-4 AZS compositions provide additional ruggedness and life in exceptionally corrosive sidewall areas. Substitution of reduced void or tilt cast blocks for regular cast blocks also requires careful analysis.

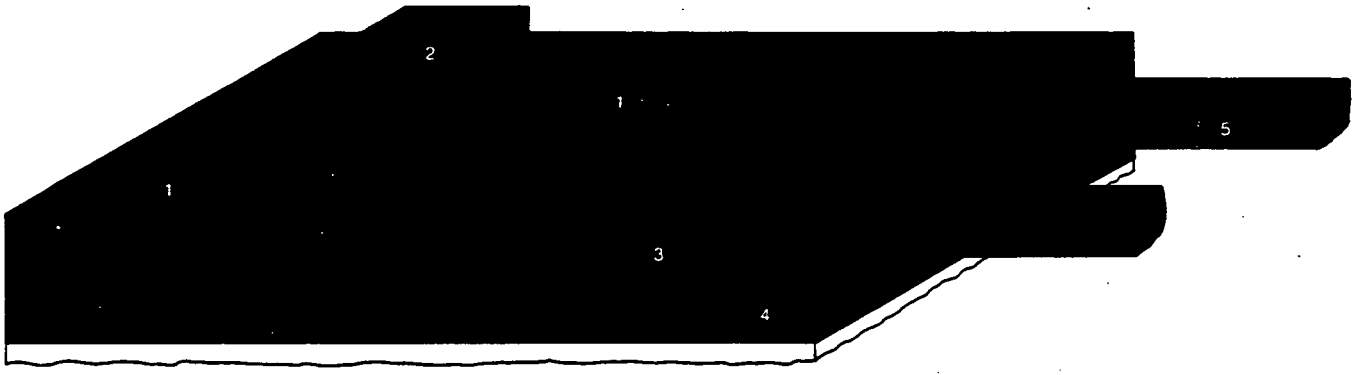
Conditioned AZS compositions are recommended throughout the refining end. These materials have less tendency to exude glassy phase. Seeds, blisters, and zirconia cords are minimized. Type M alpha-beta alumina, the world refractory standard for most refiners, is not recommended at the higher refiner temperatures associated with borosilicate glasses.

This information represents The Carborundum Company's initial recommendations for refractory selection. Because of varying and unique conditions alternative refractories may be better suited to specific conditions. Consult with our refractory representative for other alternatives.

000338

Monofrax B–Textile Fiberglass Tank

- S-3 Conditioned AZS
- S-4 Conditioned AZS
- S-5 Conditioned AZS
- H Beta Alumina
- M Alpha-Beta Alumina
- K-3 Chrome-Alumina
- E Chrome Spinel
- Monofrax B



	Furnace Area	Recommended Monofrax Refractory Type	Recommended Casting Technique
Melter	① Sidewalls	B	EPIC-3
	② Doghouse Sidewalls	B	EPIC-3
	③ Bubblers	B	EPIC-3
	④ Paving	B	EPIC-3
	⑤ Distributor Blocks	B	EPIC-3

Fusion cast Monofrax B high purity chrome was developed for lining furnaces that melt highly corrosive glasses and slags where the coloration potential from chrome oxide in the refractory is of no concern. The low porosity and impermeability of this very dense refractory gives Monofrax B refractories much higher corrosion resistance to textile fiberglass at and below the melt line when compared to other chrome bearing refractories.

With the capability of manufacturing Monofrax B refractories in much larger sizes, virtually all horizontal joints which have been necessary with the use of traditional slip cast chrome products can be eliminated.

The combination of higher corrosion resistance, increased block sizing along with improved resistance to thermal shock and sublimation makes Monofrax B refractories an excellent choice for textile fiberglass refractory requirements.

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Recommended Block Sizes

Maximum Recommended Sizes for Monofrax Refractory Blocks (inches*)

Monofrax fused cast refractories are manufactured within preferred dimensional guidelines. Maximum recommended dimensions are listed by thickness for each refractory type. Refractories can be produced in other-than-preferred dimensions, but at a price premium.

Block Thickness		S-3 S-4	S-3, S-4 EPIC-3 DCL Tile	S-5 EPIC-3 DCL Tile	M	H	A	K-3	K-3 EPIC-3	E EPIC-3
2	Length	18	—	—	—	—	—	—	—	—
	Width	9	—	—	—	—	—	—	—	—
3	Length	18	24	18	—	—	—	—	18	18
	Width	12	18	12	—	—	—	—	12	12
4	Length	24	24	24	18	18	18	18	18	24
	Width	12	18	15	12	12	9	12	14	14
5	Length	30	30	30	20	24	18	20	24	28
	Width	15	18	15	15	15	12	12	14	14
6	Length	36	36	36	30	36	24	24	24	30
	Width	18	18	15	18	18	15	15	15	14
7	Length	42	42	36	36	42	30	28	28	30
	Width	18	18	16	20	20	15	15	15	15
8	Length	48	42	42	42	48	30	30	30	30
	Width	20	20	18	24	24	16	16	16	15
9	Length	54	48	48	45	54	36	36	36	30
	Width	21	22	18	24	24	16	18	18	16
10	Length	66	62*	62	48	60	36	42	42	36
	Width	22	24	20	24	26	18	18	18	16
12	Length	72	66*	66*	60	60	42	48	54	48**
	Width	22	24	21	28	30	18	18	18	18**

*Maximum length of EPIC blocks: 72in. (1800mm)

**Maximum volume of Monofrax E refractories: 6.5 ft^3 (0.184m^3).

NOTE: Any casting over 12 in. (300mm) in thickness or 9 $ft.^3$ (0.25m^3) in volume should be checked with production control.

Other Products for Glass Furnaces

Monofrax® Cements and Ramming Mortars

To complement the large and varied selection of Monofrax fused cast refractory compositions, we provide compatible laying cements and ramming mortars. These furnace construction materials seal refractory block joints against "flame stingout"/escape of furnace atmospheres and penetration of molten glasses and provide Monofrax linings with backup monolithic layers to stop hot glass and metal attack into subsequent refractory or insulation course of lower corrosion resistance.

These materials range from a high-purity, high alumina laying cement, typified by Monofrax HMS cement for use where glass purity is of prime importance, to a chrome-alumina fused cast grain monolithic ramming material such as Monofrax K-3 RAM. With exceptionally high glass resistance where chrome coloration potential can be tolerated, it is widely used as fused cast refractory paving backup layer in wool fiberglass melters and to back up fused cast refractory sidewalls before applying heavy insulation.

HMS Air set
—High alumina superstructure joint laying cement
HMS Heat set
—High alumina refiner and melter paving cement
RAM
—Air set
—Coarse high alumina port floor ramming mortar
S
—Heat set
—Zircon alumina melter paving laying cement
K
—Air or heat set
—Chrome alumina block laying cement for wool fiberglass tanks
K-3 RAM
—Heat set
—Chrome alumina coarse fused cast grain
—Sub coarse paving monolith for wool fiberglass melters
ZA-RAM
—Air set
—AZS coarse fused cast grain
—Monolithic layer under AZS melter paving

Fiberfrax® Ceramic Fiber Insulation

Fiberfrax insulation is a strong, fluffy, lightweight alumina-silica ceramic fiber. It can be used continuously at temperatures as high as 2800° F (1538° C). It will not melt below 3200° F (1760° C). Fiberfrax ceramic fiber insulation is available in more than 50 product forms including fibers, yarn, tape, cloth, board, roving, tubing, blanket, castable, vacuum-formed shapes, and new modules.

Board products are widely used as insulation for breastwalls, portnecks and melter and refiner sidewalls. High temperature board insulation is also available for 2800° F continuous use temperature.

Fiberfrax airsetting coating cements can be dipped, sprayed, brushed, and trowelled.

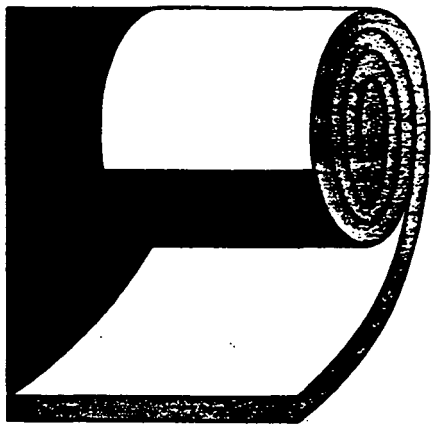
Fiberfrax insulation materials are immune to thermal shock, contain no asbestos and can be attached in a variety of ways. Density, varying with form, ranges from 4 to 30 lb/ft³ (64 to 481 kg/m³).

The thermal conductivity of Fiberfrax insulation materials is a small fraction of that of insulating firebrick.

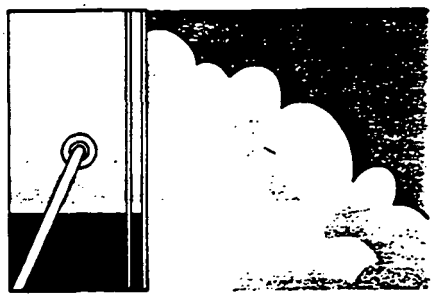
Fiberfrax products reduce heat loss by as much as 40%. Significant savings in fuel and installation costs, especially in labor and material, are realized by applying Fiberfrax insulation in many other glass tank areas such as forehearth assemblies, regenerator crowns, port necks and caps, and melter and refiner crowns. Superstructure expansion joints can be blocked off with Fiberfrax insulation during furnace heat-ups. Port neck regenerator connections and expansion joints between sidewalls and tuckstones can also be packed with Fiberfrax products.

Annealing lehrs and tempering furnaces are economically and efficiently faced or insulated with Fiberfrax products. Moist-Pack™ D blanket on hot faces withstands gas velocities of 200 ft/sec (61 m/sec).

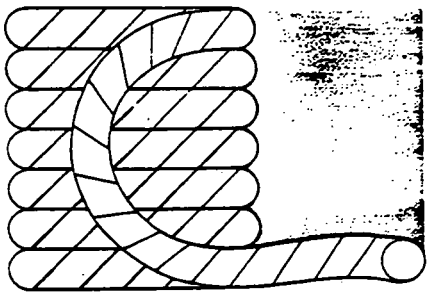
Fiberfrax insulation protects refractories against thermal shock. Paper and blanket products, cemented over burner blocks and tuckstones, minimizes the effect of thermal cycling. Fiberfrax paper placed around shapes simplifies repairs to fused cast refractories in the superstructure area. Preheating and carefully "inching" the replacement refractory into position are not required. Other applications include gasket seals for orifice rings, exit atmosphere curtains for float glass tin baths, and shielding for personnel during regenerator hot repairs. Fiberfrax insulation can also be used when batch feeders are positioned following tank heat-ups.



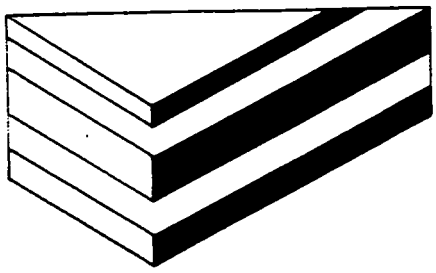
Blanket Applications
—between water cooled feeder sills and doghouse blocks
—for worker protection when feeders are installed after heat-up and during hot repairs
—for port neck caps and regenerator crowns
—around hot repair blocks to prevent thermal shock
—over silica crowns with aluminum or stainless steel covers
—as the top cover for crown insulation
—as a facing in lehr linings



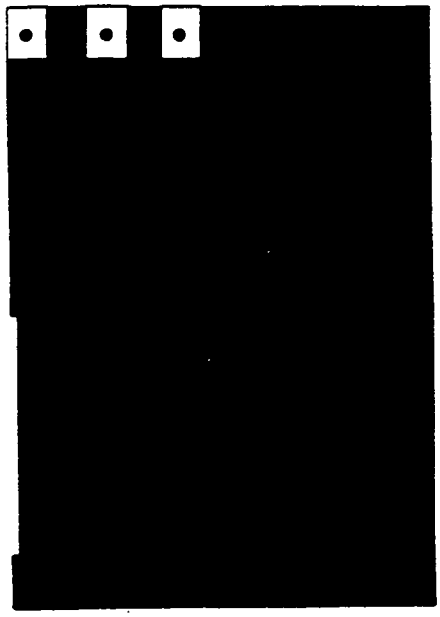
Bulk fiber Applications
—in expansion joints
—around feeder bowls
—in tin bath back-up



Rope Applications
—in expansion joints between sidewalls and tuckstones and plate blocks
—around burner blocks for easy change and improved thermal shock resistance
—as gaskets between bowl and orifice rings



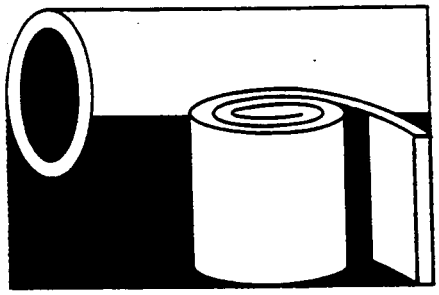
Board Applications
—for melter walls
—around burner blocks for easy change and improved thermal shock resistance
—against port neck sidewalls
—against steel
—behind melter and refiner breastwalls, backwalls and frontwalls
— as feeder channel back-up insulation for better heat balance
—for refiner walls
—between tank bottom refractory and steel



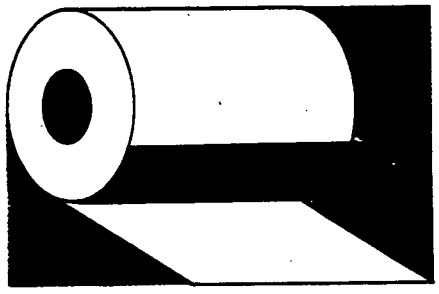
Globar® Three-Phase Heating Elements
The most important glass industry application for Globar® silicon carbide electric heating elements is temperature control of float glass tin bath sections. Corrosion resistant, three-phase CR III elements are a significant improvement over conventional three-phase units. CR III has a more stable hot zone with superior crystallization, a finer-grained, denser structure than the heating zones of a conventional three-phase element. Hot-to-cold ratio is a minimum of 15-to-1 and no heat is generated in the crossbar.

In one major float tank, CR III showed only one-third of the electrical resistance increase of conventional elements over three years. Element life is substantially greater than in conventional pieces, minimizing unplanned shutdowns.

The premium CR III element was developed for the corrosive tin bath entrance and reheat zones which have large power demand. Our experience with this premium element shows a cost-versus-service performance which allows float tank operators to set new standards for an entire tin bath heating zone.



Textile Applications
—as cloth curtains in annealing lehrs
—as atmosphere curtains in the tin bath exit
—as sleeving on TV annealing racks
—for personnel protection during hot repairs
—as tape on pusher bars, star wheels and pushouts replacing asbestos
—as tape for runner covers in tubing lehrs to replace asbestos



Paper Applications
—glued to tuckstone bottoms
—around burner blocks for easy change and improved thermal shock resistance
—against steel
—around hot repair blocks to prevent thermal shock
—for gaskets between bowl and orifice rings
—between electrode coolers and refractory blocks

A Monofrax refractory system, tailored to specific tank operating conditions, may mean appreciably longer campaigns, lower fuel and repair costs, and a higher percentage of acceptable ware. We will send a refractory expert to discuss and study your problems. Write or call:

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Fused Cast Plant
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Falconer, New York 14733
Telephone: 716-483-7200
Telex: 91-6403
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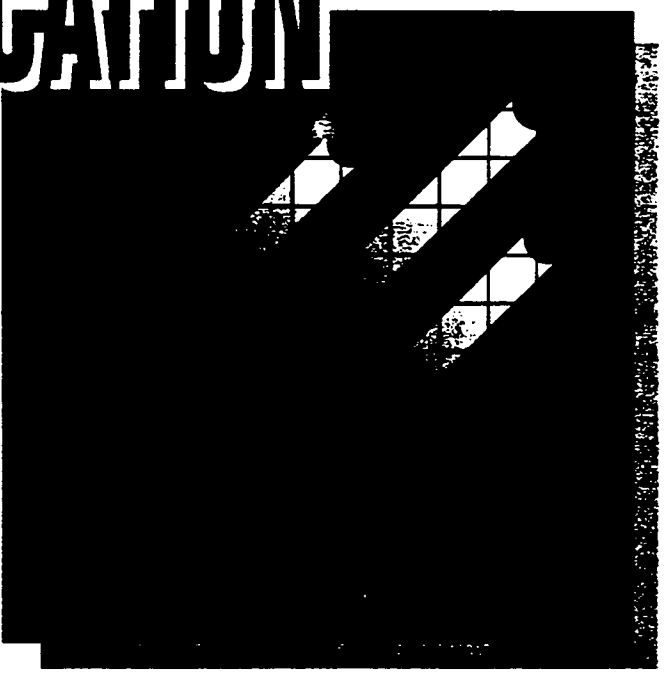
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REFERENCE 11

MONOFRAX® APPLICATION BRIEF



Refractory Performance In Oxy-Fuel Glass Tanks

GRP
MONOFRAX PRODUCTS SINCE 1931

000345

ONGOING INVESTIGATION OF OXY-FUEL FIRING IMPACT ON CORROSION OF NON-GLASS CONTACT REFRACTORIES (PART 2)

A Gupta. SM Winder (Monofrax Refractories)

ABSTRACT

The use of Oxy-fuel combustion in place of Air-fuel has led the glass industry to search for more corrosion resistant refractories for glass melting furnace crown application. A laboratory test facility was developed to compare performance of refractories, in corrosive atmospheres generated by glass melts under Air-fuel and Oxy-fuel combustion. Generic glass compositions, considered representative of Container, Fiber and TV glasses, were selected for study.

A range of fused cast and bonded refractories were subjected to vapor phase corrosion under Oxy-fuel and Air-fuel atmospheres in the test facility. Micro-analysis was used in order to determine diffusion depths and corrosion mechanisms in selected refractories and results were compared with data from thermodynamic modeling and phase diagrams.

Thermodynamic calculations and laboratory corrosion testing indicate that pressed and fired Silica and Mullite refractories are susceptible to enhanced corrosion under Oxy-fuel combustion environments. Fused cast alumina refractories showed excellent resistance to vapor corrosion in Soda-Lime Container glass. Fused cast alpha-beta Alumina is recommended as a candidate for crown refractory application.

The 'acceleration' of corrosion in a conventional crucible vapor corrosion test can lead to incorrect material selection.

1. INTRODUCTION

The benefits of conversion from an Air-fuel combustion system to an Oxy-fuel system have been well documented in numerous articles. Depending on the driving force for conversion, these benefits include reduced NOx and SOx emissions and particulate carryover, as well as an improvement in glass quality and higher throughput.

To date, over 60 major (>20 tons/day) furnaces have been converted to Oxy-fuel combustion technology. While most of the conversions have previously taken place in the Specialty glass and Fiber glass segments, Container Glass conversions are currently increasing[1].

Silica refractory has historically been the most widely applied material in furnace crown construction, under Air-fuel combustion conditions. Preliminary reports from Oxy-fuel conversions cite increased occurrence of problematic phenomena, including ratholing and liquid phase rundown. The effect of this exacerbated corrosion under Oxy-fuel combustion has led to increased maintenance activity and, in some cases, downtime. Although many glass companies have selected other refractories for crown application, including Mullite, Zircon, and fused cast AZS and Alumina, the selection criteria in general has lacked a scientific basis.

Obviously, as Oxy-fuel glass melting is gaining momentum, the selection of refractories for crown and superstructure has become an increasingly important issue. Monofrax Refractories and Air-Products and Chemicals, Inc. have initiated a joint refractories research program to address the refractory selection issue. It is intended to examine the corrosion mechanisms of a wide variety of fused cast and bonded refractories under Air-fuel and Oxy-fuel firing conditions.

2. MONOFRAX REFRACTORIES- AIR PRODUCTS OXY-FUEL PROGRAM

The scope of the program is to consider interaction of vapor phase components generated over glasses from 3 glass industry segments (currently active in converting to Oxy-fuel), with a range of refractories. This program has 3 major components:

- Thermodynamic modeling and application of phase diagrams.
- Refractories corrosion testing in a laboratory furnace.
- Refractories corrosion testing in 'field' Oxy-fuel glass manufacturing furnaces.

Thermodynamic modeling has been applied to simulate the ideal equilibrium conditions that exist under Air-fuel and Oxy-fuel combustion. As well as stoichiometric combustion, the effect of excess Oxygen and addition of Air (usually by leakage and cooling) were considered. Output was expressed in terms of the concentration ratios of the predominant vapor species under Oxy-fuel and Air-fuel conditions. This approach has proven useful in understanding the differences between these environments, in terms of chemistry and concentration of vapor phase species. In order to allow a qualitative performance comparison and relative ranking of the refractories under these environments, phase diagrams were applied in determining expected interactions with the predominant vapor species.

Laboratory testing has been utilized to investigate corrosion of a range of **Pressed and Fired** and **Fused Cast refractories** by vapor species generated above the following glass compositions:

- Soda-Lime-Silica Container glass,
- Borosilicate Fiber glass,
- Lead-Silicate 'Crystal-Glassware' and TV funnel glasses.

Refractory compositions tested to date include Silica, Mullite, Alumina, Zircon, and AZS. In addition, testing of Chrome-based refractories under Borosilicate glass vapors will be the subject of further investigation. Data collected during this laboratory testing represents refractory corrosion occurring (at a natural rate) on a timescale of a few days.

Longer-term data, from natural rate testing over weeks and months, is currently being collected from refractory samples placed in field customers glass production furnaces. This data collected from the real world, non-ideal environment - typical of glass melting - will be used as a validation check on the laboratory generated data.

3. EXPERIMENTAL PROCEDURE

Natural rate vapor phase corrosion from glass industry cullet was investigated under Oxy-fuel and Air-fuel environments in the laboratory facility. Alkali enriched glass compositions, as applied by the glass industry to 'accelerate' corrosion, were also studied in separate trials.

3.1 Oxy-fuel / Air-fuel Laboratory Facility

A schematic of the facility is shown in Fig. 1. Stoichiometric Oxy-fuel combustion of Methane gas is achieved via the reaction:-

$$CH_4 + 2O_2 \rightarrow CO_2 + 2H_2O$$

Air-fuel combustion conditions are obtained by addition of the correct fraction of N_2 gas to this reaction. A stoichiometric flame (Oxy-fuel or Air-fuel) is thus obtained in the combustion chamber by mass flow control of Methane, Oxygen and Nitrogen gases. The CO_2 and H_2O combustion products pass into a refractory reaction vessel via a ceramic tube. This vessel houses a glass bath over which the refractory test samples are placed. Combustion gases react with the molten glass bath surface and generate vapor phase products which then interact

with the refractory samples. Vessel pressure is maintained at approximately ambient pressure by exit of the gases through a flue.

As well as hosting either Oxy-fuel or Air-fuel modes of combustion, the facility is also capable of simulating various environmental parameters, as considered typical for different types of glass compositions and furnace designs. Typical values of parameters such as glass to refractory surface area ratio, combustion volume to glass surface area ratio, gas flow rates, and melting temperature were provided by the glass melting industry and used in laboratory trials.

Corrosion Experiments

A **Soda-Lime-Silica glass** composition considered typical of the Flint Container industry was used in corrosion experiments. The experiments were performed at 1510°C for durations of 50h and 100h under both Oxy-fuel and Air-fuel combustion atmospheres.

A **Lead 'Crystal' Glassware** composition and a **TV Funnel glass** composition were subjected to Oxyfuel combustion at 1450°C and 1510°C respectively, for 50h.

Following reaction, all samples were photographed and subsequently prepared for micro-analytical investigation. The results are discussed in Section 4.

3.2 Alkali Enriched Vapor Corrosion Experiments

The glass industry generally utilizes the ASTM 'crucible test' in order to assess vapor phase corrosion of refractories. 'Accelerated corrosion' is achieved in this test by use of batch components that are known to be volatile, without addition of the non-volatile components. Some glass producers prefer to 'accelerate corrosion' by addition of (~2.5X) excess volatile components to normal glass cullet. In such tests, the test refractory sample is placed above the crucible, as shown in Fig. 2. This test is usually performed in an electrically heated furnace under ambient atmosphere in the absence of gaseous combustion products.

The glass industry has recently applied such 'accelerated' tests, in the absence of controlled H_2O vapor content, to the selection of refractories for Oxy-fuel furnace crown application. This has correctly resulted in the selection of fused-cast Alumina refractory. However, the optimum choice between an αβ–Alumina (Monofrax M) or β–Alumina (Monofrax H) depends upon the glass melt composition and application temperature. In particular, alkali content of the glass is a deciding factor. Under Oxy-fuel combustion (high P_{H_2O}), the major species present are NaOH over Soda-Lime melts, PbO over Lead-Silicate melts, and HBO over Borosilicate melts. An 'accelerated' Oxy-fuel test actively increases the concentration of these species, shifting αβ phase stability fields. An 'accelerated' ASTM type test (uncontrolled P_{H_2O}) would lead to uncontrolled formation of a range of different volatile alkali species, at undefined concentrations. This may prevent correctly optimized selection between α–Alumina and αβ–Alumina products.

In order to demonstrate this effect, a corrosion experiment was performed on both types of fused cast Alumina refractories. Cullet of the typical flint container composition previously studied, was enriched with Na_2CO_3 to produce a series of 6 glasses. Glass frits containing equivalent soda ratios of 1.0, 1.3, 1.6, 1.8, 2.0, and 2.2X that of the base glass were prepared and pre-melted. Use of this homogeneous pre-melted glass product avoided any frothing associated with decomposition of carbonates, during the subsequent corrosion test.

A refractory muffle was especially built to provide six separate compartments to hold the six different glasses in Pt crucibles, see Fig. 3. Samples of Monofrax M and H refractories were placed above each crucible. The test was conducted

under Oxy-fuel conditions at 1470°C for a short duration of 2h. Samples were studied in the SEM to ascertain the stable (α or β) Alumina phase nucleating new growth on the exposed surfaces.

Thermodynamic calculations suggest that Alumina refractories should be present in the phase when exposed to Container glass and modified glass with soda ratios to ~2 (~26wt% Na_2O). At ratios above ~2 the preferred phase is β.

4. RESULTS AND DISCUSSION

The results of both natural corrosion rate testing under both Air-fuel and Oxy-fuel conditions, and 'accelerated' corrosion rate testing under Oxy-fuel conditions, are presented and discussed in this section.

4.1 Thermodynamic Modeling and Application Of Phase Diagrams

Phase equilibria studies for interaction of various refractories with vapors generated above the different glass melts are considered:

Flint Container Glass

Output from the thermodynamic modeling may be summarized as follows:

- NaOH is the predominant vapor phase species for both Air-fuel and Oxy-fuel.
- The Oxy-fuel : Air-fuel NaOH concentration ratio is ~2.
- NaOH concentration increases with increasing temperature.

Since creation of H_2O during both Air-fuel and Oxy-fuel combustion leads to NaOH formation, any testing procedure that does not control H_2O concentration in the environment will not relate to testing under true combustion conditions.

Increased NaOH levels under Oxy-fuel combustion may be expected to enhance corrosion of refractories. In particular, Silica - or any refractory containing a Silica rich phase (Mullite, AZS etc.) - are expected to corrode at an enhanced rate. The in-flux of Na^+ and OH^- to these refractories actually alters their structure, with increased formation of lower viscosity liquids. Thus, simple analysis based upon Ficks Law (assuming constant diffusion coefficients to describe behavior) is no longer possible.

In the presence of a temperature gradient through the refractory crown thickness, condensation of NaOH in open joints is expected to occur. Melting at 1510°C under equilibrium saturation conditions, ~100°C temperature drop should cause ~50% of the vapor phase to condense onto a refractory joint surface. The absolute amount of reactive condensation is approximately doubled under Oxy-fuel conditions. This behavior is a basic mechanism that leads to 'ratholing' type of joint corrosion, which should therefore be exacerbated under Oxy-fuel conditions.

Liquid phase formation in Silica refractory, and liquid phase with concurrent Alumina precipitation in Mullite refractory, are predicted from the phase diagrams. Thermodynamic calculations suggest that Alumina is stable in the Flint Container Oxy-fuel environment.
Thus, thermodynamically, Alumina refractories are expected to outperform Silica and Mullite in a corrosive Flint Container Oxy-fuel furnace.

Alkali Borosilicate Wool Fiber Glass

In this type of glass one might expect to find Na and B species in the vapor phase. However, thermodynamic modeling predicts HBO to be by far the predominant species. Furthermore, the equilibrium concentration of HBO under Oxy-fuel was found to be approximately 3.4 times its concentration under Air-fuel. This ratio is almost equal to the ideal ratio of gas volumes under Oxy-fuel and Air-fuel combustion (3.5), suggesting that there is a direct concentration effect on this vapor phase species under Oxy-fuel.

Consultation of the phase diagram suggests that Mullite should exhibit superior corrosion resistance to Borosilicate glass vapors compared with Silica. However, Alumina refractories again demonstrate improved intrinsic thermodynamic stability.

TV Funnel Glass

Thermodynamic modeling of 24% PbO TV funnel glass predicts PbO to be the predominant vapor phase species, followed by KOH, Pb and NaOH in order of decreasing concentration. These modeling results are in accord with observations from the TV funnel glass industry and the laboratory test facility, regarding formation of metallic lead and diffusion of K^+ into refractories.

The modeling also predicts PbO vapor concentration to be approximately 2.5x higher in Oxy-fuel than its concentration in Air-fuel conditions. The predicted concentration of PbO increases with increasing temperature.

In addition to modeling under stoichiometric conditions, modeling was also performed under simulated excess Oxygen and excess Air atmosphere. Essentially, the concentration of PbO was found to increase slightly with total excess Oxygen levels up to ~1%. The increase in PbO occurs by oxidation of metallic Pb whose concentration is therefore reduced in the environment. The rate of increase in PbO concentration was found to decrease above ~1% excess Oxygen.

TV Panel Glass

Behavior of a Lead-free TV Panel glass composition was modeled. KOH vapor concentrations were found to exceed NaOH, and amounts of both species increased with temperature. The amount of KOH under Oxy-fuel conditions was found to be ~2X the concentration in Air-fuel.

4.2 Micro-Analytical Investigation

Pressed & Fired Silica and Mullite, and fused cast CS3 AZS (34% ZrO_2), Monofrax M ($\alpha\beta$–Al_2O_3) and Monofrax H (β–Al_2O_3) refractories were selected for micro-analytical investigation. Fig. 4 and Fig. 5 are photographs of samples tested for 50h and 100h respectively. A considerable amount of corrosion can be seen on the Silica sample under Oxy-fuel conditions.

Silica

The microstructure of virgin Silica refractory contains a distribution of large polycrystalline Silica grain agglomerates, surrounded by fine grained Silica. A Silicate boundary phase containing CaO, Al_2O_3, and Fe_2O_3, is also present, along with ~24% porosity. Exposure of this refractory surface to NaOH from the glass melting environment results in dissolution of fine grained Silica particles. The resulting low viscosity Sodium Silicate liquid is then absorbed within the porous regions in the refractory due to capillary forces. This preferential dissolution of fine grained silica particles leaves behind the more slowly dissolving coarse agglomerates on the surface, - well known in the glass melting industry as 'frosting'.

This sodium silicate liquid was observed to depths of ~15mm after only 100h of exposure, using SEM/EDS. However, grain boundary analysis using SIMS proved that in-diffusion of Na^+ had occurred to considerably greater distances. Furthermore, the Na_2O concentration at refractory grain boundaries was found to be ~1.5X higher under Oxy-fuel than under air-fuel conditions. At longer exposure times, the refractory is expected to saturate with liquid, causing run down into the host glass.

It is clear from the results that Silica refractory is subjected to significantly accelerated corrosion under Oxy-fuel

000350

conditions in the laboratory furnace. Both exacerbated ratholing and accelerated corrosion at the hot face are expected in application. This leads to the conclusion that Silica material does not represent the optimum choice for extended refractory service life.

Mullite

The microstructure of virgin Mullite refractory consists of a distribution of large polycrystalline Mullite agglomerates surrounded by fine grained Mullite bond, with ~15% apparent porosity. Exposure of this refractory surface to NaOH from the glass melting environment results in liquid formation due to dissolution of fine grained Mullite particles. The resulting Sodium Aluminosilicate liquid also contains precipitated Alumina laths. This refractory represents an improvement over Silica, due to the lower volume of more viscous liquid formed, and the decreased kinetics of formation dictated by its lower porosity. Although Mullite appears to perform slightly better than Silica in short term corrosion tests, it is thermodynamically unstable against NaOH, KOH etc. in Oxy-fuel environments, and also does not represent the optimum choice for extended refractory service life.

Fused Cast AZS (34% ZrO_2)

The microstructure of Fused cast AZS consists of three phases:

- Primary ZrO_2 dendrites,
- Al_2O_3 plates containing co-precipitated ZrO_2,
- Sodium aluminosilicate glass phase, ~20wt% (~35vol%).
- Apparent porosity, ~1%.

Under Air-fuel and Oxy-fuel corrosion conditions, AZS refractory shows evidence of concurrent exudation (of the in-situ glass phase) and corrosion, in the short term. The presence of alkali vapor species leads to dissolution of the Alumina phase at exposure temperatures. This phase exceeds saturation concentration in the liquid, and is crystallized upon subsequent cooling - leading to formation of an opaque glassy surface deposit. In contrast, exudation of AZS refractories, performed under ASTM specified procedure (absence of corroding species), results in a clear, transparent glass on the surface of the specimen.

AZS samples exposed to Oxy-fuel glass melting atmospheres exhibit more corrosion than material exposed to Air-fuel. Interference due to the occurrence of short-term exudation requires that longer term corrosion in the "field" will be necessary to quantify the difference. This product is considerably less affected than the porous Silica and Mullite refractories.

Fused Cast Alumina Refractories

Two types of refractory are currently commercially available, and are recommended for use in areas where batch carryover, and subsequent contamination by silicate, is not an issue:

Monofrax M (αβ Alumina)

The microstructure of Monofrax M consists of ~45% α Alumina and ~55% β Alumina phases, distributed randomly. Boundary phases containing Si, Na, Ca, and Al are also present at ~1%. There is ~2% apparent porosity in this refractory.

Micro-analytical investigation showed presence of a layer of (Alumina at the surface, after exposure to Oxy-fuel and Air-fuel glass melting. The α layer seen at the surface of corroded samples was observed to contain finely dispersed porosity. This occurs during formation of the α layer, due to the loss of NaOH from the (alumina grains, and is accompanied by ~17% volume reduction resulting in formation of the porosity.

Monofrax H (β Alumina)

This refractory consists essentially of large β alumina grains with negligible grain boundary phase and ~4% apparent

porosity. As for Monofrax M, exposure of this refractory to Air-fuel or Oxy-fuel glass melting also caused generation of an α Alumina surface layer and associated porosity.

Monofrax M vs Monofrax H Comparison
The surfaces of both Alumina refractories were converted to an α alumina layer. The thickness of this layer was found to increase linearly with short exposure times (up to 100h). Considerable experience in application of these refractories in glass melting furnaces suggests that this effect passivates over the long term. This layer represents a stable phase in application and as such is not considered to be a problem. However, since the αβ Alumina refractory undergoes less transformation creating less new porosity, this is considered the optimum choice for above-glass application in most (low alkali) glass melting environments. Longer term field testing under Oxyfuel conditions is currently being performed.

4.3 Alkali Enriched Vapor Corrosion Experiments

Monofrax M and H samples from each of the six crucibles (described in Section 3.2) were analyzed using SEM/EDS to determine the relative stability of α and β alumina phases. Analytical observations are summarized as follows:

- α Alumina is the stable phase when Monofrax M and H are exposed to glass melts containing up to ~ 24-26wt% Na_2O (temperature dependent).
- β Alumina is the stable phase when Monofrax M and H are exposed to glass melts containing more than ~26wt% Na_2O.

These empirical results are in good agreement with thermodynamic calculations. Furthermore, it is clear that use of an 'accelerated' test which increases alkali vapor species above normal levels may lead to a shift in the equilibrium Alumina phase present during testing. In other words, Alumina may be artificially stabilized during 'accelerated' testing, when under normal operating conditions (at lower alkali levels) the phase is stable. Fused cast Alumina refractory selection should be based on the amount of volatile alkali in the melting environment, and this is directly dependent on the glass alkali content and the expected melting temperature.

5. CONCLUSIONS

- Thermodynamic calculations and results of laboratory testing indicate that Oxy-fuel combustion causes an increased concentration of volatile corrosive species above glass melts. This leads to enhanced corrosion of refractories, compared to Air-fuel combustion.

- Silica refractories, and other materials with a major Silica component, are especially vulnerable to exacerbated corrosive attack, due to liquid phase formation. Alumina based refractories are more thermodynamically stable, exhibiting innocuous transformation from β to α phase under most glass melting conditions. Monofrax M αβ Alumina is considered the preferred material for most glass melting environments.

- Pressed and fired refractories exhibit rapid reaction due to their high porosity content. Dense fused cast materials show superior corrosion resistance.

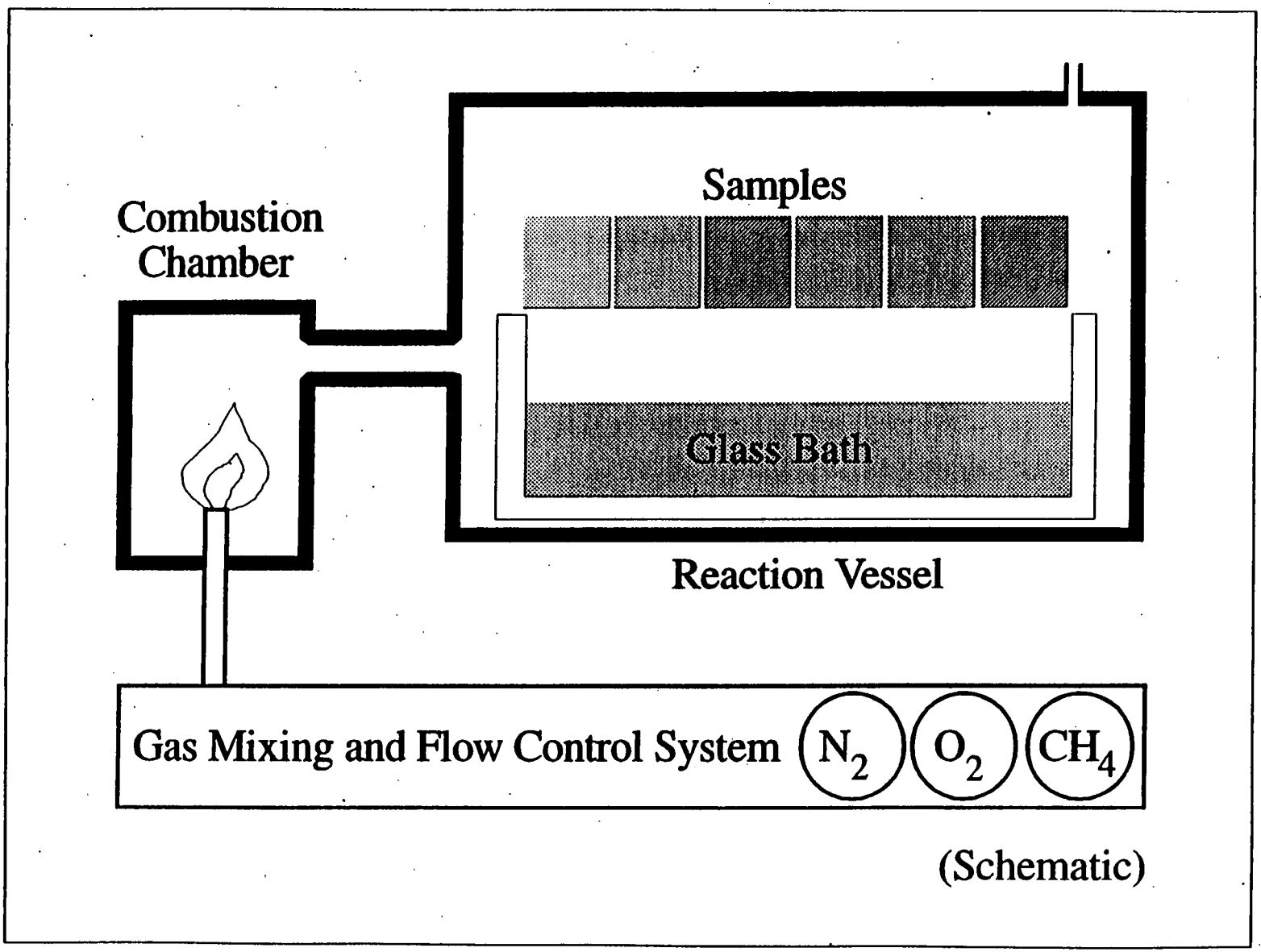
- 'Accelerated' vapor corrosion testing for refractory selection can lead to incorrect material selection.

Acknowledgment
The valuable input of S Gould, KR Selkregg and JH Gibson is appreciated.

References

[1]. Eleazer P.B., Slavejkov A.G., "Clean Firing of Glass Furnaces Through The Use of Oxygen", Proc. 54th Glass Problems Conference, (1994).

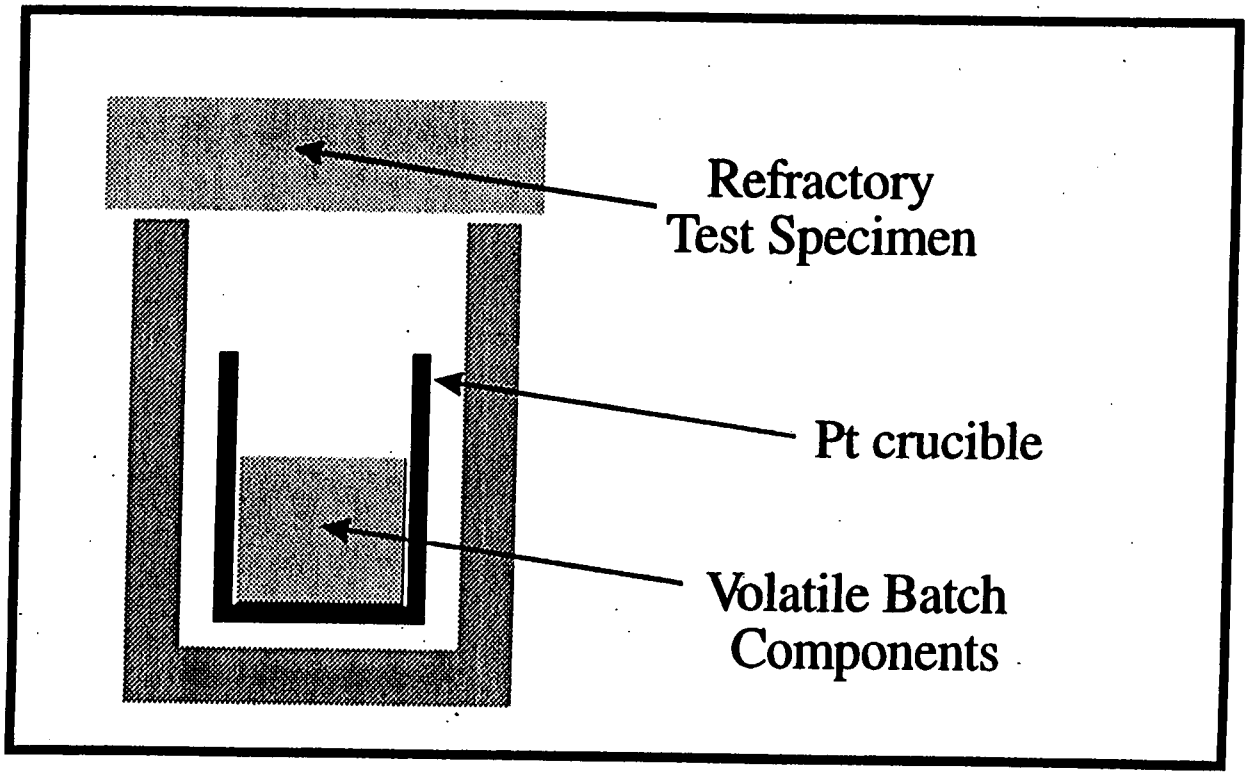
Figure 1. Oxy-Fuel/Air-Fuel Laboratory Test Furnace



000353
Reference 11-9

Figure 2. Crucible Test For Vapor Phase Corrosion

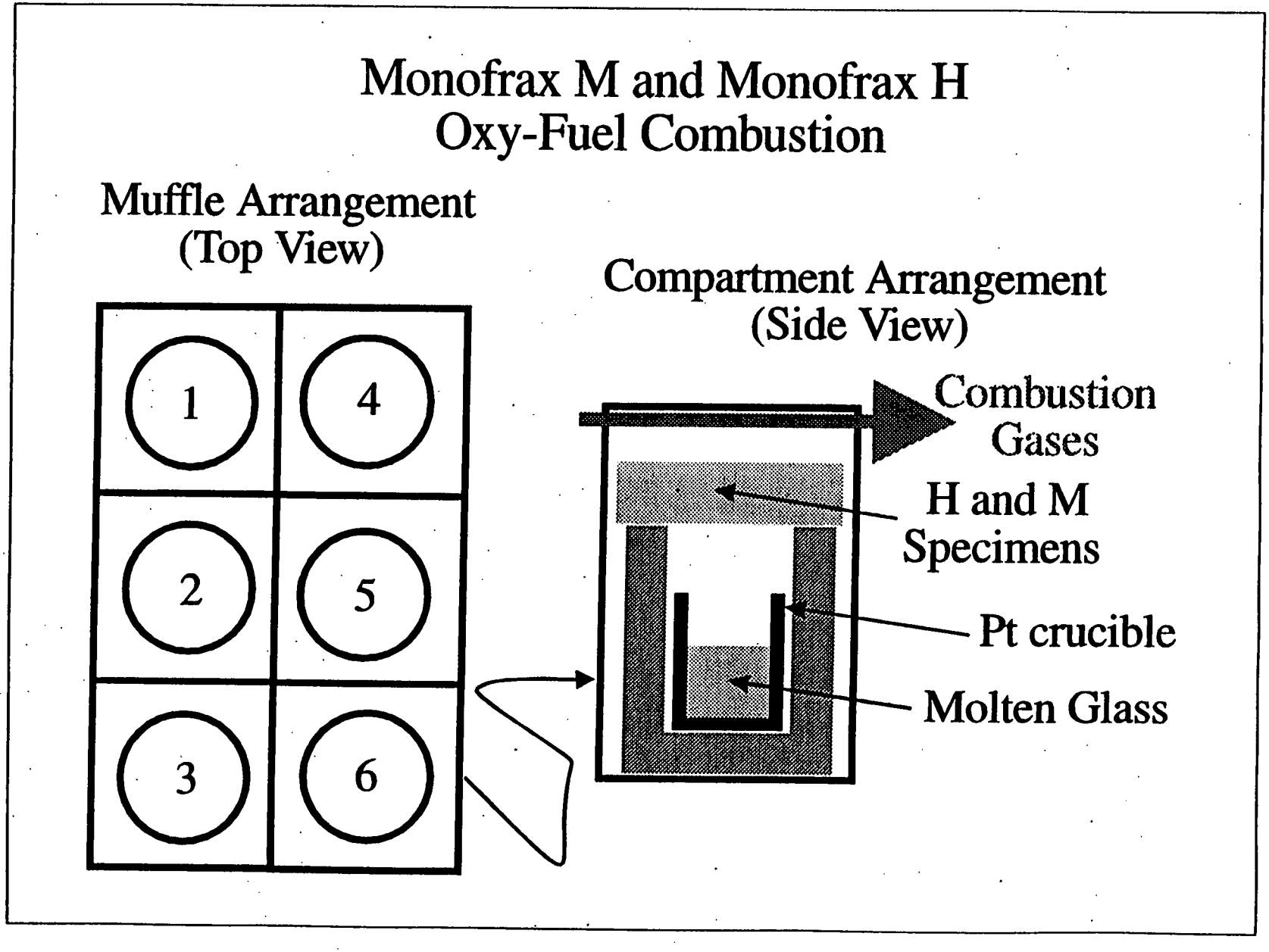
General Procedure Within Glass Industry
Includes 'Acceleration' by Increasing Volatile Components



Electrically Heated Muffle Furnace
Does Not Include Combustion Products
Interaction with H2O Not Simulated

000354

Figure 3. Alpha-Beta Alumina Stability



000355

209

Figure 4(a).

Silica, Monofrax M. CS3 AZS, and Monofrax H samples after 50h test in Flint Container Glass under Oxy-fuel and Air-fuel conditions.

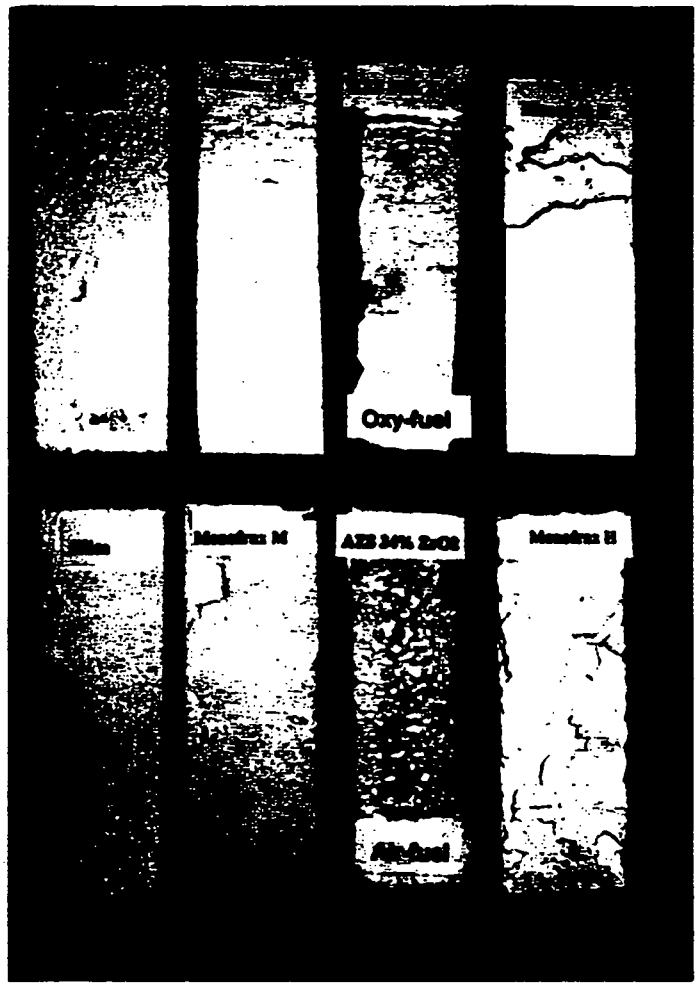
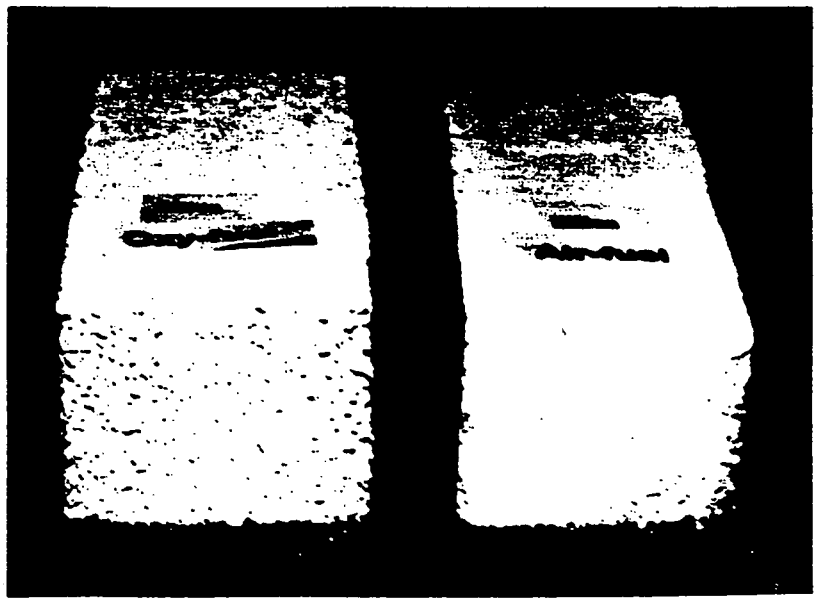


Figure 4(b).

Close up of Silica Sample after 50h test Flint Container Glass under Oxy-fuel and Air-fuel.



Reference 11-12

Figure 5(a).

Silica, Monofrax M, CS3 AZS, and Monofrax H samples after 100h test in Flint Container Glass under Oxy-fuel and Air-fuel conditions.

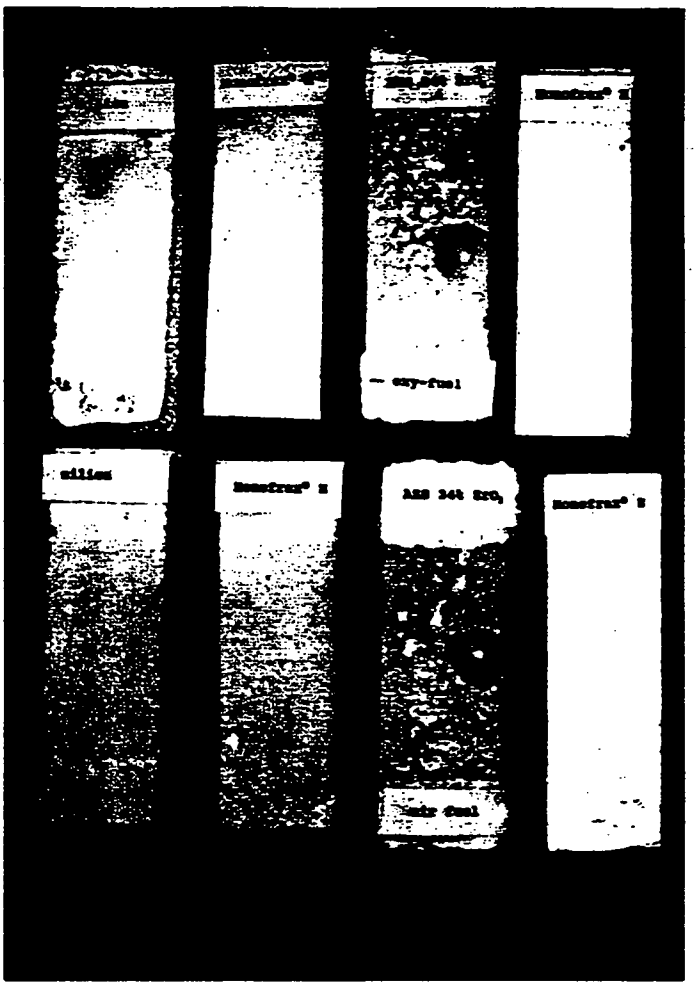
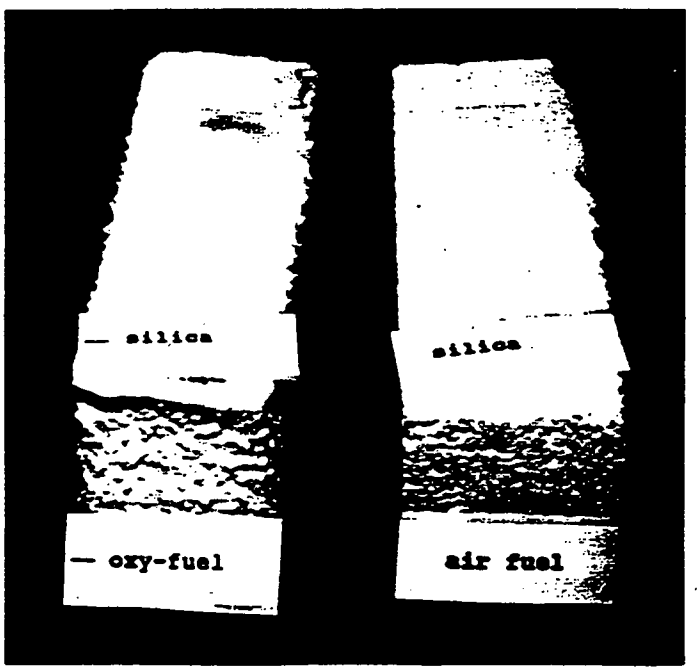


Figure 5(b).

Close up of Silica Sample after 100h test Flint Container Glass under Oxy-fuel and Air-fuel.



000357

Reference 11-13

MONOFRAX® PRODUCTS SINCE 1931

GRP Inc.
1870 New York Avenue
Falconer, New York 14733-1797 USA
Telephone 716 483-7200
Telefax 716 665-2478

Form A-3069
Effective 9-96
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Monofrax is a registered trademark of GRP Inc., a wholly owned subsidiary of the Carborundum Corporation.

000338

Reference 11-14

REFERENCE 12

Fernald Environmental
FERMCO
Restoration Management Corporation P. O. Box 538704 Cincinnati, Ohio 45253-8704 (513) 648-3000

September 12, 1996

Fernald Environmental Management Project
Letter No. C:WMTSP(SP):96-0010

Mr. Emory Toomey
GTS Duratek
8955 Guilford Road, Suite 200
Columbia, MD 21046

Dear Mr. Toomey:

DURAMELTER 1000 HT, ACCEPTANCE ISSUES

References: 1) Letter No. C:P:(FRVP):96-0080, Dennis Nixon to Emory Toomey, "Duramelter 1000 HT, Additional Acceptance Issues," dated August 9, 1996.

2) Letter No. C:P:(FRVP):96-0067, Dennis Nixon to Emory Toomey, "Duramelter 1000HT, Acceptance Issues," dated July 5, 1996.

3) Letter No. C:P:(FRVP):96-0063, Dennis A. Nixon to Emory Toomey, "Duramelter 1000HT, Side Chamber Feeder," dated June 11, 1996.

In addition to the three referenced letters regarding issues related to the acceptability and/or operability of the Duramelter 1000 HT, FERMCO has identified a number of issues prior to and during the 36-hour Acceptance Test.

These issues have been discussed in depth during field activities and are now being formally documented as follows:

- Discharge Chamber - The refractory, as previously documented, is cracked and the problem has become worse during high temperature testing (1350°C). This has not allowed FERMCO to perform the 8-hour Gem Machine Acceptance Test as prescribed in the Campaign #2 Pilot Plant System Operability Test (SOT) Procedure . FERMCO is awaiting further guidance on repair/replacement of this component.

- Melter Feed Pump - The Moyno feed pump has failed causing material spills and damaged pump components. The pump has been rebuilt and/or replaced on a number of occasions. FERMCO questions the application of this pump for the required services.

- Lid Heaters - The melter lid heaters are becoming a maintenance issue with multiple failures (4). FERMCO is concerned that this may become a continued problem for melter operation. Please advise as to potential improvements for extended heater life.

- Air Bubbler - The Number 1 air bubbler in the bottom of the melter has receded into the refractory and is no longer functional. The air bubblers provide critical agitation to the melt pool. FERMCO requests a recommended repair for this component.

- Bottom Drains - As previously identified, the bottom drains have failed and have been subsequently replaced. While replacing the drains, the inner tubing and standoff were removed. The 304SS tubing displayed rust and magnetic field attraction which are inconsistent with 304SS. A Nonconformance Report has been prepared (Attachment 1) requiring GTS-Duratek explanation and proposed disposition. This finding provides further concern as to the quality of workmanship utilized on the failed bottom drain assemblies.

- Electrode Chamber Foaming - On September 10, 1996, the FERMCO Operations and technical staff observed foaming in the west electrode chamber of the melter. This is a serious concern in regard to the integrity of the "E-Wall." Analysis of the electrode chamber glass is currently being performed to provide further insight into the concern.

The 36-Hour Acceptance Test was complete on September 7, 1996 in accordance with the Pilot Plant Systems Operability Test Procedure, Glass Campaign 2. However, contractual acceptance is contingent upon GTS-Duratek adequately addressing the exceptions identified in this letter as well as the referenced correspondence. Please find attached a copy of the SOT for Campaign #2, 36-hour Acceptance Test (Attachment 2).

As before, please respond on these items with a path forward no later than September 18, 1996, in order to expedite closure of the melter acceptance.

If you have any questions, please contact me at (513) 648-4800.

Sincerely,

Dennis A. Nixon
Deputy Pilot Plant Project Manager

Fernald Environmental
FERMCO
Restoration Management Corporation

DAN:kdg
Attachments

c: N. Akgunduz, DOE, MS45
J. Bradburne, MS17
C. Coker, GTS Duratek
M. Connors, MS52-4
C. Griffin, MS52-4
R. Heck, MS52-5
B. May, MS52-4
R. Norton, MS52-5
D. Paine, MS52-4
K. Solomon, MS52-4
C. White, DOE, MS45
File Record Storage Copy 120

ATTACHMENT 1

000363

Reference 12-4

Side 1 of 2

Nonconformance Report

Revision: 0

(Preparer Completes Blocks 2 through 13)

1. NONCONFORMANCE REPORT NUMBER: (This number is assigned by The P/QA Nonconformance Administrator.)	DR 96-0082
2. TYPE OF NONCONFORMANCE REPORT: Check *OBSERVATION* if the identified condition has little or no impact on the quality of an item, the quality of work, or the reliability of documentation.. An observation, if resolved, could lead to excellence in operations. Check *FINDING* if the identified condition represents a procedural or program deviation which impacts the quality of work or the reliability of documentation.. Check *DEVIATION* for hardware items when the condition represents a departure from specified requirements or specifications. Check *CORRECTIVE ACTION REPORT* if the condition represents trended deviations from specified requirements, a programmatic breakdown, or a Significant Condition Adverse to Quality.	OBSERVATION [ ] FINDING [ ] DEVIATION [X] CORRECTIVE ACTION REPORT [ ]
3. DATE DISCOVERED: Enter the date the nonconformance was identified.	3. 09-04-96
4. RESPONSIBLE ORGANIZATION: Enter the Division (4a.) and Department (4b.) of the organization responsible for correcting the nonconformance. (If known)	4a. Duratek 4b. VITPP Engineering
5. RESPONSIBLE MANAGER: Enter the Division Manager (5a.) and Department Manager (5b.) of the organization responsible for correcting the nonconformance. (If known)	5a. Dennis Nixon 5b. Dennis Nixon
6. LOCATION: Identify where the nonconformance was observed.	6. VITPP Melter Bottom Drain tube
7. ASSESSMENT ACTIVITY: Enter the type and number of the assessment that was being performed when the nonconformance was identified. (e.g. Audit 195-19, QEP Number, Walk-through, etc.)	7. Repair of Bottom Drain Assembly

8. REQUIREMENTS: (Identify and Quote the requirement directly from the document (procedure, specification, drawing, etc.) that best describes the acceptance criteria for the item or activity.)

Duratek drawing CRU4-M-M-013 identifies material requirements of the bottom drain as being 304 SS.

9. NONCONFORMANCE: (Fully describe the nonconformance as it relates to the requirements. (Use supplemental sheets as required.)

The bottom drain tubing identified as 304 SS on the tubing and the standoff weld to the tubing are displaying rust, tube blockage and magnetic field attraction.

10. PREPARED BY: L. Sexton	11. Phone: 4801	12. Mail Stop: 52-4	13. Date: 09-06-96

(All Employees Send or Take the Original of this report to the P/QA Representative)

PERFORMANCE QUALITY ASSURANCE EVALUATION

14. QUALITY ASSURANCE PROGRAM REQUIREMENT: (Enter the Criterion and paragraph number of the Quality Assurance Program, (RM-0012), requirement that best describes the acceptance criteria for the item or process being assessed.)	14. 7.3.3
15. DIVISION/DEPARTMENT REQUIREMENT: (Enter the document number and paragraph of the division or department requirement that best describes the acceptance criteria for the item or process being assessed. (Drawing Number, Spec. Number, Procedure Number, as well as the Paragraph , etc.)	15. CRU4-M-M-013
16. NOTIFICATION: (Enter the date the responsible manager was notified of the nonconformance. Ensure manager agrees a nonconformance exists.)	16. 09-04-96
17. TRENDING INFORMATION: (Enter the paragraph number from the Matrix of Nonconformances (Attachment D, QA-0001) that best describes the identified nonconformance. (e.g., 1.0 F ii))	17. Cii
18. TAGGING REQUIRED: [ ] YES [X] NO / 19. P/QA REP.: L. Sexton	20. DATE: 09-04-96
21. P/QA MANAGEMENT REVIEW: V. Childress	21. DATE: 09/09/96
23. P/QA DIV. MANAGER: (CARs Only) N/A	24. DATE:
(P/QA Representative obtains the Nonconformance Report Number from the P/QA Nonconformance Administrator and enters the number in Block 1 above)	25. RESPONSIBLE MANAGER RESPOND BY: (Date) 09-20-96

ATTACHMENT 2

000365

Reference 12-6

ORIGINAL

Test Copy

607

VITRIFICATION PILOT PLANT STARTUP PROGRAM

PHASE I

TEST PROCEDURE 2504-SU-0035

PILOT PLANT SYSTEMS OPERABILITY TEST PROCEDURE

GLASS CAMPAIGN 2

Rev. 0
8-20-96

APPROVED BY:

VITPP Quality Assurance - Vic Childress	Date 8-21-96
VITPP OS&H - Doug Daniels	Date 8/20/96
VITPP Regulatory Programs - Jeff Stone	Date 8/20/96
VITPP Operations Manager - Joel Bradburne	Date 8-20-96
VITPP Manager - Dennis Nixon	Date 8/20/96
VITPP Start-Up - Rod Hiestand	Date 8/20/96
VITPP Testing/Data Manager - Rod Gimpel	Date 8/20/96
SILOS Project Engineering Manager - Mark Dehring	Date 8/20/96

Fernald Environmental Restoration Management Corporation
Fernald, Ohio

Reference 12-000366

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Attachments

Attachment 1	Signature Record Log
Attachment 2	Campaign 2 Acceptance Surrogate Batch Sheet
Attachment 3	Campaign 2 Surrogate Batch Sheet (Blank Quantities)
Attachment 4	LPM Melter Feed vs. Wt.% Solids in Melter Feed for 1 MT/D
Attachment 5	Melter Feed Pump Calibration
Attachment 6	Test Performance Parameters for SOT Campaign 2 at the VITPP
Attachment 7A	Melter Feed\Off-Gas Data Sheet
Attachment 7B	Off-Gas Pressure and Temperature Data Sheet (for North Pad)
Attachment 8	Campaign 2 Transition Glass Batch Sheet
Attachment 9	Campaign 2 1250°C Surrogate Batch Sheet
Attachment 10	Data for GTS Duratek
Attachment 10.1	Duramelter 1000-HT Acceptance Test Table
Attachment 10.2	Gem Machine Acceptance Testing Table

1.0 PURPOSE

This document defines and comprises the System Operability (SO) test procedure for Campaign 2 of Melter operations. Campaign 2 is the testing of Acceptance Glass and Series D glass which is the surrogate for a vitrified product of waste (70% K-65 material and 30% Silo 3 material) and requisite additives. This SO testing covers:

- The displacement of benign glass from the Melter.
- The 36 hour Acceptance Testing of the Melter for final acceptance by FERMCO.
- The 8 Hour Gem Machine Performance Test
- The preparation of Series D glass slurry.
- The operation of the Melter and the Gem Machine at 1 MT/D to satisfy the procurement specification.
- The adjustment of Melter temperature to correspond to Series D glass formulation.

This testing will demonstrate continuous operations with Acceptance Glass and Series D surrogate under the conditions specified in the Melter and Gem Machine specification.

Specifically, Campaign 2 consists of 2 runs with the following objectives:

Run 1 — This run will feed the Acceptance Glass formula with the Melter main chamber glass temperature maintained at 1350°C. The goal of Run 1 is to perform the 36 hour acceptance test of the Melter and the 8 hour acceptance test of the Gem Machine at a minimum rate of one metric ton per day.

Run 2 — This run will feed the Series D surrogate glass formula with the Melter main chamber glass temperature maintained at 1250°C. The goal of Run 2 is to verify the ability of the Melter and support systems to produce acceptable glass for this feed formulation.

2.0 SCOPE

This procedure applies to all personnel involved in the execution or supervision of Campaign 2 of the VITPP Melter.

Campaign 2 involves slurry feeding the Melter with Acceptance Glass and Series D surrogate waste, i.e., lead and barium will be used. The feed slurries will be mixed directly in the Slurry Tanks. The Thickener will not be used for this operation.

3.0 REFERENCES

3.1 Process Flow Diagrams

No.	Title
94X-5900-F-278	HYDRAULIC WASTE RETRIEVAL AND DEWATERING
94X-5900-F-279	FEED PREPARATION AND VITRIFICATION
94X-5900-F-280	OFF-GAS TREATMENT
94X-5900-F-551	PROCESS COOLING WATER
94X-5900-F-795	RADON REMOVAL AND EMERGENCY OFF-GAS
94X-5900-F-851	FURNACE AND GEM MACHINE

3.2 Piping and Instrumentation Diagrams

No.	Title
94X-5900-N-270	FEED PREPARATION SHEET 1 OF 2
94X-5900-N-271	FEED PREPARATION SHEET 2 OF 2
94X-5900-N-272	FURNACE AND GEM MACHINE
94X-5900-N-273	OFF-GAS TREATMENT
94X-5900-N-274	WASTEWATER TREATMENT
94X-5900-N-314	PROCESS COOLING WATER
94X-5900-N-315	OFF-GAS AIR
94X-5900-N-529	ADDITIVE TRANSFER
94X-5900-N-530	SILO 3 MATERIAL TRANSFER
94X-5900-N-820	EMERGENCY OFF-GAS SYSTEM
94X-5900-N-891	ELECTRODE HOLDERS COOLING SYSTEM
CRU4-X-I-002	MELTER
CRU4-X-I-004	UTILITY AIR
CRU4-X-I-005	COOLING WATER

000369

3.3 FERMCO Procedures

In addition to all of the applicable FERMCO Operations Procedures, and Health and Safety procedures, the following VITPP Procedures also apply specifically to this SOT:

No.	Description
11-A-002	VITPP Melter Control Panel Alarms and Gem Machine Control Panel Alarms
11-C-263	VITPP Recycle Water System
11-C-266	VITPP Off-Gas Treatment System
11-C-267	VITPP Feed Preparation Phase I
11-C-269	VITPP Melter Operation
11-C-270	VITPP Process Cooling Water System
11-C-271	VITPP Electrode Holders Cooling System Operation
11-C-272	VITPP Gem Machine Operation
11-C-274	VITPP Treated Water (TW) System
11-C-276	VITPP Phase I Process Sampling Procedure
11-C-278	VITPP Roller Conveyor Operations
11-C-279	VITPP Utility Air System
11-E-001	VITPP Emergency Operating Procedure, Loss of Electrical Power
VIT-1000	VITPP Carcinogen Control Plan. Phase I Bag Dump Activities

3.4 GTS Duratek Procedures

No.	Description
OP-CRU4-05	Temperature and Power Control Procedure
OP-CRU4-07	Maintenance Procedures for the Melter
PI-CRU4-04	GTS Duratek DuraMelter™ 1000-HT 36 Hour Test Plan
PI-CRU4-06	GTS Duratek Gem Making Machine Test Plan

3.5 General References

Phase I Work Plan, WP-25-0007
Instrument Index, VITPP PO 85 & 92
VITPP Technical Specifications
Equipment Standard Operating Procedures
Vendor Operating Manuals, all.
FERMCO Lockout/Tagout Procedure, OP-0004.
Material Safety Data Sheets for VITPP Substances.
Startup Plan, 18-SU-0001
SO Test Plan, 18-SU-0002
Phase I Test Plan, 18-SU-0003
Safety Performance Requirement Manual, RM-0021
Phase I Operational Safety Guide, VIT-OSG-1007.2
Project Specific Plan, Phase I Process Sampling, 2504-SU-0011

VITRIFICATION PILOT PLANT STARTUP PROGRAM - PHASE I - PILOT PLANT SYSTEMS OPERABILITY TEST PROCEDURES GLASS CAMPAIGN 2	TEST PROCEDURE NO. 2504-SU-0035
	Effective: August 1996 Revision No. 0
	Page 7 of 23

4.0 PREREQUISITES

This section lists required conditions that must be verified by the Test Coordinator prior to SO testing.

NOTE: Not all situations are known or foreseeable. Unusual conditions or circumstances could change the steps in this procedure or add steps.

4.1 Lock(s) and Tag(s) have been removed from equipment to be tested.

Paul D. Plumb 8/26/96
Test Coordinator Date

4.2 The Facility Owner has been notified of intention to start test.

Thomas Cofield 8-23-96
Test Coordinator Date

4.3 Required equipment maintenance and pre-start service has been completed.

Paul D. Plumb 8/26/96
Test Coordinator Date

4.4 Process Water (treated water from FEMP site), local Cooling Tower water, and fire protection water from the FEMP site are available to support testing.

Richard Rathgeber 8/22/96
Test Coordinator Date

4.5 Instrument Air from the VITPP air compressor and Uninterruptable (bottled) Air is available.

Richard Rathgeber 8/22/96
Test Coordinator Date

4.6 Electrical power is available to the VITPP to the extent necessary to support the specific electrical line-up attachment for the applicable section being performed.

Richard Rathgeber 8/22/96
Test Coordinator Date

4.7 The Off-Gas System and Emergency Off-Gas System are operating in accordance with Procedure 11-C-266, "VITPP Off-Gas Treatment System."

Richard Rathgeber 8/22/96
Test Coordinator Date

4.8 Drums are in the Startup, Emergency, and Gem positions.

Thomas L. Crawford 8-23-96
Test Coordinator Date

4.9  Operators and persons involved with Campaign 2 have read or been briefed on the appropriate section(s) of this procedure and have signed the Signature Record Log (Attachment 1) prior to commencing their assigned task.

Thomas L. Crawford 8-26-96
Test Coordinator Date

4.10  A sufficient supply of slurry/surrogate chemicals (as specified in Attachments 2, 3, 8 and 9) is available to support this SOT.

Thomas L. Crawford 8-23-96
Test Coordinator Date

4.11 A sufficient supply of 55 gal drums (minimum of 20) are available to support this SOT.

Richard Rathgeber 8/22/96
Test Coordinator Date

VITRIFICATION PILOT PLANT STARTUP PROGRAM - PHASE I - PILOT PLANT SYSTEMS OPERABILITY TEST PROCEDURES GLASS CAMPAIGN 2	TEST PROCEDURE NO. 2504-SU-0035
	Effective: August 1996 Revision No: 0
	Page 9 of 23

4.12 A sufficient supply of side-chamber frit (minimum of 2000 lb) is readily available to support this SOT.

Richard Rathgeber 7/22/96
Test Coordinator Date

4.13 The Scrubber has been charged with caustic per SOP 11-C-266, VITPP Off-Gas Treatment System.

Fred D. [illegible] 8/26/96
Test Coordinator Date

4.14 The Startup diverter is positioned over the (east) Startup drum and Startup gate is OPEN.

Thomas L. Crawford 8-25-96
Test Coordinator Date

4.15 A sufficient supply of sampling equipment and supplies are available to support this SOT.

Thomas L. Crawford 8-23-96
Test Coordinator Date

4.16 The Analytical Lab has been notified that slurry feed operations are commencing.

Mary K. Thomas 8/22/96
Test Coordinator Date

4.17 Notify VITPP QA prior to initiating Section 7.0.

Richard Rathgeber 8/22/96 Call Slip Taylor @ 19:30
Test Coordinator Date

MB 8/22/96 19:40

000374

4.18 The following Operator Aids have been reviewed and updated:

Utility Rack settings, Melter Control Panel Setpoints, SCR Control Panel Setpoints, Gem Machine Control Panel Setpoints, and Control Room Statue Board.

Paul D. Fish 8/26/96

Test Coordinator Date

4.19 ~~Flushing~~, treatment and disposal of slurry batches containing lead or barium compounds ~~has been planned~~ with subsequent approval by the appropriate site groups, (e.g. Environmental Compliance, AWWT, etc.).

Thomas L. Caldwell 8.25.96

Test Coordinator Date

4.20 A sulfur dioxide (SO_2) monitor shall be installed and operable in the Melter room with an alarm set point of 2.5 ppm.

Thomas L. Caldwell 8.25.96

Test Coordinator Date

4.22 Exceptions to above prerequisites shall be noted in the Test Log.

Thomas L. Caldwell 8.26.96

Test Coordinator Date

Operations personnel are prepared to track the use of lead and barium compounds used in Campaign 2 Slurries. Inventory of lead and barium will be maintained including available quantities, added quantities, mixed quantities, and quantities and disposition of spilled material containing lead and barium compounds. Unused slurry samples and Flushes containing these compounds will be returned to the slurry tanks after analysis is performed consistent with Environmental/ AWWA. Inventory sheets found in Attachment II as Attachments IIA, IIB, IIC, and IID.

Reference 12-16

5.0 TEST EQUIPMENT

NONE

6.0 PRECAUTIONS and LIMITATIONS

Special system or equipment operating conditions which could be dangerous (e.g. close proximity of personnel to rotating or moving equipment or equipment that is thermally hot) will be labelled and/or barricaded and the proper safe method of operation will be identified.

Specific operating parameters (e.g. design limits for temperature, pressure, or flow), which if exceeded could cause personal injury or equipment damage, will be identified and limitations specified.

Equipment or systems that must be started or stopped in a certain sequence are identified in Standard Operating Procedures. Notifications should be made to the Shift Supervisor in the VITPP of the starting and stopping of systems or equipment (motors, blowers, fans, pumps, etc.).

Required notifications or approvals necessary to prevent problem conditions should be brought to the attention of the Test Coordinator through test briefings.

6.1 All personnel performing steps within this procedure shall familiarize themselves with this procedure to the degree their test function dictates and indicate such by placing their printed name, signature, initials, and badge number on the Signature Record Log (Attachment 1).

6.2 The FERMCO Lockout/Tagout (Hazardous Energy and Material Control) Procedure, OP-0004, will be administered, and only trained and authorized personnel will conduct the lockout/tagout sequence.

6.3 Sections/steps in this procedure may be performed out of sequence or concurrently as determined necessary by the Test Coordinator with authorization from the Shift Manager. Special care and review by the Test Coordinator should be given in the event sections/steps are performed out of sequence. The exceptions shall be documented according to project procedures and logged in the Test Log.

6.4 If this test is being performed to demonstrate the operability of select components, any steps not applicable to the component(s) being tested may be noted N/A, as determined by the Test Coordinator.

6.5 In the event that conditions require components to be disabled, jumpered, or re-aligned to support unanticipated test conditions, the Test Coordinator, Shift Manager, and System Engineer must approve. Explanations and justification and sign-off by the Test Coordinator and Shift Manager for all changes in configuration must be entered into the Test Log.

6.6 **This campaign uses lead oxide, nickel oxide, crystalline silica and vanadium pentoxide which are either highly toxic or carcinogenic chemicals.** Strict adherence to Radiological Work Permit (RWP),and the VITPP Carcinogen Control Plan-Phase 1 Bag Dump Aditivities (VIT-1000) shall be maintained, especially with respect to engineering, administrative and PPE controls. Any situation which could result in the inhalation, ingestion, or absorption of a hazardous material shall immediately be reported to a Supervisor or to the Assistant Emergency Duty Officer (AEDO), who will immediately report the circumstances to Medical and Industrial Hygiene. The involved personnel shall be directed by the Supervisor or AEDO as to when and where to report for medical evaluation, completion of an Incident Investigation Report (IIR) (Form No. FS-F-1458), and submitting bioassay samples (e.g. blood, urine). Employees are responsible for complying with any additional requirements as specified by OS&H.

6.7 The Shift Manager shall have responsibility for all testing activities.

6.8 The Test Coordinator shall have responsibility for coordinating all testing and data gathering activities, including gathering data specifically requested by the GTS Duratek technical support person.

6.9 The Shift Supervisor shall have responsibility for execution of all equipment operation adjustments and energy isolation to support testing activities.

6.10 The System Engineer shall be responsible for on-shift engineering support and ensuring that systems are available and operational to support the testing activities.

6.11 All conductive items in contact with the molten glass pool have the potential to be electrically energized. Never attempt to defeat or remove any protective covers, cages, or barriers unless the electrodes are de-energized and appropriate supervisory permission has been explicitly received.

6.12 All exterior surfaces of the Melter, in contact with the molten glass discharge stream, off-gas piping ducts, and glass receptacles can become extremely hot. Never attempt to handle components or newly formed glass without appropriate heat-resistant clothing. The minimum required PPE includes heat-resistant leather gloves, heat resistant apron, and heat-resistant leather sleeves or comparable.

6.13 Failure to follow the requirements of this procedure may result in severe personnel injury and/or damage to equipment and components.

6.14 The air bubblers (FI-001 through FI-004) and air trickle flow (FI-006) must be energized AT ALL TIMES.

6.15 Contact Industrial Hygiene for a heat stress survey when the ambient air temperature in the melter room exceeds 80°F.

6.16 If the Melter is idled for greater than 3 days, then consideration should be given to turning over the glass composition to the startup composition.

6.17 The bottom drains, electrodes, center chamber feed tube, center chamber hopper, side drain and side feed hoppers must have Melter cooling water flowing through them **AT ALL TIMES.**

7.0 DETAILED TEST PROCEDURE

Steps that record data will require the Test Coordinator to note instrument readings, initials, and date in the attachments provided, or record in the Test Log. Other operating data that are routinely recorded by the operators will be recorded on operator Round Sheets and the DCS log. Additionally, DCS operating data is stored in the VPPS database.

This Test Procedure consists of the step-by-step performance of testing activities. Appropriate areas will include a place for action step sign-off and/or verification (and a place for recording data). Testing is accomplished by implementation of this document along with the performance of other equipment Standard Operating Procedures (SOPs), and Long Term Orders as required. Other items included may be warnings, cautions, notes, hold, or witness points, and system restoration. Pending review of the results from the performance of this procedure, certain steps may be changed (through proper change control methods) to obtain a specific result.

7.1 TEST: Transition Glass in Center Chamber and Heat-Up Melter to 1350°C

NOTE: Operators shall complete Attachments 7A and 7B every 30 minutes when the Melter system is stable during batch feeding.

NOTE: Test Coordinator shall complete Attachment 6 for each batch.

7.1.1 Ramp up Melter temperature to 1250°C (+/- 20°C) per SOP 11-C-269, "VITPP Melter Operation".

7.1.2 Prepare a batch of glass slurry according to the formula on the Campaign Transition Glass Batch Sheet (see Attachment 8).

7.1.3 Collect sample per PSP (Project Specific Plan, 2504-SU-0011) from the Slurry Tank approximately 30 minutes after chemical addition is completed.

7.1.4 Submit the sample for analysis per the PSP and Test Coordinator.

7.1.5 Review analytical results.

7.1.6 (3) Verify batch is acceptable. 000379

VITRIFICATION PILOT PLANT STARTUP PROGRAM - PHASE I - PILOT PLANT SYSTEMS OPERABILITY TEST PROCEDURES GLASS CAMPAIGN 2	TEST PROCEDURE NO. 2504-SU-0035
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E, 8-25-96 7.1.7 Confirm the drums are empty or tare weights are known.

E, 8-26-96 7.1.8 Discharge glass to Startup or Emergency drum until approximately 10 percent of the center chamber is drained. (Approximately 400 pounds).

E, 8-26-96 7.1.9 Begin feeding slurry to the Melter. Increase feed rate at 25 rpm/min towards target feed rate to establish a feed rate that creates a stable and desirable cold cap, (approximately 100 - 300 rpm).

E, 8-26-96 7.1.10 Continue to feed Melter until the slurry tank weight that the Melter is being feed from decreases by one sixth, then stop feeding the Melter.

___/___ 7.1.11 ~~Discharge glass to Startup or Emergency drum until approximately 10 percent of the center chamber is drained. (Approximately 400 pounds). If the glass does not flow freely through the discharge chamber (due to high viscosity), then increase the Melter temperature to 1350°C (+/- 20°C).~~ ICP 03-9 E 8-26

P, 8/24/96 7.1.12 Please Tie. A Resume feeding slurry to the Melter. Increase feed rate at 25 rpm/min towards target feed rate to establish a feed rate that creates a stable and desirable cold cap, (approximately 100 - 300 rpm). Add approx 59# Of Magnesium Phosphate

P, 8/27/96 7.1.13 Continue to feed Melter until the slurry tank weight that the Melter is being feed from decreases by one sixth ~~then stop feeding the Melter.~~

P, 8/27/96 7.1.14 Discharge glass to Startup or Emergency drum until approximately 10 percent of the center chamber is drained. (Approximately 400 pounds). If the glass does not flow freely through the discharge chamber (due to high viscosity), then increase the Melter temperature to 1350°C (+/- 20°C), if not already done.

P, 8/27/96 7.1.15 ~~Add approx 59# Of Magnesium Phosphate at the direction of the Test Coordinator~~ A ~~Resume feeding slurry to the Melter. Increase feed rate at 25 rpm/min towards target feed rate to establish a feed rate that creates a stable and desirable cold cap, (approximately 100 - 300 rpm).~~ Add approx 59# Of Magnesium Phosphate at the direction of the Test Coordinator until the 354# inventory has been added to Melter. IC 03-8

___/___	7.1.16	Continue to feed Melter until the slurry tank weight that the Melter is being feed from decreases by one sixth ~~then stop feeding the Melter~~.
___/___	7.1.17	Discharge glass to Startup or Emergency drum until approximately 10 percent of the center chamber is drained. (Approximately 400 pounds). If the glass does not flow freely through the discharge chamber (due to high viscosity), then increase the Melter temperature to 1350°C (+/- 20°C), if not already done.
___/___	7.1.18	Resume feeding slurry to the Melter. Increase feed rate at 25 rpm/min towards target feed rate to establish a feed rate that creates a stable and desirable cold cap, (approximately 100 - 300 rpm).
___/___	7.1.19	Continue to feed Melter until the slurry tank weight that the Melter is being feed from decreases by one sixth ~~then stop feeding the Melter.~~
___/___	7.1.20	Discharge glass to Startup or Emergency drum until approximately 10 percent of the center chamber is drained. (Approximately 400 pounds). If the glass does not flow freely through the discharge chamber (due to high viscosity), then increase the Melter temperature to 1350°C (+/- 20°C), if not already done.
___/___	7.1.21	Resume feeding slurry to the Melter. Increase feed rate at 25 rpm/min towards target feed rate to establish a feed rate that creates a stable and desirable cold cap, (approximately 100 - 300 rpm).
___/___	7.1.22	Continue to feed Melter until the slurry tank weight that the Melter is being fed from decreases by one sixth ~~then stop feeding the Melter~~.

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___/___	7.1.23	Discharge glass to Startup or Emergency drum until approximately 10 percent of the center chamber is drained. (Approximately 400 pounds). If the glass does not flow freely through the discharge chamber (due to high viscosity), then increase the Melter temperature to 1350°C (+/- 20°C), if not already done. KP 03-96 E 8-26-96 E
___/___	7.1.24	Resume feeding slurry to the Melter. Increase feed rate at 25 rpm/min towards target feed rate to establish a feed rate that creates a stable and desirable cold cap, (approximately 100 - 300 rpm).
KP 9-4-96	7.1.25	Continue to feed Melter until the slurry tank that the Melter is being feed from is empty, then stop feeding the Melter.
MKH 9/4/96	7.1.26	If not already done, increase Melter temperature to 1350°C (+/- 20°C) per SOP 11-C-269, "VITPP Melter Operation", and hold for a minimum of two hours. Start Time: 2100 9/3/96 PP Stop Time: 0730 9/4/96
MKH 8/27/96	7.1.27	Collect sample of center chamber glass by discharging and submit it to be analyzed for total metals.
E 9-1-96	7.1.28	Verify center chamber glass is acceptable. Rod J Gruyel 8-28-96 Data Testing Manager Date
PP, 9/4/96	7.1.29	Obtain Operations Manager's consent to continue. Judith G Primus 9-3-96 Operations Manager Date Per telecon Approved By JOEL BRADBERRY.

000382

7.2 TEST: Prepare Acceptance Glass Slurry Batch

Initials/Date		
E 1/9-3-96	7.2.1	Make a slurry batch of Acceptance Glass in a Slurry Tank using the batch formula listed in Attachment 2 and recirculate the slurry through the Melter feed loop or the Melter bypass loop (as specified by the Shift Manager) as described in SOP 11-C-267, VITPP Feed Preparation Phase I. ICP 04-96 NKM 5/28/96 28
E 1/9-3-96	7.2.2	Sample the surrogate batch in accordance with the PSP (Project Specific Plan, 2504-SU-0011), and per SOT 11-C-276, VITPP Phase 1 Process Sampling Procedure. ICP 05-96 5/30/96
E 1/9-3-96	7.2.3	Submit the sample for analysis per the PSP and Test Coordinator.
RP 9/5/96	7.2.4	Review analytical results.
NA	7.2.5	If analytical results are not acceptable, adjust slurry batch formula as necessary.
RL 9/5/96	7.2.6	Make another slurry batch of Acceptance Glass surrogate in the other Slurry Tank using the surrogate batch formula listed in the revised per ICP 08-96 Attachment 2, and recirculate the slurry through the Melter feed loop or the Melter bypass loop (as specified by the Shift Manager) as described in SOP 11-C-267, VITPP Feed Preparation Phase I. ICP 05-96 NKM 8/30 NKM 9/4
RL 9/5/96	7.2.7	Sample the surrogate batch in the second tank in accordance with the PSP, Project Specific Plan, 2504-SU-0011, and per SOT 11-C-276, VITPP Phase 1 Process Sampling Procedure.
RL 9/5/96	7.2.8	Submit the sample for analysis per the PSP and Test Coordinator.
/	7.2.9	Review analytical results.

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NOTE: Prepare additional batches of Acceptance Glass surrogate as needed. Sample each slurry batch in accordance with the PSP, Project Specific Plan, 2504-SU-0011, SOT 11-C-276, VITPP Phase 1 Process Sampling Procedure, and as directed by the Test Coordinator.

7.3 TEST: Run 36 Hour Melter Performance Test

NOTE: Operators shall complete Attachments 7A and 7B regularly every 30 minutes when the Melter system is stable during batch feed.

NOTE: Test Coordinator shall complete Attachment 6 for each batch.

NOTE: Attachments 4 and 5 are to be used to determine the Melter Feed Pump rpm to achieve one metric ton per day.

NOTE: Attachment 10.1 shall be completed at the begining every 2 hrs during the 36 hr Melter Performance Test.

Initials/Date

AM 9/4/96 7.3.1 Begin the 36 hr performance test of the Melter. The test shall be continuous at a minimum rate of one metric ton per day of glass produced for 36 hours.

SK 22 9/4/96 1007
Shift Manager Date/Time

[signature] 9-4-96 10:07
Duratek Field Rep. Date/Time

NOTE: The Gem Machine performance test (Section 7.4) shall be run concurrently with the Melter performance test after Melter feed is increased to a minimum of 1 MT/D glass rate.

NOTE: The 36 hours refers to total run time while feeding the Melter. Reasonable downtime for maintenance unrelated to the Melter and Gem Machine is allowable. Additionally, the feed rate can be adjusted to control the cold cap as approved by GTS Durateck Technical Representative.

000384

7.3.2 Continue Melter feed to maintain steady state operation and hold with concurrence from the GTS Duratek technical support person.

7.3.3 Operators shall complete Attachment 10.1, Duramelter Acceptance Testing Table.

7.3.4 Commence discharging the Melter into a drum as advised by the GTS Duratek technical support person.

7.3.5 Continue to operate Melter at a steady state with concurrence from GTS Duratek Technical Support person.

7.3.6 Completed 36 hr Acceptance Test:

9-7-96 1620
Shift Manager Date/Time

9/7/96 1440
Durateck Field Representative Date/Time

7.3.7 VITPP Manager approval of completion of the 36 hour Acceptance Test:

9/12/96 0800
VITPP Manager Date/Time

7.3.8 Lower Melter temperature to 1250° C (+/- 20° C) after completion of 36 hour test.

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7.4 TEST: Run 8 Hour Gem Machine Performance Test

NOTE: The 8 hours refers to total run time of the Gem Machine while producing gems when feeding the Melter. Reasonable downtime for maintenance unrelated to the Melter and Gem Machine is allowable. The goal is to run the Gem Machine continuously for at least two hours.

NOTE: Operators shall complete Attachments 7A and 7B initially every 30 min when the Melter system is stable during slurry feed.

NOTE: Test Coordinator shall complete Attachment 6 for each batch.

NOTE: Weigh Gem drums prior to the start of this section.

NOTE: Attachment 10.2 shall be completed every 2 hrs during the 8 hr Gem Machine Performance Test.

Initials/Date

7.4.1 With Melter being fed slurry at a steady state operating rate, (target rate of approximately 95 pounds per hour glass production rate), begin Gem Machine 8 hour performance test with concurrence from the GTS Duratek technical support person.

Shift Manager Date/Time

Duratek Field Rep. Date/Time

7.4.2 Ensure that the Gem Machine has operated during the 8 hrs and has produced a minimum of 730 lb gems (1/3 MT or a 55 gal drum about 80% full).

7.4.3 Operators shall complete Attachment 10.2, Gem Machine Acceptance Testing Table.

000386

_____ 7.4.4 Complete 8 hour Gem Machine Performance Test.

_____
Shift Manager Date/Time

_____
Duratek Field Rep. Date/Time

7.5 **TEST: Run Melter at 1250°C**

NOTE: Do not proceed to the next step until a GTS Duratek technical support person and VITPP Manager concurs that the 36 hour performance test is complete.

NOTE: Operators shall complete Attachments 7A and 7B regularly every 30 min when the Melter system is stable during slurry feed.

NOTE: Test Coordinator shall complete Attachment 6 for each batch.

Initials/Date

E, 9/9/96 7.5.1 Increase Melter temperature to 1300°C or that temperature required for glass to flow freely through the discharge chamber.

SKK 9/9/96 7.5.2 Make batch using formulation listed in Attachment 9a. ICP 10-3 SKK 9/12

E, 9/10/96 7.5.3 Start feeding Melter with Series D surrogate and additives slurry to achieve a stable cold cap.

E, 9/10/96 7.5.4 While feeding Series D glass, slowly lower temperature to 1250°C.

____ 7.5.5 Run as continuously as possible at an optimum operating rate for a minimum of 2 batches or as directed by the Operations Manager. Vary feed rate, bubbler rate, urea amounts, % solids, cold cap size, etc. to help establish operating envelope as directed in the Daily Orders. ICP 11-9 SKK 9/10

Batch and run TRANSITION BATCH, as shown in ATTACHMENT 12 (C2B96). Run BATCH C2B96 between the 2 Series D batches. E 9/10/96

___/___	7.5.6	Feed the last slurry batch (leaving both Slurry Tanks with a minimal heel) and stop feed to Melter.
___/___	7.5.7	Hold Melter at standby at a temperature to 1250°C or other temperature as directed by the Operations Manager.

TEST EXCEPTION LOG			
TE#	DATE	DESCRIPTION INCLUDE TP STEP #	COMPLETION/ APPROVAL(DATE)
1	9-7-96	Germ Machine Test 7.4 2	9-7-96

TEST EXCEPTION REPORT FORM

TE #	TD/Doc #	Originator/Date/Phone	Section/Step #
1	2504-SU-0035	M. Morse/9-7-96/5331	7.4 / 7.4.2

Exception/Discrepancy

Attempts to perform the Gem Machine 8-hour test were unsuccessful due to the double glass stream flowing from the discharge orifice.

Resolution/Correction

Remarks/Continuation

Approvals:

Mary K Morse 9-7-96 _____ _____

Test Coordinator / Date

_____ _____ _____

Facility Owner / Date

REFERENCE 13

heat flows in or out of the glass, the glass temperature becomes uniform by internal radiation.

Another recent development is the use of ictory panels which contain embedded .ing elements. These electric blocks are used over the top of forehearths which are temperature controlled by means of immersed electrodes, along the sides and bottom of forehearth channels and in the bowl, to retard the loss of heat from the glass to achieve more nearly uniform temperature conditions in the forehearth.

Electrical heating of gathering chambers by means of radiant elements and immersed electrodes makes more comfortable working conditions due to absence of flame sting-out. Surface cords due to evaporation of a glass constituent are reduced because the atmosphere in the chamber is partly saturated with the vapor and is quiescent.

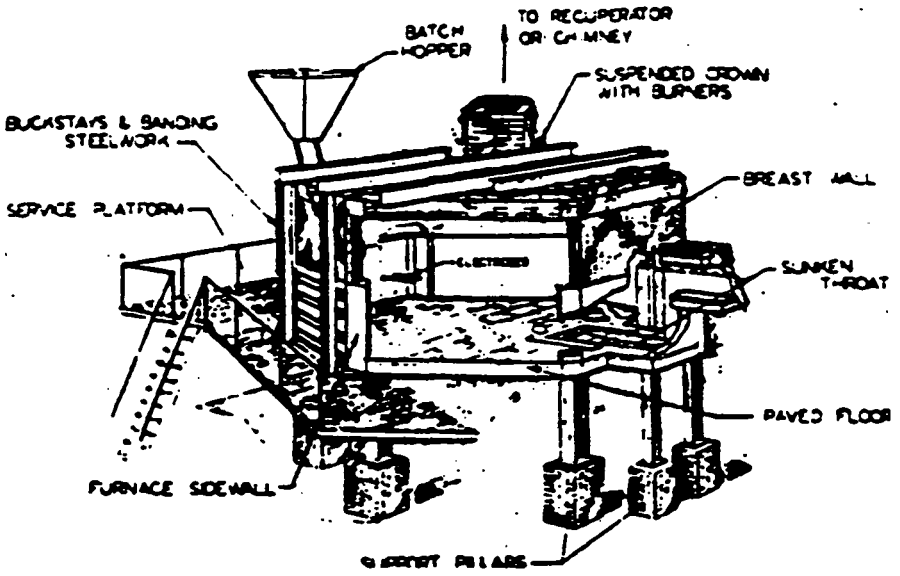


Figure 4. Mixed melt furnace.

8. Electrode Materials

There are four common electrode materials used in electric melting: molybdenum, platinum, molybdenum disilicide, and graphite, listed in the order of increasing contact resistance.

Molybdenum serves the purpose well, and is used in most applications. Molybdenum has a high melting point, low contact resistance to glass, and its oxide is non-coloring. It has the disadvantage of poor oxidation resistance, and a water jacket is ired where the electrode goes through .rnace wall.

Molybdenum disilicide is not used extensively, but is being considered because it has resistance to oxidation. It has its own disadvantages of being more reactive to oxidized glass than pure molybdenum, and not as adaptable to joining on an extension as is customary with molybdenum to compensate for wear.

Platinum resists oxidation but since it has a somewhat higher contact resistance to glass, the current density must be kept low or the frequency high to avoid sputtering the metal into the glass. When this takes place, the glass becomes foggy. Platinum has the disadvantages of high capital cost, and a significant maintenance cost because of failure due to recrystallization.

Graphite and carbon are not wetted very well by glass, and hence have a high contact resistance. Again, the current density must be kept low, or the frequency high to prevent incipient arcing which reduces silica to silicon in finely dispersed form. It is also attacked by oxidized glasses. Another disadvantage of graphite is that there are significant heat losses in the cooling of the shank which extends to the outside of the furnace.

9. Control Systems

'irnaces which are all-electric must have type of power input control, since the .tance of the glass falls as the temperature rises, and vice versa. For example, if the transformer is kept at a fixed voltage, the power input will be some definite amount at time zero. If that power is appreciably more than the furnace requires to balance the heat losses and melting load, the temperature of the bath will rise and the current will increase due to the decreased resistance. On the other hand, if the power going into the furnace is appreciably less than that required, the glass will cool and the current will decrease due to the increased resistance.

The foregoing is particularly important with glasses having a steep temperature/resistance curve, such as heat-resisting borosilicate. It is of less importance with soda-lime glasses, due to the relatively flat resistance-temperature curve at melting temperatures. It is practical to control a 60-ton electric furnace by observation of the temperature reading followed by manual changing of the transformer taps. A 60-ton furnace with this control arrangement has been operating satisfactorily for many years, and the need for tap changes occurs only a few times in 24 hours.

All-electric furnace power-control systems that may be used are as follows:

(1) Transformer with many taps giving various output voltages, selected manually or automatically with the power off.

(2) Transformer with many taps giving output voltages, selected manually or automatically with the power on. This is more convenient than the first method.

(3) Transformer of fixed voltage or with a limited number of taps, with buck-and-boost voltage regulator to obtain intermediate voltage, selected manually or automatically with the power on. This is equally as good as the second method.

(4) Transformer of fixed voltage or with a limited number of taps, with a saturable reactor to control the power input, selected manually or automatically. This method has the disadvantage of poor power factor, unless corrected by capacitors.

(5) Transformer of limited voltage or with a limited number of taps, with a silicon-controlled-rectifier to control the input voltage.

Booster power-control systems can be any of the above, but automatic control is not required; system No. 1 is usually practiced. Most of the heat is supplied by the burning fuel, and the overall temperature of the furnace is controlled by the operator who controls the fuel input. For example, if the temperature in the furnace begins to rise due to the startup of a booster, the operator of the furnace takes note of the temperature increase and reduces the fuel fire. In this way, the booster type of installation is stable.

The temperature of the glass in an all-electric furnace is measured by means of a thermocouple working with the usual recorder-controller type of instrument. This instrument then controls the motor on the voltage regulator or tap-changer, or it signals for the operator. The thermocouple may be the usual platinum/platinum-rhodium type installed part way through the bottom blocks or an optical-radiation type pyrometer used with a closed end molybdenum immersion tube through the sidewall.

10. Operation

Operation of all-electric furnaces is essentially no more difficult than the operation of fuel-fired furnaces. It is necessary for the operator to be able to read meters and make adjustments to the controls. The factors to be watched are: power input, glass temperature, batch charging uniformity, cooling water temperature, and electrode condition. If desired, the all-electric furnace can be made fully automatic, with no operator required, except for occasional checking.

Maintenance consists in checking the electrodes every week and making adjustments when necessary, and general checking of blocks, water system, batch handling equipment, thermocouples, and other electrical equipment.

000392

REFERENCE 14

sistance is given by Gailhbaud[19] as

$$R = \frac{3}{2}R_1 + \frac{3}{2}\frac{\rho_{el}}{\pi h}\left(\ln\left|\frac{d}{h}\right| + 1{\cdot}441 - 5\ln\left|2 - \frac{\delta}{d}\right|\right). \quad (8.33)$$

The symbols are described in Figure 8.25 and R_1 is defined by the relationship already given in Section 8.4.3.2.

8.5 Electrode materials

The development of electric melting is largely linked to the discovery and development of electrode materials. The properties and criteria that an electrode material must meet are:

1. resistance to corrosion by molten glass and refractories, irrespective of oxygen partial pressure and current density up to 1700°C
2. resistance to oxidation in air at least up to 800°C
3. electrical conductivity similar to that of metals
4. adequate mechanical strength up to 1700°C
5. adequate thermal shock resistance
6. properties for manufacture should be similar to those of metals
7. an economically viable price.

The ideal electrode material has not yet been found and probably never will be, so compromises must be made, which means that, depending on the application and the technical and economic conditions, different electrode materials will be selected.

During the early development of electric melting, electrodes were made of pure metals such as iron and nickel or alloys of these, and they had to be intensively cooled to provide adequate mechanical properties. With a few exceptions, such materials are of no importance as electrode materials today and the field is led by molybdenum, platinum, tin oxide, and, up to a point, graphite.

8.5.1 Molybdenum

Molybdenum, a metal of high strength at high temperature and a melting point of 2610°C, meets requirements (1) and (2) up to a point and (3) to (7) adequately, and is now the most commonly used electrode material. In many ways the development of this material until it was suitable for use in molten glass has been largely responsible for the wide distribution of electric glass melting.

The physical properties of molybdenum as used for electrodes are given in Table 8.3; its electrical resistivity at room temperature is three times that of copper but by 1500°C it is thirty times that of copper.

Molybdenum electrodes are particularly suitable for glasses melted under reducing conditions but glass constituents such as lead, arsenic, antimony, and, to a certain extent, SO^{2-} have an adverse effect. They are in the electrochemical series above molybdenum and their oxides would therefore be reduced to the parent metal even if no current were flowing; even 1% PbO in a glass produces noticeable corrosion of molybdenum but it is claimed that special electrical networks (Figure 8.26) can lead to a reduction in the attack of lead on molybdenum.[24,25] In practice, the use of molybdenum as an electrode material has been found to be satisfactory provided that, for each 1000 kg of sand in the batch, the

Table 8.3. *Physical properties of molybdenum*

Melting point	(°C)	2630 ± 40
Vapour pressure	(mbar)	$8{\cdot}5 \times 10^{-9}$ at 1500°C $10{\cdot}7 \times 10^{-7}$ at 1800°C $5{\cdot}5 \times 10^{-5}$ at 2000°C
Specific heat	(J/g K)	0·27 at 20°C 0·31 at 1000°C 0·33 at 1400°C
Thermal conductivity	(W/m K)	152 at 20°C 103 at 1000°C 66 at 1600°C
Thermal expansion	(10^{-6}/K)	5·3 at 20°C
Specific electrical conductivity	(μΩ cm)	5 at 20°C 27 at 1000°C 43 at 1500°C 60 at 2000°C

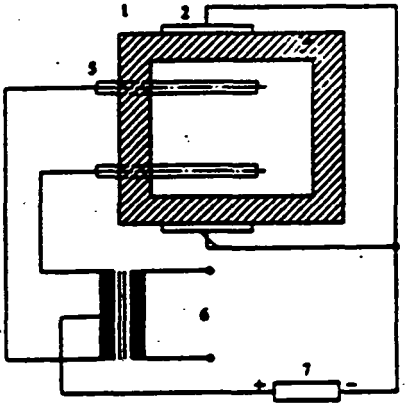


Figure 8.26. Protection of electrodes by a direct current voltage

000394

Reference 14

SIGNATURE PAGE

2/25/97
Raymond H. Reinhart, Team Leader — Date
Fluor Daniel Fernald

2/25/97
Gail E. Bingham — Date
Independent Consultant

2/26/97
Joel B. Bradburne — Date
Fluor Daniel Fernald

2/28/97
Hamid Hojaji — Date
~~Duratek~~ VSL / GUA

Vijay Jain — Date
West Valley Nuclear Services, Co.

2/25/97
Ronald L. Joseph — Date
Fluor Daniel Fernald

2-28-97
Joseph G. Metzler — Date
Electrician

2/25/97
Donald Paine — Date
Fluor Daniel Fernald

2/25/97
Kareld A. Solomon — Date
Fluor Daniel Fernald

2-26-97
Ron C. Worsley — Date
Fluor Daniel Fernald

ATTACHMENT B

VITRIFICATION PILOT PLANT

SAFETY REVIEW TEAM REPORT

Report No. 40100-RP-0002

Vitrification Pilot Plant Melter Incident: Safety Review Team

February 13, 1997

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Attachment 2: Bubbler Tubes

Attachment 3: Summary of SO_2 Monitor Printouts

Attachment 4: Estimated Exposure Rate and Airborne Radioactivity Concentration - Calculation No. SD-S&H-BAS-3034

Attachment 5: Organization

Attachment 6: Team Member Qualifications

Attachment 7: Documents Reviewed

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List of Acronyms and Abbreviations

AEDO	Assistant Emergency Duty Officer
ALARA	As Low As Reasonably Achievable
ARARs	Applicable or relevant and appropriate requirement
ASR	Auditable Safety Record ASR 95-0006 for: Phase I Vitrification Pilot Plant Operations
AWWT	Advanced waste water treatment
BDC	bottom drain container
BOP	Balance of Plant
BST	building sump tank
CM	configuration management
CO_2	carbon dioxide)
DOE	Department of Energy
ED	engineering design
FAT&LC	Fernald Atomic Trades & Labor Council
FDF	Fluor Daniel Fernald
FEMP	Fernald Environmental Management Project
FEP	erythrocyte protoporphyrin
FHAU	Fire Hazards Analysis Update
FS	Fire Safety
FSAR	Final Safety Analysis Report
gpm	gallons per minute
HAZOP	Hazard and Operability Analysis Report
HC-3	Hazard Category 3
HNT	high nitrate tank
ISRC	Independent Safety Review Committee
LH	Low Hazard
MAWS	Minimum Additive Waste Stabilization
MCC	motor control center
mg/l	milligram per liter
NO_x	oxides of nitrogen
NPDES	National Pollution Discharge Elimination System
NRC	National Response Center
°C	degrees Celsius
°F	degrees Fahrenheit
OS	Occupational Safety
OSG	Operational Safety Guidance Document
OU4	Operable Unit 4
PEP	Project Execution Plan
PG-3	Performance Grade of 3
PHA	Preliminary Hazard Analysis Report
PHASP	Project Specific Health & Safety Plan
PPE	personnel protection equipment
ppm	parts per million
PSAR	Preliminary Safety Analysis Report
RCF	Refractory Ceramic Fiber
RCRA	Resource Conservation and Recovery Act

List of Acronyms and Abbreviations (Continued)

SA	Safety Analysis
SCR	silicon control rectifiers
SO_2	sulfur dioxide
SO_x	oxides of sulfur
SRT	Safety Review Team
SSC	structures, systems, and components
STEL	short-term exposure limit
TCLP	Toxicity Characteristic Leaching Procedure
TRB	Technical Review Board
TWA	time-weighted average
USQ	Unreviewed Safety Question
USQD	Unreviewed Safety Question Decision
VITPP	Vitrification Pilot Plant
VSL	Vitreous State Laboratory
wt%	weight per cent
ZPP	zinc protoporphyrin
μg/dl	micrograms per deciliter
$\mu g/m^3$	micro grams per cubic meter

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Executive Summary

Fluor Daniel Fernald (FDF) chartered a Safety Review Team (SRT) as part of the investigation of the breach of the VITPP melter on December 26, 1996. The SRT evaluated the adequacy of implementing controls for normal operations and emergencies within the safety basis, and the appropriateness of the response to the incident. The investigation included review of safety documentation, operating procedures, test plan, and emergency procedures.

This report presents the SRT evaluation of the response to the incident, safety and health impacts on- and off-site, lessons learned, and recommendations regarding improvements to the safety analysis process for VITPP operations.

The execution of this project was in accordance with requirements established by the Department of Energy (DOE). Thorough scientific and engineering discipline was not exercised at its initiation nor throughout the duration of this project. Continuous attention to potential safety concerns resulting from the deterioration of melter components was not effectively factored into revisions of the safety basis documents, procedures, nor test plans. The discipline gained from all aspects of operational experience and personnel training ensured that the incident resulted in no personnel injuries nor environmental damage.

The Safety Review Team identified five (5) recommendations, and three (3) lessons learned:

Lesson I (Safety Analysis):

The safety assessments and hazard analysis processes for the VITPP inadequately assessed the potential for casualties resulting from incompletely understood wastage in the Melter. (Wastage is the combined effects of corrosion, erosion and electrochemical reactions which results in degradation of materials.)

Recommendation I:

Project management must assure that periodic independent technical review and direction is provided during all phases of the project. This must include safety, design and operational considerations, and it must be available during early phases of the project..

Recommendation 2:

Because of the technical complexity of this project, project management must assure that a thorough, detailed hazard assessment is performed by an integrated team with adequate technical resources to establish the safety basis, and that this

safety basis is revised as new data and facts become available.

Lesson 2 (Design Change Control):

The Design Change Control process existing at the time was ineffective in assuring that changes are adequate to maintain operations within the safety basis.

Recommendation 3:

Project management must ensure that management control processes are effectively implemented throughout the life of the project to maintain integrity of the safety basis. Not only must this include a consideration of the FDF design change control process, but also the design and safety interface between FDF and subcontractor scope of work.

Lesson 3 (Emergency Response):

Good emergency response to casualty conditions requires good design, disciplined operations, and continuous training.

Recommendation 4:

The discipline gained from all aspects of operational experience and personnel training ensured that the incident resulted in no personnel injuries. Project management must ensure that such training is a continuing requirement for a project of this complexity.

Recommendation 5:

Project management must ensure that there are appropriate designs and/or effective administrative controls and/or project procedures to mitigate and confine the consequences of a potential breach of the melter which could spill molten glass onto the floor of the melter room, as well as, the consequences resulting from emergency response.

Introduction

The Safety Review Team (SRT) for the investigation of the Vitrification Pilot Plant incident was chartered by Fluor Daniel Fernald (FDF) to determine the adequacy of implementing controls for normal operations and emergencies within the safety basis, and to evaluate the appropriateness of the incident response in terms of procedures and processes used. The SRT was to include in its report an evaluation of the appropriateness of response to the incident, an evaluation of safety and health impacts on and off-site, lessons learned and recommendations regarding controls for safety.

The Vitrification Pilot Plant (VITPP) is located on the west perimeter of the site and is the administrative responsibility of Fernald Residues Vitrification Plant Project. The area includes four waste storage silos, their ancillary structures and the surrounding soils, and the new VITPP. Silos 1 and 2, also known as the K-65 Silos, contain radium-bearing, low-level radioactive waste. Silo 3 contains dry, radioactive, metal oxide waste. Silo 4 was never used and is empty except for rainwater seepage. The VITPP contains the vitrification furnace and systems for product-forming and handling, off-gas treatment, furnace feed preparation and transfer, wastewater treatment, process support auxiliaries, and a control room.

VITPP Operations

The VITPP was constructed to continue treatability studies on the wastes contained in Silos 1, 2, and 3. Two phases of the pilot scale treatability study were to be performed to meet acceptance criteria and performance objectives. Phase I is the start-up of the vitrification furnace, product-forming equipment, the off-gas system, and furnace feed system to verify operation prior to vitrifying mixed waste. Phase I operations utilized surrogate material to test the equipment and finalize initial "glass making recipes" for Phase II. Phase II operations were planned to vitrify K-65 and metal oxide radioactive waste.

Phase I operations began with the bake-out of the furnace interior, driving off moisture and construction residues. Glass frit was charged into the furnace to seal construction orifices and allow power to be applied to the melter electrodes. After initial furnace bake out and start-up, a series of operational campaigns were planned to:

- Perform acceptance testing of the furnace from the vendor;
- Correlate temperature, power, current, and voltage;
- Determine the maximum glass production output of the furnace;
- Synchronize support systems with furnace and product forming operations;
- Finalize initial glass formulations for Phase II testing; and

- Familiarize operators with the vitrification process prior to Phase II.

The campaigns utilize a surrogate material, consisting mostly of sand, BentoGrout, and water. Chemicals are added to the surrogate material in the slurry feed tanks. The chemicals added vary for each test campaign and mimic specific characteristics of the waste to be vitrified in Phase II, aid in the formation of an acceptable glass product, or provide tracers for evaluating the leachability of the product.

The vitrification furnace (or melter) is joule heated. Electric current passes directly through the molten glass. There are separate chambers for the electrodes, melting, and discharge. The furnace chamber is lined with refractory bricks and operates in the range of 1,050 to 1,400 degrees Celsius (°C) or 1,992 - 2,552 degrees Fahrenheit (°F). Air bubblers agitate the molten glass. A slight negative pressure is maintained by venting the furnace into a forced-draft off-gas system. The melter is controlled by regulating the feed material. Feed is supplied to the furnace via a positive displacement pump. Molten material is transferred from the melting chamber to the discharge chamber by an air lift mechanism. Air bubbled from the bottom of the discharge chamber lifts the molten glass above the discharge hole. Glass flows into the product forming machine.

Summary of Incident

On December 26, 1996, at approximately 22:22 hours, molten glass was discovered leaking from the bottom of the melter. A maintenance supervisor had entered the melter room in response to a system engineer's request for instrument mechanic support. A camera had recently been adjusted by an instrument mechanic and the melter operator, who was located on the second floor of the melter room, noticed a wisp of smoke in the vicinity of the camera. When the maintenance supervisor entered the melter room (on the ground floor), she noticed a small stream of molten glass flowing from the bottom of the VITPP melter.

Prior to this discovery, the VITPP melter was in a normal idle (non-feeding) operating mode. The material in the melter contained some concentrations of barium and lead, but no radioactive materials. The complement of shift personnel included 9 VITPP team members, 6 maintenance personnel, and 1 radiological control technician. Occurrence Report OH-FN-FDF-FEMP-1996-0075 describes the immediate actions taken and the results. (See Attachment 1).

During the first five minutes of the melter breach, the glass pour stream had caught a cable on its descent thus steering the molten glass onto the epoxy painted floor, and the epoxy paint coating ignited. As the molten glass continued

to flow from the melter, the hole widened to approximately 2 inches in diameter. Some of the flow initially fell outside of the bottom drain container (BDC). Approximately one cubic yard of surrogate molten glass discharged from the melter to the BDC, and approximately 20 per cent of the molten glass discharged onto the floor. The quick and effective responses performed by the shift team and the emergency response team resulted in:

- No injuries or fatalities,
- No impact to the off-site or on-site environment, and
- Minimal damage to equipment.

Three incident evaluation teams were established, and the Team Leader Kick-Off Meeting was held on January 3, 1997. There are:

- ***Data Analysis and Path Forward Team***, N. Akgündüz (DOE), D. Nixon (FDF)
- ***Safety Review Team,*** L. Bogar (FDF), R. Crawford (FDF)
- ***Incident Analysis Team***, R. Reinhart (FDF)

The objective of the three review teams is to fully evaluate the melter incident and set a short-term path forward for Phase 1 operations. The goal is to have this accomplished by mid-February, 1997. This report presents the results of the Safety Review Team.

SRT Investigation Team

The Safety Review Team (SRT) membership was drawn from a wide variety of technical and operations expertise within Fluor Daniel Fernald (FDF). The SRT was augmented with Dave Jacoboski and Eric Harper to provide first-hand knowledge and information on Vitrification Pilot Plant operations, Rod Gimpel who provided requested information on Redox Couples in Silicate Melts, and Robert Frost who evaluated the environmental impact and estimated the exposure rate and airborne radioactivity concentration from a postulated failure of a VITPP Melter assumed to contain K-65 materials. In addition, Bob Tabor, safety representative for the Fernald Atomic Trades & Labor Council (FAT&LC), participated in some of the SRT meetings. Department of Energy (DOE) oversight of the work of the SRT was provided by Peter Darnell. The cooperation and assistance of all these persons is greatly appreciated by the Safety Review Team. They have contributed important information for this report.

The first meeting of the SRT was convened on January 7, 1997, and the Team decided to form three sub-teams to facilitate their investigations. The sub-teams are listed below:

Sub-Team 1: Review of Safety Basis Documentation

Raymond Crawford, Team Co-Leader
R. Douglas Daniels, Team Member
This team reviewed the safety basis documentation.

Sub-Team 2: Off-Normal Scenarios and Test Corrections

Donald Nordquist, Team Member
Q. Eric Harper, Project Support
This team reviewed operating and maintenance procedures, the test plan and casualty alarm procedures to determine if these provided appropriate controls to anticipate an incident such as the one which occurred.

Sub-Team 3: Response to Incident

William Previty, Team Member
Lou Bogar, Team Co-Leader
Dave Jacoboski, Project Support
This team reviewed procedures and training to determine if the response actions to the incident were appropriate.

The SRT also had extensive discussions with Rod Gimpel to obtain detail information on materials performance monitoring in the Pilot Plant which could have affected the integrity of the VITPP melter.

Safety Basis Documentation

Requirements

The preliminary hazard categorization documented in the Preliminary Safety Analysis Report (PSAR) for Operable Unit 4 is a Hazard Category 3 (HC-3). The categorization was performed in accordance with DOE 5480.23 *Nuclear Safety Analysis Reports* and following the guidelines of DRAFT DOE-STD-3005-YR, *Evaluation Guidelines for Accident Analysis and Safety Structures, Systems, and Components*. Construction of the VITPP was authorized by the DOE approved PSAR. The PSAR defines the safety basis for construction, design objectives and measures necessary to ensure that the facility will be constructed and operated in a safe manner. The PSAR also addresses the safety of existing silos 1, 2, and 3. The PSAR does not address operations with surrogate, non-radioactive material (Phase I).

The operations of the VITPP during Phase I were analyzed under a Safety Assessment, documented in the *Auditable Safety Record ASR 95-0006 for: Phase*

I Vitrification Pilot Plant Operations (ASR). The hazard analysis was performed utilizing the guidance of DOE-STD-1027-92, *Hazard Categorization and Accident Analysis Techniques for Compliance with DOE Order 5480.23, Nuclear Safety Analysis Reports*. The ASR establishes the safety envelope for Phase I activities and contains the facility hazard categorization (per DOE-STD-5502-94), analysis of facility hazards, and description of controls. The ASR does not address radioactive operations (Phase II).

The Final Safety Analysis Report (FSAR) provides the safety basis for the conduct of Phase II activities. The FSAR will be used to document the evaluation of all operational hazards, occupational, environmental and system safety, associated with the project. The report also lists the mitigation and control measures necessary to address the hazards identified. The FSAR is currently under development.

Chronology of Safety Basis Development

April 19, 1994: U.S. DOE, *Preliminary Safety Analysis Report (PSAR) for Operable Unit 4,* FEMP-2337, Rev 0. The PSAR is the Authorization Basis for design and construction of the VITPP. The PSAR provides the safety basis for continued silo operations.

July 7, 1994: FERMCO, Safety Analysis (SA), *Integrated Hazard Analysis: Preliminary Hazard Analysis Plan.* The document is the first in a series of documents leading to the completion of the Final Hazard Analysis Report for VITPP operations.

July 20, 1994: FERMCO, (SA), *Preliminary Integrated Hazard Analysis (PHA) for the CRU4 Pilot Plant.* The first "integrated" identification of the hazards associated with the construction and operation of the VITPP to the scope covered by the PSAR. The analysis is finalized by the FHAR.

February 2, 1995: FERMCO, (OU4), *Request for Safety Assessment, Operable Unit 4, Phase I.* Operations with non radioactive surrogate material are not within the scope of the existing safety basis documentation (PSAR).

April 5, 1995: FERMCO, Fire Safety (FS), *Fire Hazards Analysis.* Further analysis required by PSAR and PHA. The analysis was conducted in accordance with DOE Order 5480.7A, Fire Protection. Initially approved 4/5/95. Update issued (2/2/96). Recommendation made by FS were resolved and

documented in *Close Out of Fire Hazard Analysis Recommendations* M:P:(FRVP):96-0153 (3/21/96).

July 26, 1995: FERMCO, (SA), *Human Factors Engineering Design Review.* Further analysis as required by PSAR and PHA under Safety Assessment No. 94-0045. Additional design changes required an update to the report which is documented in *Final Human Factors Evaluation* SA 95-0045 Rev 1. (3/22/96). Recommendations made by (SA) were resolved and documented in Close out of Human Factor Review Comments, M:P:(FRVP):96-0242 (5/2/96).

Sept. 29, 1995: FERMCO, (SA), *Hazard and Operability Analysis Report for the Operable Unit 4 Vitrification Pilot Plant*, 25-HS-0005. Further analysis as required by PHA. The report presents the results and recommendations from the HAZOP of the vitrification furnace, gem maker, and off-gas system.

Oct. 19, 1995: FERMCO, (SA), *Final Hazard Analysis Report for Operable Unit 4*, 25-HS-0004. This report summarizes the findings from the HAZOP and PHA, which provides the hazard analysis required by DOE 5480.23 for the facility Final Safety Analysis Report (FSAR).

Dec. 20, 1995: FERMCO, (SA), *Auditable Safety Record 95-0006 for: Phase I Vitrification Pilot Plant Operations.* Result of the request for safety assessment issued 2/2/95. Provides the safety and authorization basis for Phase I Operations. Provides the hazard categorization (per DOE-STD-5502-94) of the facility. The facility is categorized as an Industrial Facility. Revisions: Rev 0. (12/20/95); Rev 1. (4/3/96); page change (4/7/96); and page change (8/20/96).

February 15, 1996: FERMCO, Occupational Safety (OS), *Vitrification Pilot Plant Phase I Operational Safety Guidance Document,* was prepared to cover issues normally addressed by a Project Specific Health and Safety Plan. Revisions: Rev. 1 (03/28/96, Rev. 2 (05/01/96), and Rev. 3 (12/16/96).

Safety Basis of Phases I and II: Design and Construction

The safety authorization for the design and construction of the facility is the Preliminary Safety Analysis Report (PSAR). The PSAR was prepared in accordance with the requirements of DOE Order 5480.23, and initially establishes the hazard categorization for the facility as a Hazard Category 3 (HC-3). The PSAR was developed using the approved preliminary design (Title I)

information. The approval of the PSAR followed by the subsequent approval of the Final Safety Analysis Report (FSAR) would bring OU4 into compliance to operate as a Hazard Category 3 facility.

Stated within the PSAR:

- "This *Preliminary Safety Analysis Report (PSAR) provides the safety basis for the construction of the Operable Unit 4 (OU4) Vitrification Pilot Plant at the Fernald Environmental Management Project (FEMP)." (Pg ES-1)*

- *"This document does not evaluate the initial testing of the system with non-radioactive materials or subsequent storage of materials." (Pg ES-1)*

The PSAR lists further safety analysis to be performed to support the FSAR. The list includes, but is not limited to:

- Fire Hazard Analysis per DOE 5480.7A
- Human Factors Analysis
- Final Hazards Analysis

Development of the Final Hazards Analysis

A Final Hazard Analysis was performed to conduct follow-up, systematic hazard analysis and qualitative accident analysis based on the results of the PSAR. The FHAR provides the hazard analysis required by DOE 5480.23 for the FSAR. The development of the FHAR occurred over several months due to the slow development of the facility and melter designs. The final report was issued in October, 1994. The following analyses were key in the development of the FHAR.

- Preliminary Hazard Analysis Report (PHA)
- Hazard and Operability Analysis Report (HAZOP)

The FHAR includes analysis of a leak or spill of molten glass from the melter. A failure of the melter was listed as "unlikely." As a Control/Mitigator, the report states that the design includes containment for the furnace melt. No other design detail (i.e., design capacity, configuration) of the containment is presented.

Preliminary Hazards Analysis Report

The Integrated Hazard Analysis: Preliminary Hazard Analysis (PHA) Plan is the document prepared by FERMCO Safety Analysis (SA) that establishes the schedule and scope of activities for developing the preliminary hazards analysis. The PHA Team was identified in the Plan, and the first meeting was scheduled to convene on June 17, 1994. The PHA Team consists of a designated person from each of the following organizations:

- DOE-FN
- FERMCO Safety Analysis
- FERMCO Engineering
- FERMCO RSE (PSAR Coordinator)
- FERMCO H&S
- FERMCO RSE (Start-up/Operations)
- PARSONS Safety Analysis
- PARSONS Design (Engineering)

- *"The purpose of the preliminary hazard analysis (PHA) is to provide an integrated identification of hazards associated with construction and operation to the scope of work covered by the Preliminary Safety Analysis Report."*

- *"The PHA is the first step of developing a comprehensive Integrated Final Hazard Analysis which will be the basis for the Final Safety Analysis Report (FSAR)."*

The findings of the PHA team are documented in the Preliminary Integrated Hazard Analysis (PHA) for the CRU4 Pilot Plant. The document identifies the standard hazards, safety significant items, and risks requiring additional analysis. A leak or spill of molten glass or heavy metals from furnace is listed as one of twelve hazards found to have unmitigated frequency and consequences meeting the criteria for safety significance. A melter leak/spill is considered as an unlikely event (10^{-1} per year > n ≥ 10^{-4} per year)[1] with moderate consequence (less than one fatality or five serious injuries). This classification was carried forward to the FHAR. Listed as mitigators and corrective actions for a melter leak/spill:

- Construction acceptance testing is to be performed prior to startup
- Containment is required (Bottom Drain Container)
- Project Engineer shall ensure the design includes adequate containment, dikes, and spill barriers
- Facility Owner shall ensure that the work area minimizes the storage of combustible materials in the vitrification area.
- Supervisors shall ensure successful completion of operator training and qualification for the vitrification system operation and furnace operation.
- Lessons Learned from the Minimum Additive Waste Stabilization (MAWS) Vitrification project in Plant 9 are incorporated into procedures to provide safe work practices during operation of the Vitrification Pilot Plant.

DOE-STD-3009-94, "*Preparation Guide for U.S. Department of Energy Nonreactor Nuclear Facility Safety Analysis Reports,*" July 1994, describes "***unlikely***" events to be in the frequency range of $10^{-2} \geq p \geq 10^{-4}$ which is contrary to that stated in the PHA.

Additional safety analysis required by PHA:

- Fire Hazard Analysis is required
- Human Factors Analysis is required
- Hazard and Operability(HAZOP)

Hazard and Operability Analysis Report (HAZOP)

The report closes a commitment found in the PHA to perform a formal hazard and operability analysis of the furnace, gem maker, and off-gas system. A series of design review meetings were held with the HAZOP team between October 24, 1994 and May 17, 1995. The findings from the meetings were listed as recommendations and were carried forward until resolved.

The results of the first meeting are documented in *Vitrification Pilot Plant HAZOP Number 1*. The document:

- Describes the formation of the team,
- Lists the initial recommendations of the team. Concerning a melter leak, recommendation (3) states *"Consider performing the pilot plant run without feeding the air sparger tubes into the furnace. Evaluate the degree of erosion/corrosion of the floor around the air sparger inlets at the end of the pilot plant run. The HAZOP team is concerned about the possibility of a tube breaking during an attempt to feed it into the furnace, resulting in personnel injuries or fatalities."*

  The team is concerned that operations requiring access to the bottom of the melter may present an unsafe condition. The initial design requires manual advancement of the bubbler tubes, which the team considers to be an unacceptable risk. The preliminary information from the melter vendor requires the bubbler tubes to be fed manually into the melter on a frequent basis due to corrosion losses. The scenario considered by the HAZOP team is that the fragile tube breaks while replacing, resulting in a molten glass leak while personnel are under the melter. These concerns are conveyed to the design team and the vendor.

- States that additional design information is necessary to continue the analysis. Recommends further analysis of the furnace and gem maker.

A second meeting was scheduled and is documented in Vitrification Pilot Plant Process Hazard Analysis. The document:

- Defines the need for a final report ,
- Lists additional recommendations. Recommendation (5) states: *"Consider developing a method to measure the amount of erosion of the bottom of the melter around the air sparger penetrations."*

- The team shows concern for the postulated upset from excessive corrosion of the bubbler tubes and the melter bottom. The previous recommendations (1) to development of a method to measure the amount of erosion of the bottom of the melter around the air sparger penetrations, and (2) to operate without advancing the tubes has been dropped from the list.

The third and final meeting of the HAZOP team resulted in the drafting and submittal of the final HAZOP report. The report lists 11 recommendations that are to be considered by the VITPP design team and formally resolved. None of the remaining recommendations refer to bubbler tube advancement or loss due to the corrosive environment. Closure of the previous recommendations were not documented. The final report states: *"The findings, which are listed as recommendations, were carried forward until resolved."*

No other documentation has been located with regard to and (1) the manual insertion of the bubbler tubes; and, (2) measurement of the amount of erosion. A chronology of events regarding the bubbler tubes is provided in Attachment 2.

Auditable Safety Record (ASR)

The hazard categorization and safety envelope for Phase I operations are documented in the ASR. Although the preliminary categorization of the facility is HC-3 in accordance with the PSAR, the VITPP under Phase I configuration and operation is categorized as an Industrial Facility in accordance with DOE-STD-5502-94. The hazard analysis was performed in accordance with the guidelines of DOE-STD-1027-92. The existing FHAR is referenced for those hazards applicable to Phase I including a leak/spill from the melter.

Phase I Operational Safety Guidance Document (OSG)

The purpose of the OSG is to provide additional detailed information relating to the safe operation of the VITPP facility. The document is prepared in accordance with the guidelines established in the Safety Performance Requirements Manual, RM-0021, SPR 2-1, Operating Facility Health & Safety Briefing - Visitor Access. The OSG is used in lieu of a Project Specific Health & Safety Plan (PHASP) and includes a standard Safety Requirements Matrix. VITPP operations do not require the use of a PHASP as described in 29 CFR 1910.120, Hazardous Waste Operations and Emergency Response.

Design Change Control

Requirements

Configuration Management (CM) is required to ensure the systematic evaluation, coordination, disposition, documentation, implementation, and verification of all design changes, and their impact on safety, cost, schedule, and technical baseline.

The general requirements for VITPP CM and design control are described in the Project Execution Plan for Operable Unit 4 Vitrification Pilot Plant (PEP), Section 5.5. The PEP states that VITPP activities will be performed in accordance with the FEMP Engineering Division CM procedure, 12-5004, (superseded by Configuration Management, CM-0001, April 1996) and the FERMCO Configuration Management Plan (PL-3035). The designer of the melter was responsible for the control of design changes associated with their design. FERMCO was responsible for the control of design changes associated with the Balance of Plant (BOP). The PEP also states that the performance grades for VITPP structures, systems, and components (SSC) have been established by the Pilot Plant Management Team and approved by the CM Technical Review Board.

The project-specific requirements for CM and engineering design (ED) are listed in the PEP Attachment 1, Project Specific Requirements, Section 1, Configuration Management, and Section 2, Engineering Design. The implementing procedures listed for VITPP CM are as follows:

- CM-0001, Configuration Management
- PL-3035, Configuration Management Plan
- ED-CM01, Configuration Management Sitewide Implementation Plan

The additional implementing procedures listed for ED are as follows:

- 12-4007, Drawing Preparation
- 12-4010, Design Verification
- 12-5001, Centralized Control of Project Documents
- 12-6002, Engineering Processes of As-Built Drawings,
- 12-5002, Engineering Design Change Process
- ED-12-4003, Design Criteria Package
- ED-12-4004, Design Package
- ED-12-4001, Functional Requirements Document
- ED-12-4005, Calculations, Preparation and Review Process
- ED-12-4006, Specification Preparation and Issue
- ED-12-4009, Process Flow Diagram Preparation and Issue
- ED-12-4011, Vendor Submittal for Engineered Items

Performance Grading

Performance grading of SSCs is used to assure that the level of detail required for analysis, documentation, and application of engineering principles that comply with the requirements is commensurate with relative importance to safety (Hazard Category), complexity of the activity, facility life cycle, or importance to project mission.

A Performance Grade of 3 (PG-3) or higher is generally given to an SSC deemed safety significant. In accordance with the requirements of PL-3045, an SSC is considered PG-3 if:

> *"The SSC is part of a "safety" system in a HC-3 HC-4 or Low Hazard (LH) facility and whose failure fails a preventative or mitigative function necessary to ensure that there is no unacceptable risk to the project worker."*
>
> *"The SSC's failure by itself or in combination with one or more SSCs may result in loss of function of any emergency handling, hazard recovery, fire suppression, emergency preparedness, commination, or emergency power system that may be needed to preserve the health and safety of the facility workers, collocated workers, and visitors."*
>
> *"An SSC whose failure fails a preventative or mitigative function necessary to ensure that regulatory imposed release limits are not exceeded."*

An SSC that is designated as PG-1, PG-2, or PG-3 requires independent design review during definitive (Title II) design. The Technical Review Board (TRB) will evaluate and approve the PG for the SSC. The TRB is a group of senior managers chaired by the Vice President of the Engineering Division. Engineering, construction, environmental safety and health, quality assurance, and project and information control are permanent TRB members. All design changes that affect PG-1, PG-2, or PG-3 will be reviewed by the TRB.

The VITPP Project Execution Plan (PEP) lists the following SSCs as PG-3:

- Vitrification Melter
- Melter and Gem Machine Interface Panel
- Melter Bottom Drain Container
- Carbon Bed System
- Radon Monitors
- Silo 3 Surge Bin System
- Emergency Off-gas Rupture Disk
- Emergency Off-gas Vent Valve
- Emergency Off-gas Knockout Pot Pressure Relief Valve

Design Changes

The procedures for engineering design changes were provided by ED-12-5002, Engineering Design Change Processes. In referencing potential impacts to the established safety basis, ED-12-5002 requires the cognizant Project Engineer:

> *"To perform a detailed Project Impact Assessment based on the changed parameters identifying the impact to safety documents, project baseline,*

configuration management, and/or potentially impacted functional; area requirements in accordance with:

a) If an Unreviewed Safety Question (USQ) determination needs to be conducted, the Project Engineer will provide screening per Site Procedure NS-0002 "USQD and Safety Evaluation System" of all DCNs against an approved SAR and/or other existing safety analysis documents prior to evaluation, implementation, and/or completion of the DCN."

The Site procedure NS-0002 states that:

"If a DOE approved SAR for (the) proposed activity does not exist, the change will not be reviewed within the USQD process but will be reviewed by the originating organization via the technical review process established within their department."

The PSAR for the VITPP was developed during preliminary (Title I) design, and did not provide an effective comparison for design change review because of its lack of detailed design information. Additionally, project personnel did not regard the PSAR as the correct safety basis for their current project activities. Therefore, the USQD process was not applied. This concept was assumed to be acceptable because it was planned that a detailed FSAR would be submitted for DOE approval following completion of Title II design. At that time the USQD process would be invoked. Prior to FSAR submittal, however, a review process for design changes was established using ED-12-5002. Design changes involving SSCs that were defined as PG-3 were submitted to FDF SA for additional review against the safety basis (PSAR or ASR). After issuing the ASR, all prior DCNs were reviewed by FDF SA for potential impact to the safety basis.

Melter Design Changes

The melter design was controlled by the vendor as proprietary; design control and configuration management of the melter design was performed by DURATEK. Melter design changes were handled using DURATEK's Engineering Change Notice (ECN) procedures. FDF requested and received DURATEK ECNs for informational purposes. FDF personnel did not participate in approval of ECNs. There was no disciplined practice which required distribution of ECNs to other FDF groups potentially affected by the ECNs.

Bottom Drain Container Design Control

The VITPP design includes the use of a melter bottom drain container (BDC) to capture the melter contents in the event of initiating the bottom drain system and to provide containment from a melter leak/spill. The "footprint" of the BDC was

smaller than the footprint of the Melter. Prior to the event, the refractory-lined steel container was positioned on a roller conveyor directly below the melter vessel.

As a result of the melter leak, the BDC container filled to capacity with some glass overflowing onto the furnace room floor. The volume of overflow is approximately 11 ft^3. The volume of the BDC is about 29 ft^3, which results in a total spill volume of about 40 ft^3, or 140 per cent of the BDC capacity. Additionally, calculating the melter volume from design drawings yields an estimated melter volume of 31 ft^3,which is beyond the capacity of the BDC.

After researching project records, the safety review team was unable to locate any documentation of design criteria (other than the approved drawings), that were used to provide the design basis of the BDC. According to correspondence from PARSONS to FDF dated 1/10/97 (PARSONS ID 04:085:144:0001-97):

> *"The original design basis of the Bottom Drain Container (BDC) was that it was to receive the full contents of the VITPP melter following initiation of bottom drain from all three melter chambers. The melter volume was calculated from DURATEK drawings as approximately 31.2 cubic feet (ft^3). The original capacity of the container was conservatively designed at 48 ft^3 (154 per cent of melter capacity). This design was performed prior to confirmation of all the height dimensions in the furnace room due to lack of specific design information from DURATEK. Based on the relative simplicity of the calculation, a bottom drain container capacity calculation was not formally documented."*

Therefore it is concluded that, although the BDC is identified as containment in the event of a melter leak/spill (PHA, FHAR, ASR) and is identified as a PG-3 SSC, formal design criteria (i.e minimum capacity, capture size, etc,) were not developed. Furthermore, the omission of a capacity specification was carried through three design changes, safety analysis review, and Technical Review Board evaluation.

The following discussion is provided to illustrate the control process employed for the design of the BDC.

Design Change Notice DCN68, Rev 0, Approved 10/11/95

As the DURATEK design was finalized, height and size requirements in and around the container were also firmed up. DCN CONS\1636:068 subsequently requested PARSONS to revise the drain container design per FERMCO direction (vendor drawings). This FERMCO direction resulted in a reduction in the container height of approximately 10 inches, reducing the drain container capacity to 35 ft^3 (112 per cent capacity). No formal calculation was documented during the design change. The DCN indicated that this reduced volume was

acceptable to VITPP operations. No basis for this decision is provided with the DCN.

Design Change Notice DCN CONS\1636: 068 Rev 1 Approved 1/11/96

Revision No.1 to DCN CONS\1636:068 requested that heat transfer analysis be performed to evaluate the affect on the container and adjacent equipment resulting from a bottom draining. PARSONS performed an analysis which indicated that 4" of refractory would reduce container surface temperatures to below 600°F. FERMCO resolved the DCN by directing the addition of 4 inches of castable refractory to two of the four bottom drain containers (BDCs). This would result in one backup BDC with 4 inches of castable refractory. The addition of the castable refractory into the BDC reduced the container capacity to approximately 20 ft^3 or approximately 65 per cent of the assumed melter capacity. Again, no formal calculation is available in the design change documentation.

Safety Analysis reviewed the DCN and stated:

> *"The drain container size change does not affect the safety of the operation as long as the volume is not reduced to a level below the furnace capacity. With the change, there is still adequate capacity to contain the bottom drains if the feed is promptly discontinued."*

Memorandum from Silos Project Engineering Manager to Technical Review Board (TRB) on 1/25/96 documents closure of TRB action items with regard to changes made to PG-3 components of the VITPP (M:P:(FRVP):96-0035. The memorandum states:

> *"DCN 068, Rev 00 and 01 reduces the height of the drain container to allow its movement under the gem machine heads. A layer of refractory material is added to the bottom and sides of the container to reduce thermal transfer. The drain container size does not affect the safety of the operation as the volume is not reduced to a level below the furnace capacity. With the change, there is still adequate capacity to contain the bottom drains if the feed is promptly discontinued. The refractory reduces the metal temperature adding safety to the melter bottom drain process."*

Design Change Notice DCN CONS\1636: 068 Rev 2 Approved 3/12/96

Revision 2 of DCN CONS\1636:068 requested PARSONS to recommend an alternate insulation since the material referenced in Rev 1 may absorb moisture. PARSONS states:

> *"PARSONS initially proposed the use of a 2"* (inch) *thick ceramic wool blanket. The blanket would be covered by a liner panel on the bottom of the container but would be exposed on the sides of the container. Upon filling,*

the insulation on the sides of the container would compress to ½" to provide additional capacity."

During review of the Rev. 02 PARSONS response, FERMCO Safety expressed concern that uncovered ceramic blanketing may promote airborne Refractory Ceramic Fiber (RCF) in the work area, which is a significant worker inhalation hazard. FERMCO recommends the use of a castable refractory. The material selected is a 4.5 inch thick thermal ceramics 80-D firebrick. The firebrick is comparable in size to the insulation described in Rev 1; it is assumed that Rev 2 would have resulted in a capacity of approximately 20 ft^3. A volume calculation was not documented in the design change.

Design Change Notice DCN CONS\1636: 068 Rev 3 Approved 4/9/96

The firebrick is unavailable, FERMCO needs an alternate means of protection. In addition the FERMCO DCN Inquiry states:

"The two outer bottom drains appear to be close to the east/west edges of the drain container. Is there a potential problem here?"

The effective height of the container is increased by 3 inches by adding a vertical segment. In addition to the height addition, a 6 inch angled deflector is added to the top east/ west sides of the container. The intent of the deflector is to increase the capture "footprint" of the container. Additionally the 4.5 inch thick firebrick is replaced with 2.25 inch thick castable refractory. The capacity of the container has been increased to 29 ft^3 or about 93 per cent of full melter capacity. Although a formal calculation has not been retrieved, notes from a meeting held on 4/3/96 indicate that calculations were performed. Both PARSONS and FERMCO recognize that the current design results in an under-capacity condition. Further discussions indicate that down sizing the refractory to a thickness of 2 inches with the height change described above would provide full capacity. A formal volume calculation is not available in the design change documentation.

Safety Analysis reviews DCN 068, Rev 03 and responds on 4/6/96:

"I reviewed DCN: 068.3 and there is no impact to the safety basis."

Memorandum from Silos Project Engineering Manager to Technical Review Board (TRB) on 4/19/96 documents closure of TRB action items with regard to changes made to PG-3 components of the VITPP (M:P:(FRVP):96-0207).

- *"DCN 068, Rev 02 and 03, changed the material used to line the interior of the drain container for reduction of thermal transfer. The castable material, per Rev 01, was deleted due to the possibility of moisture adsorption and unacceptable performance. Ceramic firebrick, per Rev 02, was not available to meet our schedule."*

- *"The Hi-Alumina castable material with ceramic paper, per Rev 03, is available and a cost savings from other materials. It reduces the container metal temperature adding safety to the bottom drain process. Deflector shields to the sides of the container were added as a safety feature to assure proper glass flow."*

Off-Normal Scenarios and Test Corrections

Materials Performance Evaluation

Safety documents, procedures and the test plan were reviewed to determine what materials, i.e., refractory, electrode material, bubbler material, etc., had been identified for monitoring. What monitoring occurred and what were the results? Should additional materials have been identified? If materials had been identified for monitoring and the monitoring did not occur, what was the reason?

Erosion and corrosion of melter refractory material was identified in the test plan to be monitored by measuring the difference in chromium concentration between the feed material and the glass. Relatively high concentrations of chromium would indicate excessive erosion/corrosion. It is not apparent this monitoring occurred.

Redox potential of the melt was determined to be an important factor, but was not monitored.

Molten metal (resulting from redox reactions) on the bottom of the melter was recognized as a possibility. It was anticipated that it would be difficult to detect but was not monitored.

There were no test specimens (coupons) such as refractory, bubbler or electrode materials , in the melter for monitoring. This was discussed and dismissed because of the planned short time of operation for Phase 1.

The test plan defines coupons and their use in the glass bath to test the corrosive and/or reactive impacts of the molten glass. But coupons were not used in the melter. In summary, there were no specific actions taken to monitor erosion or corrosion of materials in the melter.

Phase I Test Plan Operations

The Phase 1 Test Plan, 18-SU-0003 Rev 2, was reviewed to determine if it addressed issues such as redox, contamination of the side chambers, erosion/corrosion of refractory, breached electrodes, metal precipitates, and erosion/corrosion of bubbler tubes or a leak from the bottom of the melter, as well as the recommended steps to mitigate those issues. Were the recommended steps translated into procedures? In either case, were the recommended steps followed?

The test plan addressed the following issues noted above as follows:

- **Redox** The expected condition as noted in the Test Plan was oxidizing, evidenced by amber brown to black glass color. If a reducing condition existed, as evidenced by green or blue color, the response described in the test plan was to adjust glass formula. This was not translated to procedures or otherwise implemented.
- **Contamination of side chambers** This condition was discussed and it was noted that contamination would result in accelerated electrode erosion or corrosion. Procedures addressed the need to maintain glass level in the side chambers.
- **Erosion/corrosion of electrodes** This condition was discussed and it was determined that this condition could be caused by center chamber glass finding its way into the side chambers. Adding frit to side chambers to maintain side chamber glass level higher than center chamber was the recommended action. This action was in procedures and followed, however because of the cracks in one of the side walls, the center chamber glass did migrate into the adjacent side chamber, resulting in extensive electrode erosion/corrosion in that side chamber.
- **Metal precipitates** This condition was discussed, noted as difficult to detect, but metal precipitates were expected to remain suspended with adequate agitation. No procedural guidance existed which addressed metal precipitates or their collection on the bottom of the melter.
- **Bubbler tube erosion/corrosion** was not addressed in the test plan.
- **A leak from the melter** was not addressed in the test plan.

Response to Off-Normal Conditions

The following safety documents and procedures were reviewed for definition of off-normal conditions:

- PSAR - Rev 0, April 1994
- OSG Rev 3, December 1996
- PHA, July 1994
- FHAR, September 1995

- ASR, March 1996
- HAZOP, September 1995
- Fire Hazards Analysis Update, February 1996

The OSG addresses the following: radiant heat from the melter, burns from the melter, viewing molten glass through didymium lens, electrical hazards from the melter. A leak of molten glass was not addressed.

The PHA identified a leak from the melter, determined it to be an "unlikely" event with "moderate" consequences, mitigated by a containment box under the melter. This evaluation was carried forward to the FHAR (see below). It is not clear if the design at this time included bubbler tubes.

The FHAR reported six safety-significant hazards, one of which was a leak/spill of molten glass or heavy metals from the melter. The engineered control to mitigate this hazard was the containment box under the melter. There were no administrative controls specified.

- The leak/spill was determined to be an "unlikely" event with "moderate" consequence.
- The melter design was not modified to mitigate this hazard.

The PSAR addressed a leak from the melter, with the same probability of occurrence and consequences as the PHA.

- It is not clear if the design included bubbler tubes.
- The leak/spill was determined to be an "unlikely" event with "moderate" consequence.
- The cause of a leak/spill was noted as a material defect or worker error during installation. The design includes a containment.

The ASR noted that the FHAR identified a leak/spill hazard and mitigated it with a containment vessel under the melter.

The HAZOP identified a leak from the melter as a possible deviation and noted that "no safety consequences were expected".

The Fire Hazards Analysis Update (FHAU) identified the possibility of a fire in the melter room caused by combustible materials contacting the hot surfaces. The mitigator was good housekeeping. The FHAU called for an emergency response drill at the VITPP which was conducted both prior to and during the readiness assessment.

In summary, there were several safety documents that recognized the potential for a leak in the melter. The probability of occurrence was determined to be "unlikely" with "moderate" consequence. The basis for these determinations is unclear. The mitigation for the leak was the containment box under the melter.

Problem Identification and Resolution

During VITPP melter operations, unusual occurrences were identified by project personnel. In this Section, an operational problem with the VITPP melter is described, and the actions that were taken to respond to the problem are discussed. This is not meant to relate this specific problem to the VITPP melter failure, but to provide an example, in general terms, of how the project identified and responded to a VITPP melter problem.

A "glow" was noted at Bottom Drain No. 3 on September 24, 1996. The following discussion indicates the problem-solving approach that was taken to address the "glow".

Periodic measurements of melter electrical parameters were started; separate cooling was established to bottom drain No. 3; the temperature of the discharge chamber was reduced to monitor the effect on the "glow"; the current-to-ground was measured; photo infrared imaging was performed to look for hot spots. The glow continued while these activities were ongoing.

A meeting was held with DURATEK on October 10, 1996. The actions resulting from that meeting are summarized as follows:

- Perform a baseline thermal imaging
- Cooling plates were to be selected to cool the melter outer shell on the south side and the sides of the discharge chamber
- A boroscope was to be used to look at the bottom drain
- The fabrication of a new bottom drain sleeve with larger tubing for greater flow was to be completed.

Problems with cooling to all three bottom drains were documented. The "glow" apparently continued.

On December 16, 1996 a pile of molten metal was observed on top of the coupling on bottom drain No. 3. The metal was removed and sent to the lab for analysis. On December 19, 1996, the lab results indicated that the metal in question was 58 per cent lead. This scenario was developed through a review of shift logs, which indicates that operational problems were typically documented and actions were taken in an attempt to solve each problem.

Response to Incident

Description of Incident

On December 26, 1996 at the Fluor Daniel Fernald Environmental Management Project Vitrification Pilot Plant, a breach occurred in the bottom of the melter, causing the entire contents of non-radioactive molten glass to leak from the melter. The majority of the molten glass flowed into a bottom drain container; however, a small stream deflected onto an epoxy coated concrete floor causing a fire. Emergency Response personnel put out the fire with water. There were no personnel injuries and there was no on-site or off-site environmental impact from the incident.

This incident, categorized as an ***Unusual Occurrence***, was reported in the Fernald Environmental Management Project Occurrence Report, OH-FN-FDF-FEMP-1996-0075 Vitrification Pilot Plant Melter Breach, dated December 27, 1996, and Initial Update dated January 14, 1997.

Discovery of Melter Breach

At approximately 2222 on December 26, 1996, glass was discovered leaking from the bottom of the melter at the FDF VITPP. A maintenance supervisor had entered the melter room in response to a system engineer's request for instrument mechanic support. A camera had recently been adjusted by an Instrument mechanic and the melter operator, who was located on the second floor of the melter room, noticed a wisp of smoke in the vicinity of the camera. When the maintenance supervisor entered the melter room on the ground floor, she noticed a small stream of molten glass flowing from the bottom of the melter.

Initial Conditions

Prior to the incident, the VITPP was in a normal IDLE operating mode with no slurry feed and no glass discharge operations in progress. Operations were being conducted in support of developing vitrification technology while processing surrogate test materials. The material in the melter contained some concentrations of barium and lead. The material was not radioactive. The complement of shift personnel included nine VITPP team members, six maintenance personnel, and one radiological control technician. The system engineer, the melter operator, and another plant operator were in melter room when the maintenance supervisor entered the melter room.

The melter and support systems were in a normal equipment lineup for the operations in progress. Equipment and instrumentation that were out of commission or in reduced status did not impact any aspects of this incident.

Equipment and instrumentation alarm status prior to the incident did not impact the incident.

Immediate Actions

The maintenance supervisor immediately reported the molten glass leak, the size of a pencil stream, to the system engineer who was on the second floor of the melter room. At approximately 2223 the system engineer directed the control room operator to ramp-down (decrease to zero output) power to the silicon control rectifiers (SCR) No. 1 and No. 2 to electrically shutdown the melter. The system engineer then notified the shift manager, who was located in the operations support trailer, T-121, adjacent to the plant, of the incident via radio. The maintenance supervisor notified fire and safety personnel via radio that smoke was present in the melter room and that no fire was observed. Smoke was being generated from a thin plastic sheet covering the top of the bottom drain container. At 2228, the communications center received the first smoke alarm from the VITPP. The communications center operator immediately notified the assistant emergency duty officer (AEDO). The system engineer had directed all personnel in the melter room to stand by the exit to the room on the first floor while the shift supervisor manned the control room. Several minutes after the melter breach, the hole widened to approximately two inches; a cable deflected a small stream of molten glass from pouring directly into the drain container located under the melter. The deflected stream of molten glass poured onto the epoxy-coated floor; the floor ignited. The shift supervisor immediately telephoned the Communications Center that there was a fire and that emergency assistance was required.

The shift manager took charge at the scene after arriving in the melter room, immediately after being notified of the breach. He directed a maintenance electrician on scene to open the local electrical disconnect to SCR No. 1, located in the melter room in the near vicinity of the fire. The maintenance electrician expended a carbon dioxide (CO_2) fire extinguisher attempting to control the flames and put out the fire. The shift manager used a water hose to partially divert the glass pour stream back into the bottom drain container. The water nozzle was then directed to cool and "freeze" the glass steam. This attempt was abandoned when it was determined to be ineffective in reducing flow from the melter. Unable to control the leak or the fire and with smoke accumulating in the melter room, the shift manager directed all personnel to leave the melter room. At approximately 2234, the shift supervisor in response to the shift manager directed the control room operator to initiate an emergency shutdown of the VITPP by depressing the emergency stop button, and ordered the evacuation of all personnel from the building. Two maintenance electricians opened the main electrical disconnects to SCRs No. 1 and No. 2 to electrically isolate the melter prior to their evacuation.

As the molten glass continued to flow from the melter, the glass that had been deflected to the concrete floor had spread to an area of about two feet square. A second portable fire extinguisher (dry chemical) had been used unsuccessfully to put out the fire. Emergency response personnel arrived on the scene by 2240 and put out the fire by flooding the floor with water. Approximately one cubic yard of surrogate glass discharged from the melter to the bottom drain container and approximately 20 per cent of the glass was deflected onto the floor.

Following plant evacuation, an accountability of all shift personnel was performed and all personnel were accounted for. Some key personnel were directed by the shift manager to assist with the control and recovery action. The operations manager had been telephoned and informed of the incident by the shift manager.

At the time of the glass spill from the VITPP melter, a MDA Scientific 7100 monitor was operating to measure general area sulfur dioxide (SO_2) levels. The monitor was located on the west side of the third level of the melter room. Levels measured by the monitor indicated the 1-minute average, 15-minute short-term exposure limit (STEL), and 8-hour time-weighted average (TWA) levels of SO_2 from December 16, at 19:37 hours through December 26, 1996, at 14:24 hours were 0.0 ppm, as printed out on 8-hour summaries. Also, the 15-minute STEL and 8-hour TWA levels of SO_2 from December 26, at 14:24 hours through December 26, 1996 at 22:24 hours were 0.0 ppm, as printed out on an 8-hour summary. (See Attachment 3 for the data.)

When measured SO_2 levels exceed a pre-set alarm criterion level of 2.5 parts per million (ppm), the monitor sounds an external alarm (Westminster clock electronic chime), and prints on thermal paper the average level approximately every 25 seconds. These alarm printouts continue until the measured levels no longer exceed the alarm point. After each printout, the indicating tape is advanced and monitoring resumes with freshly exposed tape.

The alarm printout started at 22:23 hours on December 26, 1996. There were 34 alarm printouts recorded on the thermal paper before an optics fault condition stopped the monitoring. A review of the data and indicating tape has concluded that SO_2 levels in the exposure area were indeterminate. The measured SO_2 levels indicated on the printouts are not representative of SO_2 concentration for the following reasons:

- The entire indicating tape, not just the spots where air is drawn through, was discolored pink. This may have been caused by acid gases, water, and/or heat. The thermal paper used by the monitor's printer also was darkened.

- The monitor measures reflected light from the indicating tape which develops a pink stain from SO_2 reaction with the chemicals impregnated into the tape. There were 34 dark gray/black spots present on the tape where soot augmented the normal pink color stain. The monitor's detector was not able

to distinguish darkening caused by ordinary particulate from that caused by SO_2.

- All but four of the readings are outside the calibrated range of the monitor, which is 0.1 to 20.0 ppm.

- The SO_2 monitor was observed to have a powder baked onto all of its exposed surfaces, and some interior surfaces as well. This is believed to be due to the discharge of the dry powder fire extinguisher.

While the monitor cannot provide any valid exposure data on SO_2, its internal crystal controlled clock does indicate the time (December 26, 1996 at 22:23 hours) when smoke started rising to the third floor of the melter room. The section of indicating tape with dark grey/black spots was sent to a laboratory facility for metals analysis and approximate concentrations calculated, since both the flowrate (0.5 liters per minute) and sample time (approximately 15 seconds) are known. The metal fumes are expected to rise and concentrate in the vicinity of the monitor (west side of the third floor in the melter room), given the loss of building ventilation and air buoyancy due to heat. This location was not occupied during the casualty; therefore, the results will not be useful in indicating potential worker exposures. Initial responders were interviewed and no workers reported any physical distress after the incident.

Following the event, individuals determined to be potentially exposed to airborne lead were directed to FDF medical services for evaluation. In accordance with OSHA §1910.1025, Appendix C, Medical Surveillance Guidelines, a sample of the affected employee's whole blood was analyzed for levels of lead and zinc protoporphyrin (ZPP)[2]. Medical services reports that the blood lead levels for the initial responders were well below the action level of 40 micrograms per deciliter (μg/dl), with no abnormality in ZPP. Most lead levels were found to be around 1 μg/dl. These results indicate that employees were not exposed to lead fume levels that would adversely affect blood lead levels.

Incident Chronology

Times were determined from event reports, interviews, and radio recordings and instrumentation where available.

The enzyme ferrochelatase is inhibited at low blood lead levels. Inhibition of ferrochelatase leads to increased erythrocyte protoporphyrin (FEP) in the blood which can then bind to zinc to yield ZPP. There is an exponential relationship between blood lead levels greater than 40 mg/100g and the associated ZPP level, which has lead to the development of the ZPP screening test for lead exposure.

December 26, 1996

1800 Normal operations were in progress with the plant in the normal IDLE operating mode.

2155 Melter power was increased to 10 KW to raise the temperature in preparation for a scheduled SCR No. 1 outage. The outage was necessary to investigate a glowing of the melter bottom drain.

2205 Instrument mechanic discussed the west camera adjustment with the maintenance supervisor after cleaning the lens and manual picture adjustment.

2215 Melter operator called for shift manager, and described a faint indication of smoke in the vicinity of the west camera. System engineer responded for the shift manager.

2217 Maintenance supervisor was notified by the system engineer to have the instrument mechanic give the west camera a second look and check for possible damage.

2222 Housekeeping activities were in progress on the second floor of the melter room with the system engineer on the second floor in the vicinity of the west camera.

Maintenance supervisor entered the melter room from the personnel protection equipment (PPE) room and observed and reported a glass discharge from the bottom of the melter to the system engineer.

Shift manager, shift supervisor, test coordinator were located in operations support trailer, T-121. System engineer, melter operator, and a second operator, were in the melter room. The DCS operator and a second operator were in the control room. An auxiliary operator was in the VITPP pad area.

2223 System engineer notified DCS operator to ramp-down power to SCR No. 1 and SCR No. 2. System engineer radioed the shift manager.

2224 All shift team supervisory personnel met in melter room (shift manager, system engineer, shift supervisor, maintenance supervisor, and test coordinator).

2225 Maintenance supervisor notified FS by radio that there was smoke but no fire visible at the VITPP melter room. Shift supervisor proceeded to the control room.

2228 Fire alarm system automatically initiated. Communications center operator notified AEDO. (Fire alarm was electronically logged at 2225. All other times in this chronology have not been transposed to preserve best overall description and sequence of events.)

System engineer directed all personnel in the melter room to stand by the exit to melter room on the ground floor, near the PPE room door.

2229 Shift supervisor called communications center control from the VITPP control room and reported the fire alarm and smoke from melting plastic beneath melter. Control responded that FS is being dispatched.

Pencil sized molten glass stream continued leaking from the bottom of the melter. Smoke was coming from a hole burned in the plastic sheet draped over the bottom drain container. No evidence of fire.

Shift manager telephoned the operations manager to inform him of the situation.

2231 Maintenance supervisor radioed fire and safety to dispatch the fire truck.

A portion of the glass pour stream continued to be deflected by a cable beneath the melter causing a small stream to now flow directly onto the epoxy coated floor. This area of the floor, approximately two feet by two feet, ignited. Maintenance electrician expends a CO_2 fire extinguisher in attempt to put out the fire and cool the glass.

2232 Shift manager discharged a dry chemical fire extinguisher and directed the maintenance electrician to open the local electrical disconnect on SCR No. 1 cabinet.

Shift manager used water hose to successfully divert the deflected glass steam back to the bottom drain container. Shift manager attempted, with no success, to cool (freeze) the glass stream by directing water hose discharge to vicinity of the leak.

2233 Shift manager abandoned attempt to cool the glass.

2234 Shift supervisor directed DCS operator to initiate VITPP shutdown using the emergency stop button and directed all personnel to evacuate the building and to proceed to the vicinity of trailer, T-92, for accountability and further direction. System engineer directed and had two electricians open the main electrical disconnect to SCR No. 1 and SCR No. 2 in the motor control center (MCC) room.

2240 Personnel accountability completed. All personnel accounted for.

Emergency response personnel arrived on scene. Supervisory personnel and a maintenance electrician provided situational turnover including electrical power isolation status to the fire chief.

FS put out the fire and cooled the glass by flooding the melter room floor with a fire hose. Fire hose water directed at remaining molten glass coming from the hole at the bottom of the melter.

The entire molten glass contents, approximately one cubic yard, leaked from the melter. The melter contained non-radioactive, surrogate material. Approximately 20 per cent of the molten contents was deflected to the floor.

2300 Special controls established for shift turnover.

2330 Operators pump water from the melter room floor to the pad area. Water samples taken at the building sump tank prior to discharging to the high nitrate tank (HNT).

Lock and tag initiated to establish safe boundaries for investigation.

The event was initially classified as an Off-Normal Occurrence and upgraded to an Unusual Occurrence following a critique early the next morning.

Safety and Environmental Concerns

Personnel exposure to high voltage, fire, smoke, heat, and molten glass were the hazard concerns during and following the incident. Supervisory personnel promptly addressed these concerns. The system engineer immediately reduced electrical power which was providing heat to the melter. Supervisors kept personnel clear from the molten glass and immediately requested assistance from emergency response personnel at the outset of the fire. Within a few minutes after the start of the fire, a determination was made that fire extinguishers and a water hose were ineffective in combating the fire, and emergency shutdown of the VITPP was initiated, and the building was evacuated. Emergency response personnel promptly extinguished the fire and cooled the molten glass. Exposures to high voltage and heat were mitigated by plant design, including metal screens around the perimeter of the melter and a locked cage in the vicinity of transformers and exposed electrical connections. Fire protection and suppression are installed in the facility.

The environmental objective for the VITPP treatability testing was to ensure off-site protection of human health and the environment during the vitrification campaigns. The degree to which the design and operation of the plant met this environmental objective during Campaign 4, when the inadvertent melter draining occurred, can be determined by evaluation of compliance with the environmental regulatory requirements, as well as the application of environmental ALARA concepts and the degree of waste minimization that was achieved.

Phase 1 Campaign 4 blended glass additives and chemicals to create a surrogate to simulate a blend of Silo 1 and 2 residues. During Campaign 4, lead and barium compounds were added to the surrogate. These chemicals, in addition to

chromium leaching from the melter refractory, were the only hazardous substances present in significant amounts in the melter at the time of the event. No radionuclides were present in the surrogate or feed additives during Campaign 4.

The potential environmental pathways for release of hazardous constituents from the melter included the following:

- Air - Particulates and gases
- Water - Wastewater generated during cleanup
- Waste - Glass and miscellaneous material.

A discussion and evaluation of these potential environmental pathways is presented in the ***Results of Investigation*** section of this report. A calculation to provide "***An Estimate of Exposure Rate and Airborne Radioactivity Concentration from a Postulated Failure of VITPP Melter Containment***" is in ***Attachment 4.***

No environmental impacts resulted from the bottom draining event. The amount of oxides of sulfur (SO_x), oxides of nitrogen (NO_x), lead, barium, and chromium released without control is not able to be quantified. The amount of toxic material available for release to the environment, however, should be very low due to the nature of the released material (glass which quickly solidified), elimination of the source of SO_x and NO_x, binding of the metals into the glass matrix, and the short contact time with the cooling water.

Weather conditions had no impact on the incident.

Related Facts

Management and Operations Organization

The management and organizational structure is provided in Attachment 5. The operations organization was established in accordance with Site Operations Procedure OP-1001, "Operations Organization and Administration", as amplified by the Operations Manager's, "Standing Orders for Vitrification Pilot Plant Operations," M-134.

The pilot plant staffing included assigned management personnel and operating shift teams, and available specialist personnel to provide operations support. Four operating shift management and supervisory teams were staffed to support continuous (24 hours per day) pilot plant operations under various plant conditions.

The following project management positions have responsibilities related to pilot plant operations: project manager, deputy project manager, safety project

manager, engineering manager, operations manager, testing/data manager, and facility owner.

The operating shift team staffing requirements varied with plant conditions and were based on plant operating modes of RUN, IDLE, and STANDBY. Operations supervisory personnel were trained and all operators were fully qualified for the operations being conducted. The shift team working at the time of the incident had approximately seven months operating experience in the pilot plant commencing with bakeout in may 1996. The vitrification pilot plant project successfully completed a formal readiness assessment in may 1996. The areas of conduct of operations and training had been evaluated as excellent programs.

Conduct of Operations

A formal conduct of operations program was established in accordance with site policy and site operations procedures to ensure safe and reliable operations. Standing Orders provided amplifying guidance in implementing conduct of operations principles in the VITPP using a graded approach consistent with project complexity. The program emphasized operational safety, procedural compliance, positive control of the plant, and monitoring and recording plant conditions. Operations personnel had completed formal conduct of operations training. The program had been evaluated and upgraded through continuing self assessments, the conduct of a drill program, and the performance of a Readiness Assessment.

Training and Qualification

A formal training program was established for Phase I VITPP Operations and promulgated in the "Operable Unit (OU4) Vitrification Pilot Plant Project Phase I Training and Qualification Program Description," 25-TP-001. Workers were trained or briefed on the specific hazards in the work area and the procedures applicable to their work. The training program consisted of formal training including basic technical knowledge, plant specific training, and individual completion of training evaluation standards. Additional hands-on training was gained during construction of the plant and support systems, and through operational support for system operability tests.

Operations personnel had received formal training in conduct of operations. All shift teams completed operational, emergency, and casualty training during a formal drill and evolutions training program conducted in the months leading to the conduct of the readiness assessment in may 1996. Maintenance and emergency response personnel had participated in the performance of selected drills. A drill entitled, "Fire in the Melter Room" which required electrical isolation had been performed on several occasions by operations, maintenance, safety and health, and emergency response personnel.

At the time of the incident, all management and supervisory personnel had completed required training and all operators were fully trained and qualified for the duties to which they were assigned. The shift team had operated the plant for approximately nine months including startup testing, melter bakeout, and phase I operations. A low turn-over rate existed in the shift management staff, supervisors, and operators assigned to the VITPP.

Operating, Emergency, and Casualty and Alarm Response Procedures

Plant operating procedures existed for the melter and support systems. Plant specific emergency procedures existed for the VITPP "Loss of Electrical Power" and for "Plant Emergency Shutdown." VITPP alarm response procedures were developed for the Data Acquisition and Control System Alarms and for the Melter Control Panel Alarms and Gem Machine Control Panel Alarms.

Where site emergency procedures were applicable to the VITPP, project specific procedures were not developed. Site procedures pertinent to this incident included the "Building Emergency Procedure," EM-0020 which addressed action to be taken in event of fire and hazardous material spills, and the "Spill Incident Reporting and Clean-up", EM-0004.

No specific plant emergency procedure was developed to address a breach in the bottom of the melter. Based on interviews, this decision appears to have been related to supporting information in risk assessments and the what-if analysis Attachment to the ASR. A furnace leak caused by materials degradation was not listed as a possible cause of failure in the what-if analysis. One of the corrective action/mitigators in the event of a furnace leak was listed as emergency preparedness/spill response. A design mitigator was the bottom drain container.

No plant emergency procedure specifies actions to be taken in the event of a molten glass spill. Based on interviews, it was determined that management and supervisory personnel training conducted at the Vitreous State Laboratory (VSL) stated that fire extinguishers and water could be used to cool and possibly stop (freeze) a molten glass spill. This off-site training information was not incorporated in procedural guidance. The site spill procedure does not specifically address actions related to cooling of molten glass.

Emergency Preparedness

Fluor Daniel Fernald is directly responsible for off-normal occurrences and associated emergency actions. Site emergency preparedness and spill response procedures are to be followed in the event of fire or hazardous spill. In the event of an off-normal event or spill, the AEDO is notified and the appropriate compensatory measures are determined and implemented.

Evaluation of Shift Team Response

This section of the report provides an evaluation of the appropriateness of the incident response by the Shift Team. Supervisory personnel were presented with a complex casualty involving the following:

- A breach of the melter
- Spill of hazardous material
- Fire
- Heat
- High voltage.

The immediate action by the team required the application of appropriate site emergency procedures and VITPP project specific procedures. The complexity of the casualty required supervisory personnel to render prompt decisions and take appropriate casualty control actions to simultaneously combat multiple casualties with due regard for personnel safety, plant and system integrity, and environmental considerations. These procedures included the following:

Site Procedures

EM-0020 *"Building Emergency Procedure"* (Sections for Fire and Hazardous Material Spills.)
EM-0004 *"Spill Incident Reporting and Clean-up."*
EM-0010 *"Event Notification and Reporting."*

VITPP Project Specific Procedures

11-A-001 *"Vitrification Pilot Plant Data Acquisition and Control System Alarms"* (Sections ZS-002 Fire Alarm and ZS-004 Emergency Shutdown Alarm.)
11-E-002 *"Vitrification Pilot Plant Emergency Operating Procedure Emergency Shutdown."*
M-134 *"Standing Orders for Vitrification Pilot Plant Operations."*

It should be noted that there were no procedures developed by the project to address a breach in the bottom of the melter or a leak of molten glass. Information regarding this decision is presented in the section of this report

entitled ***Operating, Emergency, and Casualty and Alarm Response Procedures.*** Furthermore, there was no specific procedure to address loss of containment when the bottom drain container was unable to contain the spill of molten glass. This molten glass which was diverted by a cable and that which overflowed from the drain container was treated as a hazardous spill.

An evaluation of the specific actions required by the applicable site emergency and project procedures versus the actions taken by the shift team was conducted by this SRT. ***The actions taken to combat the casualty were evaluated as timely and appropriate.*** Supervisory personnel used good judgement both in their immediate actions to combat the casualty in the melter room and in promptly calling for emergency response assistance and evacuating all personnel from the plant when they evaluated their firefighting actions as ineffective. The system engineer's prompt decision to ramp-down SCR power and to have personnel stand clear was excellent. While the shift manager's decision to divert and attempt to cool (freeze) the molten glass was based on training received at VSL, this action had not been promulgated in any project procedure such as a molten glass spill procedure. Notifications were made in a timely manner. Several actions required by procedure were not taken. However, there was no significant effect as a result of not completing these actions. The actions not taken included the following:

- The fire box was not pulled. The fire was reported to the communications center control and the fire alarm was automatically initiated as a result of smoke in the melter room.
- Roll-up doors were not closed following evacuation. While not a significant factor in this particular incident, this action was contrary to general firefighting practices.
- Ventilation was not secured in the control room. The shift manager made the decision that this action, had it been taken, would have delayed evacuation.
- Personnel did not evacuate to the rally point. The shift supervisor directed personnel to proceed instead to the vicinity of trailer T-92 to facilitate establishing personnel accountability status and to render assistance if required.
- Personnel exposed to smoke did not immediately report for medica evaluation. While there was very limited exposure to smoke, it would have been prudent to seek medical evaluation. This was especially important because of the potential of airborne lead. Subsequent blood tests by medical indicated that there was no exposure to lead.

The overall evaluation of the appropriateness of the response by the shift team is that both supervisory, operators, and support personnel were highly effective in combating casualty. The operational discipline instilled by training and a strong conduct of operations program were significant factors which resulted in no personnel injuries and minimum material damage.

Results of Investigation

Overall Assessment

The execution of this project by FDF was in accordance with requirements established by the DOE. Thorough scientific and engineering discipline was not exercised throughout the duration of the project. Continuous attention to potential safety concerns due to the degeneration of the melter components was not effectively factored into the revisions of the safety basis documents, procedures and test plans. The discipline gained from all aspects of operational experience and personnel training ensured that the incident resulted in no personnel injuries.

Environmental Impact

The purpose of this section is to assess the environmental impacts due to the inadvertent melter bottom draining event on December 26, 1996, and identify deficiencies in system design or operating procedures, including emergency response, related to the event.

The environmental objective for the VITPP treatability testing was to ensure off-site protection of human health and the environment during the various vitrification campaigns. The degree to which the design and operation of the plant met this environmental objective during Phase I, Campaign 4, when the inadvertent melter draining occurred, can be determined by evaluation of compliance with the environmental regulatory requirements, as well as the application of environmental ALARA concepts and the degree of waste minimization that was achieved.

Phase I Campaign 4 involved the use of glass additives and chemicals to make up a surrogate feed to simulate a blend of Silo 1 and 2 residues. During Phase I, Campaign 4, lead and barium compounds were added to the surrogate. These metals, in addition to chromium leaching from the melter refractory, were the only hazardous substances present in significant amounts in the melter at the time of the event. No radionuclides were present in the surrogate or feed additives during Campaign 4.

Release Pathways

The potential environmental pathways for release of hazardous constituents from the melter included the following:

- Air - Particulates and gases
- Water - Wastewater generated during cleanup
- Waste - Glass and miscellaneous material

Environmental data taken during this part of Phase I, Campaign 4 included sampling of the air, water, and solid waste. A summary of environmental data is presented in Section 7.0 of the Phase I, Campaign 4 Report.

Air

Since the most recent addition of feed to the inventory of the melter was on 12/24/96, the glass had adequate opportunity to degas most of the SO_x and NO_x that would have been generated from the sulfates and nitrates, and release low boiling metals as aerosols to the melter off-gas treatment system prior to draining. Therefore, residual toxic gas and particulate generation from the melt pool released from the melter would be expected to be minimal. The melter would have maintained negative pressure during draining, which would have precluded release of melter off-gas into the melter room. As indicated by analysis of the filter samples taken from the SO_2 monitor located above the melter, gases and particulates were released during the melter draining event into the melter room. Based on the analytical data taken from the filters, lead concentration in the air above the melter reached 93 micro grams per cubic meter ($\mu g/m^3$) during the bottom drain event.

Molten glass released from the melter reached the melter room floor where it ignited the epoxy coating on the concrete. The fire was extinguished with potable plant water from a fire hose. The roll up doors to the melter room were open at the time of the draining. This could have allowed some smoke and fumes from the melter area to escape to the environment.

Water and Waste Water

Wastewater generated during the melter draining consisted of water used to try to cool and stop the flow of molten glass out of the melter, and water used to extinguish the fire and prevent the melter glass on the floor from flowing into the sump drain. According to the incident report prepared by FS, approximately 600 to 700 gallons of water were used to extinguish the fire. Wastewater generated in the area of the melter flowed into the sump drain, where it was pumped to the building sump tank (BST). From the BST, the wastewater was pumped to the high nitrate tank, where it blended with site wastewater before being fed to the

advanced waste water treatment (AWWT) plant for additional treatment prior to discharge under the site National Pollution Discharge Elimination System (NPDES) permit.

A portable pump was used to transfer wastewater from the melter room sump to the VITPP pad sump, where it was transferred to the BST. Since the portable pump was limited in capacity to 30 gallons per minute, and since there were no berms on the floor of the melter room, some of the water used to cool the glass on the melter room floor may have reached the roll up doors and been released off the concrete pad to the surrounding soil. No estimates of the quantity of water released in this manner are available. Wastewater was sampled in the BST on numerous occasions following the incident. Although not all wastewater analytical data are yet available, preliminary data from the BST taken during this period indicate the wastewater could not have exceeded the Toxicity Characteristic Leaching Procedure (TCLP) limits for Resource Conservation and Recovery Act (RCRA) constituents of concern.

Part of the slurry feed for Batch 10 had been prepared, and was waiting to be fed into the melter at the time of melter draining. Although lead and barium had not yet been added to the batch, a small amount of lead and barium from the residues from Batch 9 were mixed with Batch 10. This batch of slurry, along with heel and slurry tank washout water, was removed from the slurry tank to a polyethylene temporary holding tank where it was allowed to settle. The supernatant liquid was decanted and processed through the BST. The thickened sludge that remained was containerized and is currently staged on the covered concrete pad east of the melter building, pending further characterization.

Glass and Miscellaneous Waste

The glass that drained from the melter completely filled the containment vessel. Approximately 1,200 pounds of molten glass either overflowed the container, or was inadvertently diverted from the container, and reached the floor of the melter room. After cooling, the solidified glass was removed from the floor and containerized.

Samples of glass removed from the floor, and the glass in the primary container have been sampled. Each container of glass was characterized for metals in accordance with the TCLP. Regulatory TCLP-based limits reported in units of milligram per liter (mg/l) for barium, chromium, and lead are as follows:

- Barium . . . 100.0 mg/l
- Chromium . . 5.0 mg/l
- Lead . . . . . 5.0 mg/l
- Selenium . . . 1.0 mg/l.

Analytical data from the glass removed from the floor indicated that it contained an average of 2.94 weight per cent (wt%) of barium oxide , and 5.63 wt% lead oxide. Barium and lead oxides in the glass in the primary container were 3.07 and 5.53 wt% respectively. Data on center chamber glass taken directly from the melter prior to the melter incident indicated that barium and lead oxides were present at 3.43 and 6.50 wt% respectively. Additional characterization for TCLP will be done on the glass that drained. Glass that passes the TCLP will be managed as solid waste; glass that exhibits a RCRA characteristic will be managed in accordance with the RCRA regulations.

Miscellaneous waste was characterized by process knowledge, or by sampling on a case by case basis, as required, to determine proper management.

Summary of Environmental Impact

The lead and barium added to each batch of glass was monitored to enable a mass balance accounting on the glass for two reasons: (1) To determine the degree of partitioning of the metals into the glass, (2) To ensure that the metals were controlled and not released to the environment at levels that exceed regulatory limits.

Based on the data from the glass sampling, a preliminary mass balance on the Transition Batch glass that was present in the melter at the time of draining indicates that approximately 87 per cent of the lead, and 66 per cent of the barium added to the transition batch feed can be accounted for in the glass itself. Most of the remainder can be accounted for as particulate released to the melter off gas system.

There were no environmental impacts due to the bottom draining event, and no violations of the VITPP applicable or relevant and appropriate requirements (ARARs). Notification of the National Response Center (NRC) would not be required since the material was not released to the environment. The amount of SO_x, NO_x, lead, barium, and chromium released without control through the air or wastewater pathways would have been inconsequential. This is due to the nature of the released material (glass which quickly solidified), destruction of the nitrate and sulfate source of SO_x and NO_x, in the melter, binding of the metals into the glass relatively insoluble matrix, and the short contact time with the cooling water. All glass that self-drained from the melter has been containerized and accounted for. The glass will be characterized to determine proper management, including disposal, in accordance with RCRA requirements.

Lessons Learned and Recommendations

Safety Assessments and Hazard Analyses

Lesson 1:

The safety assessments and hazard analysis processes for the VITPP inadequately assessed the potential for casualties resulting from incompletely understood wastage in the melter. (Wastage is the combined effects of corrosion, erosion and electrochemical reactions which results in degradation of materials.)

Recommendation 1:

Project management must assure that periodic independent technical review and direction is provided during all phases of the project. This must include safety, design and operational considerations, and it must be available during early phases of the project..

Recommendation 2:

Because of the technical complexity of this project, project management must assure that a thorough, detailed hazard assessment is performed by an integrated team with adequate technical resources to establish the safety basis, and that this safety basis is revised as new data and facts become available.

Design Change Control

Lesson 2:

The design change control process existing at the time of the BDC design was ineffective in assuring that changes are adequate to maintain operations within the safety basis.

Recommendation 3:

Project management must ensure that management control processes are effectively implemented throughout the life of the project to maintain integrity of the safety basis. Not only must this include a consideration of the FDF design change control process, but also the design and safety interface between FDF and subcontractor scope of work.

Emergency Response

Lesson 3:

Good emergency response to casualty conditions requires good design, disciplined operations, and continuous training.

Recommendation 4:

The discipline gained from all aspects of operational experience and personnel training ensured that the incident resulted in no personnel injuries. Project management must ensure that such training is a continuing requirement for a project of this complexity.

Recommendation 5:

Project management must ensure that there are appropriate designs and/or effective administrative controls and/or project procedures to mitigate and confine the consequences of a potential breach of the VITPP melter which could spill molten glass onto the floor of the melter room, as well as, the consequences resulting from emergency response. For example, the melter room design did not include (1) curbs or dikes to contain large quantities of water, and prevent it from leaving the pad; or (2) curbs or dikes to mitigate the flow of molten glass into the sump.

Attachment I: Occurrence Report

OH-FN-FDF-FEMP-1996-0075
01/14/1997

OCCURRENCE REPORT

Fernald Environ. Mngmnt. Project
(Name of Facility)

Balance-of-Plant
(Facility Function)

Fernald Environ. Mngmnt. Project / Fluor Daniel Fernald Inc
(Name of Laboratory, Site or Organization)

Name: DON PAINE
Title: SILOS PROJECT MANAGER Telephone No.: (513)648-531(
(Facility Manager/Designee)

Name: MORROW, LAURA L
Title: TECHNICAL PROGRAM SPECIALIST Telephone No.: (513)648-417
(Originator/Transmitter)

Name: Date:
(Authorized Classifier (AC))

1. OCCURRENCE REPORT NUMBER: OH-FN-FDF-FEMP-1996-0075
   Vitrification Pilot Plant Melter Breach

2. REPORT TYPE AND DATE:

	Date	Time
[ ] Notification	12/27/1996	1152 MTZ
[X] Initial Update	01/14/1997	0858 MTZ
[ ] Latest Update		
[ ] Final		

3. OCCURRENCE CATEGORY:
   [ ] Emergency [X] Unusual [ ] Off-Normal [ ] Cancelled

4. NUMBER OF OCCURRENCES: 1 ORIG. OR:

5. DIVISION OR PROJECT: FDF/FEMP

6. SECRETARIAL OFFICE: EM - Environmental Management

7. SYSTEM, BLDG., OR EQUIPMENT:
   Vitrification Pilot Plant Melter

8. UCNI?: No

9. PLANT AREA: Vitrification Plant

10. DATE AND TIME DISCOVERED:
    12/26/1996 2230 (ETZ)

11. DATE AND TIME CATEGORIZED:
    12/27/1996 0905 (ETZ)

12. DOE NOTIFICATION:

12/27/1996 1020 (ETZ)	Rick Kahn	DOE-HQ

13. OTHER NOTIFICATIONS:

12/27/1996 0005 (ETZ)	Joe Neyer	DOE-FEMP
12/26/1996 2245 (ETZ)	Gordon Brown	DOE-FEMP
12/27/1996 0800 (ETZ)	Tim Marcus	DOE-OH

14. SUBJECT OR TITLE OF OCCURRENCE:
Vitrification Pilot Plant Melter Breach

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15. NATURE OF OCCURRENCE:
10) Cross-Category Items
C. Potential Concerns/Issues

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16. DESCRIPTION OF OCCURRENCE:
This Update Report is submitted to provide clarification and additional information which was unavailable at the time the Notification Report was submitted. A Final Report will be submitted within 45 days of categorization of this event, per DOE Order 232.1.

On December 26, 1996, at approximately 2222 hours, glass was discovered leaking from the bottom of the Melter at the Fluor Daniel Fernald (FDF) Vitrification Pilot Plant (VITPP). A Maintenance Supervisor had entered the Melter Room in response to a System Engineer's request for Instrument Mechanic support. A camera had recently been adjusted by an Instrument Mechanic and the Melter Operator, who was located on the second floor of the Melter Room, noticed a wisp of smoke in the vicinity of the camera. When the Maintenance Supervisor entered the Melter Room (on the ground floor), she noticed a small stream of molten glass flowing from the bottom of the VITPP Melter.

Prior to this discovery, the VITPP was in a normal idle operating mode, developing a vitrification technology while processing surrogate test materials. The material in the Melter contained some concentrations of barium and lead. The material was not radioactive. The compliment of shift personnel included 9 VITPP Team Members, 6 Maintenance personnel, and 1 Radiological Control Technician.

NOTIFICATIONS MADE:

AEDO - Assistant Emergency Duty Officer
EDO - Emergency Duty Officer
DOE-FEMP Duty Officer
DOE-OH Duty Officer
DOE-HQ Duty Officer
Public Affairs

17. OPERATING CONDITIONS OF FACILITY AT TIME OF OCCURRENCE:
Does Not Apply

18. ACTIVITY CATEGORY:
Normal Operations

19. IMMEDIATE ACTIONS TAKEN AND RESULTS:
The System Engineer directed the Control Room Operator to ramp-down power to Silicon Control Rectifiers (SCR) #1 and #2, as the first response to electrically isolate the Melter and radioed for the Shift Manager. The Maintenance Supervisor notified Fire & Safety personnel via radio that smoke was present in the Melter Room and that no fire was observed. At 2228 hours, the Communication Center received the first smoke alarm from the VITPP. The Communication Center Operator immediately notified the Assistant Emergency Duty Officer (AEDO). The System Engineer directed all personnel in the Melter Room to stand by the exit to the room on the first floor, near the Personal Protective Equipment (PPE) Room door while the Shift Supervisor manned the VITPP Control Room. During the first five minutes of the Melter breach, the glass pour stream had caught a cable on its descent steering the molten glass onto the epoxy painted floor and the floor ignited. The Shift Supervisor telephone from the VITPP Control Room to Communication Center that there was a fire and to dispatch the fire truck. The Shift Manager and the Maintenance Electrician worked together to open the SCR #1 local electrical disconnect (located adjacent to the fire) using a fire extinguisher to suffocate the flames from the burning epoxy.

After opening the local disconnect for SCR #1, the Shift Manager was successful in using a water hose to divert the glass pour stream back into the Bottom Drain Container. The water nozzle was then directed to "freeze" the glass stream (a method normally used in the glass industry). The attempt was abandoned after it was determined to be ineffective in reducing the flow of glass from the Melter. At 2234 hours, the Shift Supervisor directed the Control Room Operator to initiate an emergency shutdown of the VITPP by depressing the Emergency Stop Button and ordered the evacuation of all personnel from the building. Two electricians ensured that the main electrical disconnect to SCR #1 and SCR #2 were open to provide total electrical isolation before the Fire Crew arrived.

As the glass continued to flow from the Melter, the hole widened to approximately 2 inches in diameter. Some of the flow initially fell outside of the Bottom Drain Container and onto an epoxy painted concrete floor. The epoxy paint on the floor ignited and a small (about 2' X

19. IMMEDIATE ACTIONS TAKEN AND RESULTS: (continued)

2') fire resulted. Two portable fire extinguishers were unsuccessfully expended on the fire. Emergency Response personnel arrived on the scene by 2240 hours and put out the fire by flooding the floor with water. Approximately one cubic yard of surrogate glass discharged from the Melter to the bottom drain container and approximately 20% to the floor. An accountability of the evacuated personnel was performed and all were accounted for. Some key personnel were released to assist with the control and recovery actions. The quick and accurate responses performed by the Shift Team and the Emergency Response resulted in:

- no injuries or fatalities,
- no impact to off-site or on-site environment,
- minimized damage to the equipment.

The event was initially categorized as an Off-Normal Occurrence at 2350 hours by the AEDO. A post event critique was conducted on December 27 at 0830 hours. Following the critique, the event was ungraded to an Unusual Occurrence at 0905 hours.

24. EVALUATION: (By Facility Manager/Designee)
There was no significant impact to the safety and health of any off-site or on-site personnel as a result of this occurrence. Equipment damage estimates cannot be determined at this time, but will be included in the Final Report.

25. IS FURTHER EVALUATION REQUIRED?:	Yes [X]	No [ ]
IF YES - BEFORE FURTHER OPERATION?:	Yes [X]	No [ ]
BY WHOM?:	Silos Project Management	
BY WHEN?:	02/10/1997	

FACT SHEET
UNUSUAL OCCURRENCE OH-FN-FDF-FEMP-1996-0075
VITRIFICATION PILOT PLANT MELTER BREACH
DECEMBER 26, 1996

BACKGROUND/DISCUSSION

On December 26, 1996, at approximately 2222 hours, a Fluor Daniel Fernald (FDF) Vitrification Pilot Plant (VPP) Maintenance Supervisor was sent to the Melter Room by the System Engineer to inspect the West Camera for damage. The camera had recently been adjusted by an Instrument Mechanic and the Melter Operator, who was located on the second floor of the Melter Room, noticed a wisp of smoke in the vicinity of the camera. When the Maintenance Supervisor entered the Melter Room (on the ground floor), she noticed a small stream of molten glass flowing from the bottom of the VPP Melter and into the Bottom Drain Container.

Prior to this discovery, the VPP was conducting normal operations, processing surrogate material for testing and development of vitrification technologies. The material in the melter contains some concentrations of barium and lead. The material is not radioactive. The compliment of shift personnel included 9 VPP Team Members, 6 Maintenance personnel, and 1 Radiological Control Technician.

SIGNIFICANCE TO ES&H/PUBLIC PERSONNEL

There was no significant impact to the safety and health of any off-site or on-site personnel as a result of this occurrence. Equipment damage estimates cannot be determined at this time, but will be included in the Final Report.

IMMEDIATE ACTIONS TAKEN AND RESULTS

The Maintenance Supervisor alerted the System Engineer, who directed the Control Room Operator to ramp-down power to Silicon Control Rectifier (SCR) #1, in order to decrease Melter temperature. The Maintenance Supervisor notified Fire & Safety personnel via radio that smoke was present in the melter room, but no fire was observed. At 2225 hours, the Communication Center received the first smoke alarm from the VPP. The Communication Center Operator immediately notified the Assistant Emergency Duty Officer (AEDO). The System Engineer directed all personnel in the Melter Room to stand by the exit to the room on the first floor, near the Personal Protective Equipment (PPE) Room door. The Shift Manager attempted to "freeze" the glass stream (which was about the diameter of a pencil) by applying water to the stream from the Bottom Drain water hose. The attempt was abandoned after it was determined to not be effective in reducing the flow of glass from the Melter. At 2234 hours, the Shift Supervisor directed the Control Room Operator to initiate an emergency shutdown of the Melter by depressing the Emergency Stop Button and ordered the evacuation of all personnel from the building. Two electricians were ordered to open the main electrical disconnect for SCR #1 and SCR #2, which resulted in the complete electrical isolation of the Melter.
As the glass continued to flow from the Melter, the hole widened to approximately 2 inches in diameter. Some of the flow was diverted and fell outside of the Bottom Drain Container and onto a concrete floor. The epoxy paint on the floor ignited and a small (about 2' X 2') fire resulted. Two portable fire extinguishers were unsuccessfully expended on the fire. Emergency Response personnel arrived on the scene by 2240 hours and put out the fire by coating the floor with water. Approximately one cubic yard of surrogate material leaked from the Melter. An accountability of the evacuated personnel was performed and some personnel were released to assist with the control and recovery actions.

The event was initially categorized as an Off-Normal Occurrence at 2350 hours by the AEDO. A post-event critique was conducted on December 27 at 0830 hours. Following the critique, the event was up-graded to an Unusual Occurrence at 0905 hours.

NOTIFICATIONS MADE

AEDO - Assistant Emergency Duty Officer
EDO - Emergency Duty Officer
DOE-FEMP Duty Officer
DOE-OH Duty Officer
DOE-HQ Duty Officer
Public Affairs

DAILY OPERATIONS REPORT
UNUSUAL OCCURRENCE OH-FN-FDF-FEMP-1996-0075
VITRIFICATION PILOT PLANT MELTER BREACH
DECEMBER 26, 1996

- On December 26, 1996, at approximately 2222 hours, a Fluor Daniel Fernald (FDF) Vitrification Pilot Plant (VPP) Maintenance Supervisor was sent to the Melter Room by the System Engineer to inspect the West Camera for damage. The camera had recently been adjusted by an Instrument Mechanic and the Melter Operator, who was located on the second floor of the Melter Room, noticed a wisp of smoke in the vicinity of the camera.

- When the Maintenance Supervisor entered the Melter Room (on the ground floor), she noticed a small stream of molten glass flowing from the bottom of the VPP Melter and into the Bottom Drain Container.

- Prior to this discovery, the VPP was conducting normal operations, processing surrogate material for testing and development of vitrification technologies. The material in the melter contains some concentrations of barium and lead. The material is not radioactive. The compliment of shift personnel included 9 VPP Team Members, 6 Maintenance personnel, and 1 Radiological Control Technician.

- The Maintenance Supervisor alerted the System Engineer, who directed the Control Room Operator to ramp-down power to Silicon Control Rectifier (SCR) #1, in order to decrease Melter temperature. The Maintenance Supervisor notified Fire & Safety personnel via radio that smoke was present in the melter room, but no fire was observed.

- At 2225 hours, the Communication Center received the first smoke alarm from the VPP. The Communication Center Operator immediately notified the Assistant Emergency Duty Officer (AEDO). The System Engineer directed all personnel in the Melter Room to stand by the exit to the room on the first floor, near the Personal Protective Equipment (PPE) Room door.

- The Shift Manager attempted to "freeze" the glass stream (which was about the diameter of a pencil) by applying water to the stream from the Bottom Drain water hose. The attempt was abandoned after it was determined to not be effective in reducing the flow of glass from the Melter.

- At 2234 hours, the Shift Supervisor directed the Control Room Operator to initiate an emergency shutdown of the Melter by depressing the Emergency Stop Button and ordered the evacuation of all personnel from the building. Two electricians were ordered to open the main electrical disconnect for SCR #1 and SCR #2, which resulted in the complete electrical isolation of the Melter.

- As the glass continued to flow from the Melter, the hole widened to approximately 2 inches in diameter. Some of the flow was diverted and fell outside of the Bottom Drain Container and onto a concrete floor. The epoxy paint on the floor ignited and a small (about 2' X 2') fire resulted.

- Two portable fire extinguishers were unsuccessfully expended on the fire. Emergency Response personnel arrived on the scene by 2240 hours and put out the fire by coating the floor with water.

- Approximately one cubic yard of surrogate material leaked from the Melter. An accountability of the evacuated personnel was performed and some personnel were released to assist with the control and recovery actions.
- The event was initially categorized as an Off-Normal Occurrence at 2350 hours by the AEDO. A post-event critique was conducted on December 27 at 0830 hours. Following the critique, the event was up-graded to an Unusual Occurrence at 0905 hours.

- There was no significant impact to the safety and health of any off-site or on-site personnel as a result of this occurrence. Equipment damage estimates cannot be determined at this time, but will be included in the Final Report.

EVENTS

1. MELTER BREACH CAUSES FIRE AT VITRIFICATION PILOT PLANT

On December 26, 1996, at the Fernald Environmental Management Project Vitrification Pilot Plant, a fire occurred when molten glass leaked from a melter and spilled onto a concrete floor covered with epoxy. A maintenance supervisor, investigating a report of smoke, entered the vitrification pilot plant melter room and noticed a small stream of molten glass flowing from the bottom of the melter into a drain container. The supervisor notified fire and safety personnel by radio, stating there was smoke in the melter room but no fire. As the glass stream flowed from the melter, the hole widened from pencil-sized to approximately 2 inches in diameter. A small portion of the glass was diverted onto an epoxy-coated concrete floor where it ignited. Emergency response members put out the fire by coating the floor with water. Unexpected conditions often occur during startup testing of new designs. It is important to be alert for potential unexpected evolutions and to take appropriate actions to mitigate potential consequences. (ORPS Report OH-FN-FDF-FEMP-1996-0075)

Investigators determined that operators were using surrogate materials in the melter to test and develop vitrification technologies. The material contained concentrations of barium and lead, but it was not radioactive. After an operator saw a wisp of smoke in a viewing camera, the system engineer sent the maintenance supervisor to investigate. When the shift supervisor learned of the leak, he directed the control room operator to initiate an emergency shutdown of the melter and ordered evacuation of all building personnel. Investigators determined that approximately 1 cubic yard of surrogate material leaked from the melter. They found that emergency procedures were followed and personnel responded properly. The cause of the melter breach is currently being investigated.

Operating Experience Analysis and Feedback engineers reviewed the Occurrence Reporting and Processing System and found eight events related to glass or vitrification melters. The root cause reported for five events was a design problem; one event was attributed to personnel error. The following two events resulted in burned insulation and melted rubber.

- On November 5, 1996, at the Oak Ridge K-25 Site, operators reported smoke coming from the melter module of a transportable vitrification system. Operators contacted the fire department. Fire fighters applied water to stop the flow of glass from the melter, eliminating the source of smoke and steam. Fire department personnel determined that molten glass had flowed onto rubber water hoses and electrical cables. This generated the smoke and steam in the area. Investigators found that an excessive gap between two refractory blocks on the melter allowed molten glass to flow outside the containment provided by the melter. Features designed to minimize these gaps were not adequate. (ORPS Report ORO--LMES-K25WASTMAN-1996-0003)

- On December 5, 1996, at the Savannah River Site, the melter at the Reactor Materials Facility discharge chute became clogged and forced the molten glass onto the roller/cutter and conveyor. Molten glass built up on the conveyor and came in contact with the conveyor's rubber boot. Heat from the glass caused the boot to catch on fire. Fire fighters used water to extinguish the fire and stop the flow of glass. No injuries were caused by the fire. The

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cause of this event is still under investigation. (ORPS Report SR--WSRC-RMAT-1996-0008)

These events illustrate the importance of planning for unexpected evolutions when dealing with emerging technologies such as vitrification. Several vitrification designs are used across the DOE complex. NFS advocates sharing of information through user groups and list servers. OEAF has developed a *Hazard and Barrier Analysis Guidance Document*. It provides a set of simple, straightforward tools to devise more effective strategies for preventing and evaluating accidents and accident precursors that have occurred across the DOE complex. A copy is available from Richard Trevillian, (301) 903-3074. Managers and supervisors should review the guide and incorporate hazard and barrier analyses in work and operation processes.

KEYWORDS: design, fire, melter, vitrification

FUNCTIONAL AREAS: fire protection, industrial safety, research and development

2. DIESEL LUBRICATING OIL INCOMPATIBLE WITH LOW-SULFUR FUEL OIL

On December 19, 1996, the Nuclear Regulatory Commission (NRC) issued Information Notice 96-67, "Vulnerability of Emergency Diesel Generators to Fuel/Lubricating Oil Incompatibility." The notice describes degradation of two diesel engines at a commercial nuclear power plant. The degradation resulted from using lubricating oil that was incompatible with low-sulfur fuel oil. Test engineers found the degradation during pre-operational testing of a new safety-related emergency diesel generator. The problem cost the utility time and money in diagnosis and repair and, if it had gone undetected, could have disabled safety-related equipment.

In December 1995, during pre-operational full-load testing of the new diesel generator, test engineers noticed sporadic spikes in the engine crankcase pressure and found lubricating oil seeping from the crankshaft seal. They shut down the engine and performed a boroscopic inspection. One cylinder showed indications of abnormal wear. Maintenance workers replaced the cylinder liner, piston, and piston rings with a spare set. The engineers completed the full-load testing without further incident.

In January 1996, maintenance workers performed a scheduled disassembly inspection on the same engine. They found that all cylinders, including the replacement cylinder, had some degree of degradation. As a result, they inspected a non-safety diesel generator that had been installed in 1995 as a backup power supply in case of station blackout. One cylinder on the station blackout diesel exhibited degradation similar to that found in the safety-related diesel. Upon disassembly, the workers found excessive carbon deposits in all cylinders.

Utility managers assembled a team to determine the root cause of the degradation. The team determined the lubricating oil was incompatible with the type of fuel oil being used. The lubricating oil contained an additive package intended to neutralize fuel oil combustion products, including sulfuric acid, to prevent engine corrosion. The engine manufacturer originally recommended an American Petroleum Institute (API) CD-grade, synthetic lubricating oil. Their recommendation was based, in part, on a fuel oil procurement specification that required a maximum sulfur content of 0.3 percent. In early 1995, the fuel oil supplier switched to an oil with a maximum sulfur content of 0.05 percent to meet new

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Attachment 2: Bubbler Tubes

Chronology of Failure Considerations for the Air Bubbler Tubes

As part of the Melter incident investigation, the SRT performed a detailed review of project documentation that relates to the project's concern of a potential failure of the air agitation "bubbler" tubes. The following chronology summarizes the review results.

(6/2/94): DURATEK submits the technical proposal to FERMCO for a Joule-Heated Vitrification Furnace. Section 2.2.2 Agitation states:

The high sulfate and heavy metal content of the silo waste streams requires that an agitator method be provided to prevent the settling of heavy metals and the formation of a sulfate layer on top of the molten glass as well as throughly homogenize the molten pool with and without a cold cap. The design of the DuraMelter™ 1000-HT dictates that to safely and effectively stir the molten pool, commercially available molybdenum disilicide tubes be uniformly mounted through the floor of the melter down the center of the primary melt chamber. The bubble disrupts the glass pool by generating a curtain of bubbles between the electrode pairs. Convection is increased within the glass pool, and the heat transfer is accelerated to the reacting feed, thus increasing the production rate and making the process more economical. The bubblers also provides a strong oxidizing environment which promotes oxidation of metallic components in the feed and prevents them from forming a metallic (detrimental) layer on the bottom of the melter. Homogenization of the molten glass is much more likely with a bubbler system. It has proven to be effective in disrupting the formation of second phases (i.e., liquid [such as sulfate] layers on top of the molten glass). If more sulfate is present in the melter than can be entrained in the glass, the bubbling is briefly suspended to allow the sulphate layer to be drained off the top of the melt. (Ref 1.)

Attachment F of the proposal contains a list of answers to general questions. In section 3, Maintenance, DURATEK states:

The bubbler assembly will need replacing approximately every three to six months. This is a simple procedure which does not take more than thirty minutes to perform.

(6/22/94): FERMCO receives formal response from DURATEK to questions resulting from technical review of the proposal. FERMCO Question QB2:

Are the molybdenum disilicide tubes consumed and needed to be fed to keep pace with the erosion rate? Estimate the erosion rate anticipated if any.

DURATEK response: *The vendor providing the molybdenum disilicide tubes states that they are not consumed by most glass compositions. (See warranty qualification in section 2.10 of our proposal). (Ref 2.)*

2.10 Warranty states: *GTS DURATEK understands that the terms of the FERMCO warranty (does) not include damages to the equipment caused by improper operation, or corrosive attack by wastes fed into the melter by FERMCO or their designee on the melter and components supplied by GTS DURATEK. GTS DURATEK uses accepted corrosion resistant materials in melters, but further research and operating history with actual wastes to be fed to the melter is required to determine and guarantee long-term corrosion resistance. (Ref 1.)*

(10/94): HAZOP team meets to discuss melter design. Representatives from CRU4 Engineering and DURATEK are present. Meeting One: First Draft entitled "Vitrification Pilot Plant HAZOP Number 1." (Not issued) contains an initial list of recomendations. Recommendation (3) states:

Consider performing the pilot plant run without feeding the air sparger tubes into the furnace. Evaluate the degree of erosion/corrosion of the floor around the air sparger inlets at the end of the pilot plant run. The HAZOP team is concerned about the possibility of a tube breaking during an attempt to feed it into the furnace, resulting in personnel injuries or fatalities.

The preliminary information from DURATEK states that the bubbler tubes must be fed manually into the melter on a routine basis due to erosion. The scenario considered by the HAZOP team is that the tube breaks while replacing, resulting in a molten glass leak while personnel are under the melter, directly in harms way. This is the first documented indication that SA shows concern for occupancy under the melter.

(12/9/94): Table 9-5 of the Phase II Test Plan is submitted to the Assistant CRU4 Director by the VITPP Operations Manager. The table contains 53 potential off-normal melter scenarios and corrections that could be encountered during Phase I and Phase II testing. Also stated in the submittal is the recommendation that the table be

transmitted to DURATEK for their review and comment. Scenario 19 considers excessive erosion of the air bridge (tubes). The scenario states:

"Excessive erosion will require charging the bubbler tubes frequently to keep up with the wear. If wear cannot be controlled, the ends of the tubes will erode away and then erosion will take place on the melter bottom where the tubes are or were. Extensive erosion could result in a breach of the melter bottom. Methods to measure the rate of erosion or the amount of wear (real-time) needs to be developed." (Ref 3.)

(12/19/94): Parsons develops a list of ES&H questions to submit to GTS DURATEK in a Melter design review scheduled for 12/20/94. Issue 5 states:

"Are the bubblers protected from impact? Will workers be required to interface with the bubbler tubes." The concerns as stated: *"Possible worker hazard from tube shattering. Loss of melter material?"*

(12/20/94): Parsons conducts melter review with DURATEK. Attendees are representative from CRU4 Engineering, GTS DURATEK, and PARSONS Safety and Engineering. DURATEK receives the list of ES&H concerns.

(12/21/94): Meeting minutes are drafted from the design review of the melter. Item 27 states:

"DURATEK will review PARSONS' "Instrumentation" and "ES&H" notes and will respond to FERMCO.

(12/23/94): FERMCO receives DURATEK formal response to ES&H notes. (Ref 4.) Item 5 states:

"The bubble tubes will be protected from impact using an expanded metal cage. They are very brittle and will shatter if hit with a heavy/hard object. Operators will not have to interface with the bubble tubes during normal operations. Flow indication and control will be located in the control room (FERMCO's responsibility). The times that would require operators to interface with the bubbler tubes is only if they become plugged or need replacement."

This information is submitted to the HAZOP team. The team concludes that the issue of advancing the bubbler tubes is resolved.

It is the teams judgment that an engineering evaluation has been conducted and it is determined that it is not necessary to advance the bubblers during operation. The bubbler design is detailed to show "hard" connections, making it impractical to advancethe tubes routinely. The issues regarding the erosion of the tubes and the bottom of the melter still exits.

(2/9/95): The Melter/Gem Maker Off-Normal Scenarios (See 12/9/94) are transmitted to DURATEK. FERMCO requests DURATEK to review and provide formal response. (Ref 5.)

(2/15/95): DURATEK attends a two day (14 & 15) design review of the Melter/Gem Machine. Review team members are representatives from FERMCO Engineering, Battelle, Flur Daniel Enineering, FERMCO Construction, FERMCO Safety & Health, and FERMCO Operations. Twenty six other attendees of various disciplines, including the HAZOP team, were also present. Questions 145 through 156 were submitted by the HAZOP team. Question 149 states:

Is your review of the off-normal condition summary complete? Have you taken exception and/or added to the off-normal conditions listed?

This concern was taken as an action item. This question and several others were formally submitted to DURATEK for response requested on 5/5/95. Formal response was not obtained until 1/26/96. The DURATEK response to Question 149 :

The Phase I Test Plan, Attachment A, Table 9-5, "OU4 Pilot Plant Phase II Vitrification Potential Off-Normal Scenarios and Corrections" discusses all potential off-normal conditions. This information was considered in the HAZOP evaluation. (Ref 6.)

REFERENCES

1. GTS DURATEK to FERMCO, RFP 109420 Joule-Heated Glass Furnace and Amendment 1, therto, L-94845, (6/2/94)

2. GTS DURATEK Letter (DURATEK to FDF), RFP:109420: Joule-Heated Glass Furnace-Questions, L-94910, (6/22/94)

3. FERMCO, Operable Unit 4 Vitrification Pilot Plant Phase II Test Plan, 18-SU-0004, Rev O, (April 1995)

4. DURATEK Letter (DURATEK to FDF), Response to PARSONS ES&H Questions Concerning OU-4 Pilot Plant Furnace Area, L-95388, (1/23/94)

5. FERMCO Letter (FDF to DURATEK), Melter/Gem Maker Off-Normal Scenerios, C:CRUP:(CRU4/PP):95-0009, (2/9/95)

6. FERMCO Letter (FDF to DOE), Responses to the VITPP Melter/Gem Former Value Engineering Review, C:P:(FRVP):96-0012, (1/26/96)

Attachment 3: Summary of SO_2 Monitor Printouts:

Note: Prior to these measurements the monitor ran from December 16 at 19:37 hours to December 26 at 14:24 hours and recorded 0.0 ppm SO_2 for the 1-minute average, 15-minute STEL and 8-hour TWA levels on each 8 hour summary printout. Also, the monitor recorded 0.0 ppm SO_2 for the 8-hour TWA and 15-minute STEL levels of SO_2 from December 26, at 14:24 hours through December 26, 1996 at 22:24 hours. Then, on December 26, 1996 alarm printouts began with the following results:

Summary of Printouts from MDA 7100 SO_2 Monitor

24-Hour Time	Avg. ppm	24-Hour Time	Avg. ppm	24-Hour Time	Avg. ppm
22:23:23	3.6	22:28:23	30.2	22:32:47	44.2
22:23:47	18.5	22:28:47	27	22:33:11	38
22:24:06	20	22:29:11	27.6	22:33:35	27.6
22:24:44	20	22:29:35	25.7	22:33:59	28.9
22:25:08	41	22:29:59	33.1	22:34:23	28.3
22:25:32	72.3	22:30:23	32.8	22:34:47	24.8
22:25:56	51.5	22:30:47	28.9	22:35:11	24.4
22:26:20	38.7	22:31:11	29.5	22:35:35	23.7
22:26:44	22.6	22:31:35	30	22:35:59	20.9
22:27:09	25.8	22:31:59	29.1	22:36:29	17.9
22:27:34	20.7	22:32:23	30.4	terminated	
22:27:59	26.1				

Analytical Results:

The following table summarizes the results of the metals analysis for the section of indicating tape exposed during the melter fire incident (34 sample locations), and a section of unexposed "blank" tape.

ANALYTE	BLANK TAPE*	EXPOSED TAPE	
	µg/filter	µg/filter	mg/m³**
Aluminum	<3.5	9.6	2.3
Arsenic	<7.5	<7.5	<1.8
Barium	0.6	0.80	0.048
Beryllium	<0.035	<0.035	<0.0082
Cadmium	<0.25	<0.25	<0.059
Calcium	46	90	10
Chromium	<1.7	<1.7	<0.40
Cobalt	<0.43	<0.43	<0.10
Copper	1.1	2.3	0.30
Iron	4,900	6,200	320
Lead	<1.7	390	93
Lithium	<0.075	3.4	0.8
Magnesium	5.0	17.0	2.8
Manganese	<0.035	0.19	0.04
Molybdenum	<0.85	<0.85	<0.20
Nickel	<1.0	<1.0	<0.24
Phosphorus	<4.3	530	130
Platinum	<7.5	<7.5	<1.8
Selenium	<4.3	<4.3	<1.0
Silver	<0.25	<0.25	<0.059
Sodium	5,100	5,700	150
Tellurium	<2.5	<2.5	<0.59
Thallium	<7.5	<7.5	<1.8
Titanium	<0.43	<0.43	<0.10
Vanadium	<0.25	<0.25	<0.059
Yttrium	<0.043	<0.043	<0.010
Zinc	3,900	5,000	270
Zirconium	<0.25	<0.25	<0.059

NOTE: * Results cannot be reported in mg/m³ since no air volume was drawn through "blank" indicating tape.
** Results have been "blank" corrected.

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Attachment 4: Estimated Exposure Rate and Airborne Radioactivity Concentration - Calculation No. SD-S&H-BAS-3034

FERMCO-SUPPORTING DOCUMENT SYSTEM	DOC: SD-S&H-BAS-3034	CONTROL NO:

FLUOR DANIEL FERNALD

SAFETY AND HEALTH DIVISION

TITLE: An Estimate of Exposure Rate and Airborne Radioactivity Concentration from a Postulated Failure of VITPP Melter Containment.

AUTHOR: R. D. Daniels

REV NO: 0

APPROVAL: Grady Calhoun DATE: 1-30-97

Grady Calhoun, WMT&SP H&S Team Coach

Concurrence Signature:	Date:	Title:

EDC CONTROLLED COPY NO: 7509

CALCULATION WORKSHEET STATEMENT OF PROBLEM

To estimate the "worst case" exposure rates and airborne radioactive concentrations that may result from a loss of melter containment when given conditions similar to those resulting from the melter draining event that occurred on December 26, 1996 and assuming the VITPP is processing K-65 residues in lieu of non-radioactive surrogate material.

SUMMARY OF CONCLUSIONS	Originator's Signature and Date
The summary of conclusions are found in Tables 3 and 4 of this calculation.	Robert D. Daniels January 30, 1997
CHECKING METHOD	Checker's Signature
1. Review X	Roy M Crawford January 30, 1997
2. Alternate Calculation ____	
3. Performance Test ____	Signature Date

Rev. No.	Sheet No.	Description	Reviser's Signature/Date	Checker's Signature/Date	Approved by Signature/Date

An Estimate of Exposure Rate and Airborne Radioactivity Concentration from a Postulated Failure of VITPP Melter Containment.	DOC: SD-S&H-BAS-3034
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LIST OF ACRONYMS and ABBREVIATIONS

Vitrification Pilot Plant (VITPP) . . . 1
pitchblende ore residues (K-65) . . . 1
Bottom Drain Container (BDC) . . . 1
Width (W) . . . 2
Length (L) . . . 2
Height (H) . . . 2
Volume (V) . . . 2
pounds (lbs) . . . 2
kilograms (kg) . . . 2
cubic centimeters (cm^3) . . . 2
grams per cubic centimeter (g/cm^3) . . . 2
cubic feet (ft^3) . . . 2
Upper Confidence Limit (UCL) . . . 3
Remedial Investigation Report for Operable Unit 4 Feasibility Study (RI/FS) . . . 3
picocuries per gram (pCi/g) . . . 3
bismuth (Bi) . . .
lead (Pb) . . .
polonium (Po) . . . 6
grams per minute ($g\ min^{-1}$) . . . 6
Airborne Release Fraction (ARF) . . . 7
milliroentgen per hour (mR/hr) . . . 7
Derived Air Concentration (DAC) . . . 8
microcuries per milliliter of air ($\mu Ci/ml$) . . . 8

An Estimate of Exposure Rate and Airborne Radioactivity Concentration from a Postulated Failure of VITPP Melter Containment.	DOC: SD-S&H-BAS-3034
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1.0 Objective

To estimate the "worst case" exposure rates and airborne radioactive concentrations that may result from a loss of melter containment when given:

- Conditions similar to those resulting from the melter draining event that occurred on December 26, 1996,
- The VITPP is processing K-65 residues in lieu of non-radioactive surrogate material.

2.0 Background

The Vitrification Pilot Plant (VITPP) program consists of the design, construction, and operation of a one tonne per day output pilot scale vitrification facility. The VITPP was initially designed to treat pitchblende ore residues (K-65) extracted from Silo 2, bentonite clay, and Silo 3 waste material. The project was to be conducted in two phases, Phase I and Phase II. The current operating phase, (Phase I) utilizes non-radioactive bentonite and surrogate materials as feed material to the melter.

It was planned that, following successful completion of Phase I operations, the VITPP facility would be modified to support a radioactive material treatability study (Phase II). Phase II would involve the retrieval and processing of actual K-65 residues through a series of campaigns to evaluate the vitrification of actual wastes. During preparations for Phase I operations, a series of design reviews concluded that although adequate for Phase I operation, the current facility would require significant modifications beyond the present scope to support a test program using radioactive feedstock materials. Therefore, the path forward for the VITPP following Phase I is presently unresolved, pending the completion of the Independent Review Team Path Forward Decision Analysis.

While conducting Phase I (non-radioactive) operations On December 26, 1996, the VITPP experienced a loss of melter containment. A small hole approximately two inches in diameter developed in the bottom of the melter, allowing the glass to leak into the Bottom Drain Container (BDC) located beneath the melter. Although the majority of the glass was contained, an amount (~20%) spilled outside of the BDC and onto the floor. The purpose of the following calculations is to estimate the radiological impacts that would occur from the VITPP melter containment failure if one assumes the melter feed to be radioactive.

3.0 Assumptions

3.1 Geometry of the BDC

All external exposure rate calculations were performed using models developed with Grove Engineering, MicroShield® 4.21 gamma ray shielding software *(Ref. 1.)*. The geometry of the BDC is modeled using as a rectangular volume with its top open. The BDC is made of 3/16" steel and is lined with 2 1/4" thick refractory. Outer dimensional data was extracted from drawing 94X-5900-M-00808-M0010Rev 3. and corrected for the steel and refractory.

The container has the following dimensions:

- Width (W) = 79 cm
- Length (L) = 145 cm
- Height (H) = 72 cm
- Volume (V) = LxWxH = 824, 941 cm^3 or 29.13 ft^3

3.2 Spilled glass

The geometry of the spilled glass was estimated as an area source with both width and length equal to 1 meter. An area source was chosen to model worse case conditions.

3.3 Mass and Densities

The density of the waste glass was determined by weighing the filled BDC and dividing by volume. When given:

- The gross weight of the container after filling is 6,232 pounds (lbs) or 2,827 kilogra (kg),
- The tare weight of the container is 2,400 lbs (1,089 kg),
- The glass and concrete retrieved from the floor is 1,358 lbs, and
- The volume of the BDC is 824,941 cubic centimeters (cm^3),

Then the glass mass in the BDC is estimated to be 3832 lbs (1,738 kg) and the glass density is determined to be 2.11 g/cm^3.

The following additional values were used:

- Refractory density = 2.499 grams per cubic centimeter (g/cm^3) (NOVACON™ 65 cut sheet)
- Air density = 0.00122 g/cm^3 (MicroShield®)
- Steel density = 7.86 g/cm^3 (MicroShield®)

3.4 Amount of K-65 Residue

The melter is a three chamber melter with internal capacities of:

- Center Chamber 24 cubic feet (ft^3)
- Side Chambers 7.2 ft^3 (total of both side chambers)
- Total Volume 31.2 ft^3

The center chamber contains the waste glass and the side chambers contain non-radioactive frit glass. The radioactive glass in the center chamber provides approximately 77% of the to glass mass resulting from the spill. Approximately 30% of the slurry feed (by dry weight) is r radioactive glass additives.

Therefore using the volumes discussed above, it is calculated that the spilled radioactive glass would consist of 54% K-65 residue (939 kg), 23% non-radioactive additives, and 23% non-radioactive frit glass from the side chambers.

3.5 Radionuclide Inventory in the Glass

The radionuclide inventory of the K-65 residue is derived from the 95% Upper Confidence Limit (UCL) specific activity results from the sampling of Silos 1 and 2 as referenced in Table 4-2 of the Remedial Investigation Report for Operable Unit 4 Feasibility Study (RI/FS) *(Ref. 2.)*. The highest measured activity value from either silo was used to add a margin for conservatism. Initially, radionuclide concentrations that were not measured were determined assuming secular equilibrium conditions exists with the measured parent nuclides. This is a reasonable and conservative assumption (See Table I). The activity concentrations are reported in units of picocuries per gram (pCi/g) of residue.

Table 1: Radionuclide Activities in K-65 Material

Radionuclide	Concentration (pCi/g)	Radionuclide	Concentration (pCi/g)
Ac-227	7670*	Po-218	477000
Ac-228	1110	Ra-223	7670
Bi-210	202000	Ra-224	7360
Bi-211	7670	Ra-226	477000*
Bi-212	7360	Ra-228	1110
Bi-214	477000	Rn-219	7670
Fr-223	106	Rn-220	7360
Pa-231	4040*	Rn-222	477000
Pa-234	2	Th-227	7670
Pa-234m	1120	Th-228	7360*
Pb-210	202000*	Th-230	68900*
Pb-211	7670	Th-231	94
Pb-212	7360	Th-232	1110*
Pb-214	477000	Th-234	1120
Po-210	281000*	Tl-207	7670
Po-211	21	Tl-208	7360
Po-212	4716	U-234	1160*
Po-214	477000	U-235/236	94*
Po-215	7670	U-238	1120*
Po-216	7360		

Concentrations noted with an asterisk (*) are actual measured concentrations. The remaining radionuclides are decay products assumed to be in secular equilibrium.

Table 2 provides the parent radionuclide, decay products, and the appropriate branching ratios for the decay products (branching ratios for parent radionuclides are always considered equal to "1"). Decay chains are divided into partial decay chains with each "parent" a radionuclide with a measured concentration. The symbol "n/a" indicates radionuclides whose first decay product is a radionuclide with a measured concentration.

Decay chains were identified by executing the Microshield program for each radionuclide with a measured concentration.

Table 2: Decay Chain Information

Parent Radionuclide	Decay Products	Branching Ratio	Parent Radionuclide	Decay Products	Branching Ratio
U-238	Th-234	1	Th-228	Ra-224	1
	Pa-234m	1		Rn-220	1
	Pa-234	1.60E-03		Po-216	1
Ac-227	Fr-223	1.38E-02		Pb-212	1
	Th-227	1		Bi-212	1
	Ra-223	1		Tl-208	3.6E-01
	Rn-219	1		Po-212	6.4E-01
	Po-215	1	Ra-226	Rn-222	1
	Pb-211	1		Po-218	1
	Bi-211	1		Pb-214	1
	Tl-207	1		Bi-214	1
	Po-211	2.73E-03		Po-214	1
Pb-210	Bi-210	1	U-235	Th-231	1
Th-232	Ra-228	1	Po-210	n/a	n/a
	Ac-228	1	U-234	n/a	n/a
Th-230	n/a	n/a	Pa-231	n/a	n/a

The estimated amount of residues are multiplied by the activity concentrations of Table 1. To determine the equilibrium source term for the BDC and the spill. The estimated amount of K-65 wastes within each source term is found by the following general equation:

$$M_{K\text{-}65} = V_s \rho \xi$$

Where:

(a) M_{K-65} is the mass of K-65 residue in grams,
(b) V_s is the actual volume of the component (i.e. BDC and spill volume) in cm^3,
(c) ρ is the density of the glass (2.11 g/cm^3),
(d) ξ is fraction of glass that is K-65 residue. (unitless, 0.54).

3.7 Correction for Equilibrium

The primary gamma emitting radionuclides found in the waste glass are the short-lived particulate daughter products resulting from radon-222 decay. The radon inventory in the feed will be removed by the off-gas system, leaving behind its particulate daughters. The effective half-live of the radon progeny is approximately 30 minutes, therefore a melter residence time in excess of 4 hours would allow nearly all of the gamma emitting inventory to decay. If one assumes a melter throughput of 1 tonne per day, melter center chamber volume of 24 ft^3, and a glass product density of 2.9 g/cm^3 *(Ref. 3.)*, the mean residence time in the melter is about 2 days. Given the expected loss of the radon generated in the melt pool, it is unlikely that the gamma emitting radon daughters would be available in significant quantity.

To model the expected conditions, the radionuclide inventory was modified to exclude radon and certain isotopes of bismuth (Bi), lead (Pb), polonium (Po), and thallium (Tl) that result from radon decay. Thus, activity values for Bi, 211, Bi-214, Pb-211, Pb-214, Po-211, Po-214, Po-215, Po-216, Po-218, Tl-207, Rn-219, Rn-220, and Rn-222 were "zeroed" to reflect initial, non-equilibrium conditions. Then, to add a margin for conservative "worst case" results, the "zeroed" inventory was " decayed" for 14.4 hours to allow ingrowth of radon and their progeny to an approximate 10% value. The 10% source terms for the BDC and spill were saved as BDC10.MSS and Area10.MSS, respectively. Likewise, equilibrium and 0% source terms for the BDC and spill were saved as BDC.MSS, BDC0.MSS, AREA.MSS, and AREA0.MSS respectively.

3.8 Assumptions for Estimated Airborne Concentrations

The quantity of off-gas materials which may exhaust the melt as particulates during active introduction of feed into the melter is assumed as a maximum of 0.5% of the daily throughput (A standard industry assumption). When given a throughput of 1 tonne (2,200 lbs) per day, the solids are carried over at a rate of about 3.47 grams per minute ($g\ min^{-1}$). One may conservatively estimate the amount of airborne off-gas materials from the postulated event can be represented by some fraction of the total amount of off-gas materials exhausted during normal operation. The West Valley Final Safety Analysis Report (WVNS-SAR-0003) *(Ref. 4.)* states that 10 to 30% of the off-gas materials may be released from a spill occurring during introduction of feedstock into the melter. Significantly smaller amounts of off-gas materials are released from the melt after the addition of slurry feed has been stopped: The West Valley Final Safety Analysis Report states that the release of off-gas materials is only 27% of normal release after 2.5 hours, and 14% of normal release after 5.5 hours.

These values are based upon measured off-gas production rates. For this analysis, a conservative fraction of 30% of normal release was used. When it is postulated that particulates from the melt become airborne at a rate of 1.04 g min^{-1} (3.47 g min^{-1} *0.3) for a 20 minute time interval, it is estimated that a total of 21 g of particulate material becomes airborne, and be released into the melter room. The resulting Airborne Release Fraction (ARF) is 8.5E-06 which is comparable to the measured ARFs for free fall spills of slurried material *(Ref. 5.)*.

The amount of radioactive materials released from the spill is less, and is estimated by using the fraction of K-65 residue in the melt given previously (0.54). This results in a conservative estimate of 11.2 g of K-65 residue potentially becoming airborne. The total source term dispersed is determined by multiplying the activity concentrations shown in Table 1 by the estimated mass of residue. Only those constituents that significantly contribute to the potential dose are considered.

Air turbulence in the area during a postulated spill causes mixing in the melter room with uniform dispersion of the radioactive materials. The melter room volume is assumed to be approximately 41, 400 ft^3 (60 ft x 23 ft x 30 ft). Conservatively assuming an equipment displacement of 30%, yields a usable volume of 28,980 ft^3 .

4.0 Results

4.1 External Exposure rates

Exposure rates with buildup were determined using MicroShield® at a distances of 30 cm and 100 cm from the outer surface of the BDC and spill material. For the BDC, two conditions were considered; exposure from the side wall of the container (shielded) and exposure from the top (unshielded). All points of exposure are modeled at the container centerline. For the area source, the point of exposure is assumed to be at a height of 100 cm. Cases were run to show exposure rates (in air) at T=0 hours, 14.4 hours, and equilibrium conditions. The results are shown in Table 3. All exposure rates listed are in units of milliroentgen per hour (mR/hr).

Table 3: Table of Exposure Rates

Model	Distance from Source (cm)	Exposure Rate (mR/hr w buildup) T=0	Exposure Rate (mR/hr w buildup) T=14.4	Exposure Rate (mR/hr w buildup) Secular Equilibrium
Area Spill	1	1.734	5.324	46.77
	30	1.345	4.126	36.24
	100	0.7844	2.403	21.10
BDC Side Exposure	1	5.409	12.93	117.7
	30	3.291	8.377	76.65
	100	1.011	2.629	24.07
BDC Top Exposure	1	17.08	48.84	446.7
	30	7.458	21.69	198.3
	100	1.918	4.547	50.70

4.2 On-Site Airborne Concentrations

Airborne concentrations were determined and compared to the appropriate Derived Air Concentration (DAC) *(Ref. 6.)*. The DAC value is the concentration of a radionuclide in air, that if inhaled over the period of a work year (2,000 hours), would result in the derived limit (Annual Limit on Intake, ALI) for that radionuclide. For example: Assume the airborne concentration of Th-230 reaches a peak concentration that is 130 times greater than the DAC value. If one assumes a person occupies this environment for a period of say, 10 minutes without respiratory protection, then that person is estimated to have received an exposure equivalent to 1.1% of the annual limit on intake for Th-230.

The estimated concentrations are assumed to be instantaneous peak values. Airborne radioactivity concentrations are listed in units of microcuries per milliliter of air (μCi/ml). The airborne activity concentrations are as follows:

Table 4: Airborne Concentrations

Radionuclide	Relative Dose Fraction and Class *(Ref. 7.)*	Derived Airborne Concentration (DAC) (μCi/ml)	Estimated Melter Room Concentration (μCi/ml)
Ac-227	0.2548 Y	2E-12	1E-10
Pb-210	0.0774 D	1E-10	3E-09
Po-210	0.0682 D	3E-10	4E-09
Ra-226	0.1048 W	3E-10	7E-09
Th-228	0.0204 Y	7E-12	1E-10
Th-230	00.4401 Y	7E-12	9E-10
Th-232	0.0340 Y	1E-12	2E-11
Uranium	0.0003 W	3E-10	2E-11

4.3 Off-Site Airborne Concentrations

The impact to off-site receptors were evaluated using HOTSPOT *(Ref. 8.)* and ARAC *(Ref. 9.)*. Both models represent standard gaussian plume dispersion from an instantaneous, "puff" release at ground level. The stability class is assumed to be Class F with a mean wind speed of 3.4 meters per second (m/sec) to model "worst case" conditions. The results from the model were indicate that the maximum exposed individual (MEI) occurs within 100 meters (on site). At 100 meters, the models show an individual would receive a Committed Effective Dose Equivalent (CEDE) from Ra-226 of 1.50E-03 millirem. At 500 meters (nearest off-site receptor) the same individual would receive 5.3E-05 millirem. No impact to the off-site receptor is expected from this event.

5.0 Attachments

5.1 HOTSPOT Output File
5.2 ARAC Output File
5.3 Microshield® Output Files

5.0 References

1. GROVE ENGINEERING, INC., *MicroShield®*, Version 4.21. Rockville, MD.(February, 1995).

2. US DOE, *Remedial Investigation Report for Operable Unit 4, Final, Volume 1 of 3*, Fernald Environmental Management Project, Fernald, Oh. (November 1993).

3. US DOE, *Operable Unit 4 Treatability Study Report for the Vitrification of Residues from Silos 1, 2, and 3*, Fernald Environmental Management Project, Fernald, Oh. (May 1993).

4. WVDP, *Safety Analysis for Vitrification System Operations and High Level Waste Interim Storage*, WVNS-SAR-003, Rev 2, West Valley Nuclear Services Co., Inc., West Valley, NY. (June 1995).

5. U.S. DOE, *Airborne Release Fractions/Rates and Respirable Fractions for Nonreactor Nuclear Facilities*, DOE Handbook 3010-94, Vol I, Table 3-8, Washington, D.C.(December 1994).

6. Code of Federal Regulations, Energy 10 Parts 500 to End, *Occupational Radiation Protection*, 10 CFR 835, U.S. Government Printing Office, Washingtion (December, 1993).

7. FERMCO (FDF), *Technical Basis for Internal Dosimetry at the Fernald Environmental Management Project*, SD-IRST-BAS-3008, Fernald Environmental Management Project, Fernald, Oh. (December 1995).

8. Homann, Steven G., *Hotspot Health Physics Codes for the PC*, Lawrence Livermore National Laboratory, University of California, Livermore, Calif. (March 1994)

9. Abriam, Rosemary O., Baskett, Ronald L., Crew, Daryl, Moore, Miki, Spruiell, Mark E., and Vogt, Phillip J., *ARAC Site Workstation System User's Guide*, Lawrence Livermore National Laboratory, University of California, Livermore, Calif. (February 1995).

HOTSPOT 8.0 GENERAL PLUME 10-19-1996 9:05

RADIONUCLIDE : RA-226
Inhalation Class : W
HALFLIFE : 1.6E+03 Years
SOURCE TERM : 5.300E-06 Ci
RELEASE FRACTION : 1.000E-02
FILTER EFFICIENCY: 0.000000 %
EFFECTIVE RELEASE HEIGHT : 0.00 m
WIND SPEED (h=2 m): 3.4 m/s
STABILITY CLASS : F DEPOSITION VELOCITY : 1.00 cm/s
RECEPTOR HEIGHT : 0.0 m
INVERSION LAYER HEIGHT : NONE
SAMPLE TIME : 10.000 min
MAXIMUM DOSE DISTANCE : < 0.10 km MAXIMUM CEDE : > 1.5E-06 rem
Plume Centerline

D = 0.100 km	D = 0.200 km	D = 0.500 km
DEP = 5.7E-06 uCi/m^2	DEP = 1.3E-06 uCi/m^2	DEP = 2.0E-07 uCi/m^2
CHI = 5.7E-10 (Ci-s)/m^3	CHI = 1.3E-10 (Ci-s)/m^3	CHI = 2.0E-11 (Ci-s)/m^3
50-YR DOSE COMMITMENT:	50-YR DOSE COMMITMENT:	50-YR DOSE COMMITMENT:
LUNG 1.1E-05 rem	LUNG 2.6E-06 rem	LUNG 4.0E-07 rem
SURFACE BONE 5.3E-06 rem	SURFACE BONE 1.2E-06 rem	SURFACE BONE 1.9E-07 rem
EFFECTIVE DOSE EQUIVALENT 1.5E-06 rem	EFFECTIVE DOSE EQUIVALENT 3.5E-07 rem	EFFECTIVE DOSE EQUIVALENT 5.3E-08 rem
D = 1.000 km	D = 2.000 km	D = 5.000 km
DEP = 5.2E-08 uCi/m^2	DEP = 1.4E-08 uCi/m^2	DEP = 3.0E-09 uCi/m^2
CHI = 5.2E-12 (Ci-s)/m^3	CHI = 1.4E-12 (Ci-s)/m^3	CHI = 3.0E-13 (Ci-s)/m^3
50-YR DOSE COMMITMENT:	50-YR DOSE COMMITMENT:	50-YR DOSE COMMITMENT:
LUNG 1.0E-07 rem	LUNG 2.8E-08 rem	LUNG 5.9E-09 rem
SURFACE BONE 4.8E-08 rem	SURFACE BONE 1.3E-08 rem	SURFACE BONE 2.8E-09 rem
EFFECTIVE DOSE EQUIVALENT 1.4E-08 rem	EFFECTIVE DOSE EQUIVALENT 3.7E-09 rem	EFFECTIVE DOSE EQUIVALENT 8.0E-10 r
D = 10.000 km	D = 20.000 km	D = 50.000 km
DEP = 1.1E-09 uCi/m^2	DEP = 3.1E-10 uCi/m^2	DEP = 2.9E-11 uCi/m^2
CHI = 1.1E-13 (Ci-s)/m^3	CHI = 3.1E-14 (Ci-s)/m^3	CHI = 2.9E-15 (Ci-s)/m^3
50-YR DOSE COMMITMENT:	50-YR DOSE COMMITMENT:	50-YR DOSE COMMITMENT:
LUNG 2.1E-09 rem	LUNG 6.0E-10 rem	LUNG 5.8E-11 rem
SURFACE BONE 1.0E-09 rem	SURFACE BONE 2.9E-10 rem	SURFACE BONE 2.7E-11 rem
EFFECTIVE DOSE EQUIVALENT 2.9E-10 rem	EFFECTIVE DOSE EQUIVALENT 8.0E-11 rem	EFFECTIVE DOSE EQUIVALENT 7.7E-12 rem

UCRL-MA-115639 DR
REV. 1

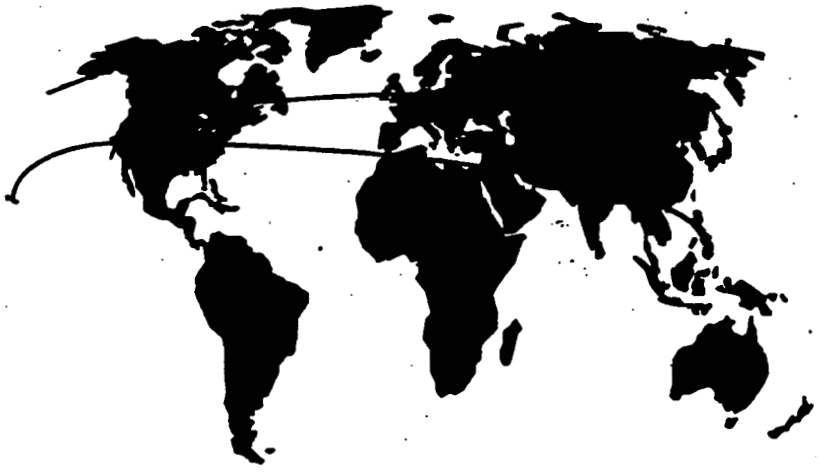
ARAC
Site Workstation System User's Guide

By

Rosemary O. Abriam, Ronald L. Baskett,
D. J. Daryl Crew, R. Miki Moore, Mark E. Spruiell, and Phillip J. Vogt



Version 2.0
February 1995



(510) 424-ARAC

EMERGENCY ONLY ***510-422-9100***

Customer Support **510-422-9159**

Exercise Scheduling **510-422-7627**

FAX: 510-423-4527 or 422-5924

000473

University of California
Lawrence Livermore National Laboratory

Gauss Parameter Summary

Run name:	Gauss 22-Jan-97 14:51 EST	Nuclide:	RA-226
Run type:	Instantaneous	Contour height:	4.9 ft
Run start:	22-Jan-97 13:47 EST	Release height:	4.9 ft
Run duration:	0:15	Location (Lat, Long):	39° 17' 57" N, 84° 41' 47" W
		Location (X, Y):	698.642, 4352.506

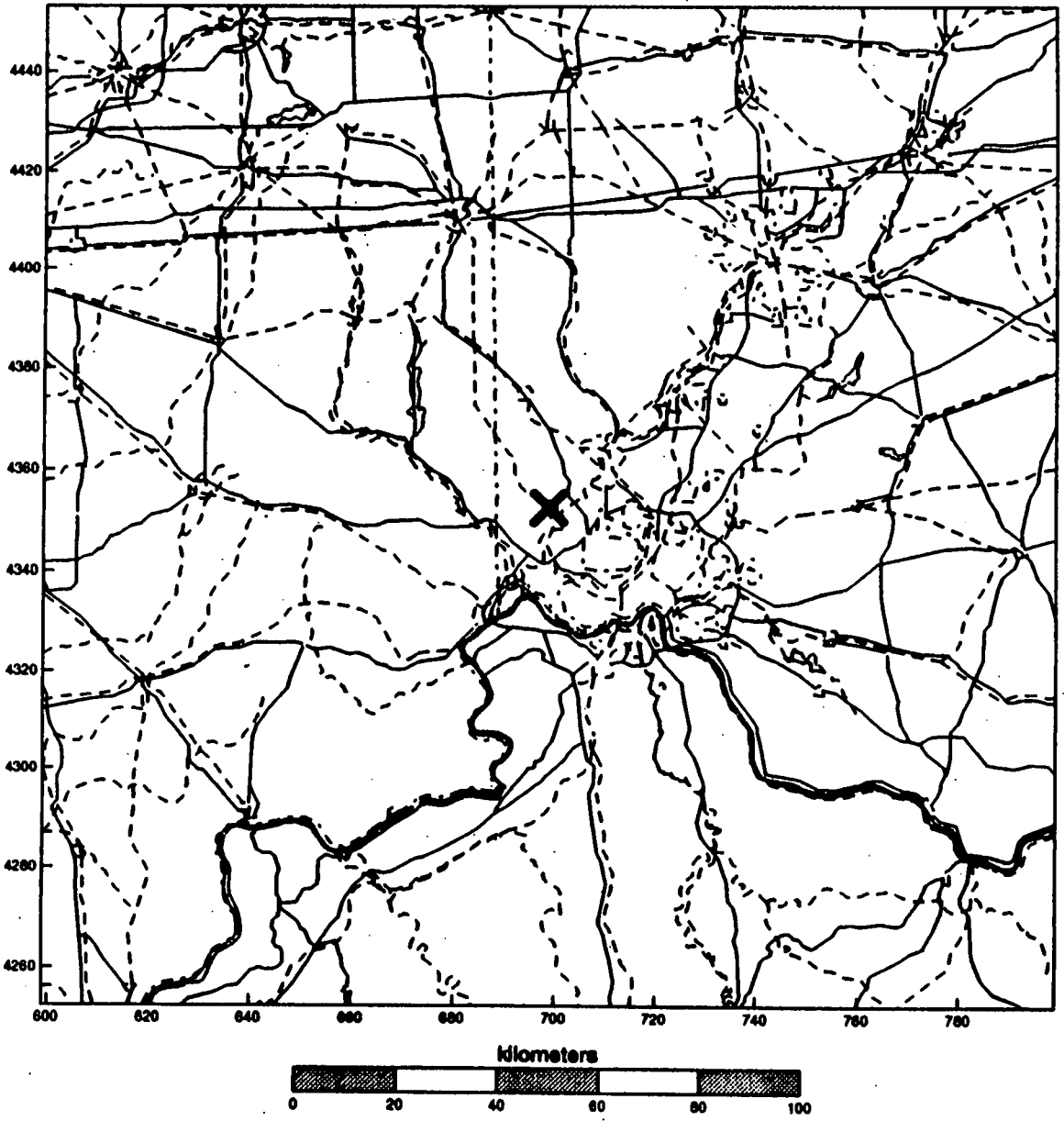
Source History

Time	Source (Ci/s)
22-Jan-97 13:47 EST	0.000E+00
22-Jan-97 14:02 EST	0.000E+00

Metdata History

Time	Wind Speed (mph)	Wind Direction	Stability Class
22-Jan-97 13:47 EST	2.2	180	D

Gaussian Plot - Gauss 22-Jan-97 14:51 EST



Contour Values (Ci/m3)	Cumulative Areas (sq mi)
>1.09E+229	0.0
>1.09E+229	0.0
>1.02E+229	0.0
> 0.00E+00	0.0
> 0.00E+00	0.0

000473

209

Questionnaire Summary for incident FERNALD 0016 22-Jan-97 15:02 EST

Incident Time
Start: 22-Jan-97 13:47 EST
Stop: 22-Jan-97 14:02 EST

Release: R#1 - Other
Location
Latitude: 39 17 57 N
Longitude: 84 41 47 W

Strength

Nuclide	Start	Stop	Source (Ci)
AC-227	22-Jan-97 13:47 EST	22-Jan-97 14:02 EST	8.59E-08
AC-228	22-Jan-97 13:47 EST	22-Jan-97 14:02 EST	1.24E-08
BI-210	22-Jan-97 13:47 EST	22-Jan-97 14:02 EST	2.26E-06
BI-211	22-Jan-97 13:47 EST	22-Jan-97 14:02 EST	8.58E-08
BI-212	22-Jan-97 13:47 EST	22-Jan-97 14:02 EST	2.55E-08
BI-214	22-Jan-97 13:47 EST	22-Jan-97 14:02 EST	5.34E-06
PA-234	22-Jan-97 13:47 EST	22-Jan-97 14:02 EST	1.24E-11
PA-234M	22-Jan-97 13:47 EST	22-Jan-97 14:02 EST	7.76E-09
PB-210	22-Jan-97 13:47 EST	22-Jan-97 14:02 EST	2.26E-06
PB-211	22-Jan-97 13:47 EST	22-Jan-97 14:02 EST	8.58E-08
PB-212	22-Jan-97 13:47 EST	22-Jan-97 14:02 EST	2.55E-08
PB-214	22-Jan-97 13:47 EST	22-Jan-97 14:02 EST	5.34E-06
PO-210	22-Jan-97 13:47 EST	22-Jan-97 14:02 EST	3.15E-06
PO-212	22-Jan-97 13:47 EST	22-Jan-97 14:02 EST	2.55E-08
PO-214	22-Jan-97 13:47 EST	22-Jan-97 14:02 EST	5.34E-06
PO-215	22-Jan-97 13:47 EST	22-Jan-97 14:02 EST	8.59E-08
PO-216	22-Jan-97 13:47 EST	22-Jan-97 14:02 EST	2.55E-08
PO-218	22-Jan-97 13:47 EST	22-Jan-97 14:02 EST	5.34E-06
RA-223	22-Jan-97 13:47 EST	22-Jan-97 14:02 EST	8.59E-08
RA-224	22-Jan-97 13:47 EST	22-Jan-97 14:02 EST	2.55E-08
RA-226	22-Jan-97 13:47 EST	22-Jan-97 14:02 EST	5.34E-06
RA-228	22-Jan-97 13:47 EST	22-Jan-97 14:02 EST	1.24E-08
TH-234	22-Jan-97 13:47 EST	22-Jan-97 14:02 EST	7.76E-09
U-238	22-Jan-97 13:47 EST	22-Jan-97 14:02 EST	7.76E-09
U-234	22-Jan-97 13:47 EST	22-Jan-97 14:02 EST	1.04E-08
TH-227	22-Jan-97 13:47 EST	22-Jan-97 14:02 EST	8.59E-08
TH-228	22-Jan-97 13:47 EST	22-Jan-97 14:02 EST	2.55E-08
TH-230	22-Jan-97 13:47 EST	22-Jan-97 14:02 EST	7.72E-07
TH-231	22-Jan-97 13:47 EST	22-Jan-97 14:02 EST	6.05E-10
TH-232	22-Jan-97 13:47 EST	22-Jan-97 14:02 EST	1.24E-08

Geometry
Height: 4.9 ft
Diameter: 9.8 ft
Gas Temp: 1000.0 deg C
Heat Emsn:
Exit Vel:

Surface Roughness
On site

Description
Postulted event . Airborne contamination as a result of melter breach at the VITPP a

Contact
Name: Mr Greg Campbell
Voice: [redacted] (Comm)
FAX: [redacted] (Comm)

MicroShield Batch Run Log

Batch Run Started on Tuesday, January 28, 1997 at 4:10 p.m.

Eighteen cases initially in the queue.

CASE	FILE	GEOM	START TIME	ELAPSED	mR/hr w/BUILDUP	SENS. CYCLE
1	30AREA	5	4:10 p.m.	0:00:15	4.677e+001	REFERENCE
X = 101				0:00:22	4.677e+001	1 of 2
X = 130				0:00:28	3.624e+001	2 of 2
4	30AREA0	5	4:11 p.m.	0:00:15	1.734e+000	REFERENCE
X = 101				0:00:22	1.734e+000	1 of 2
X = 130				0:00:28	1.345e+000	2 of 2
7	30AREA10	5	4:11 p.m.	0:00:15	5.324e+000	REFERENCE
X = 101				0:00:22	5.324e+000	1 of 2
X = 130				0:00:28	4.126e+000	2 of 2
10	30BDC	13	4:12 p.m.	0:03:55	1.177e+002	REFERENCE
X = 86.1813				0:07:41	1.177e+002	1 of 2
X = 115.181				0:11:20	7.665e+001	2 of 2
13	30BDC0	13	4:23 p.m.	0:03:55	5.409e+000	REFERENCE
X = 86.1813				0:07:40	5.409e+000	1 of 2
X = 115.181				0:11:19	3.291e+000	2 of 2
16	30BDC10	13	4:34 p.m.	0:03:55	1.293e+001	REFERENCE
X = 86.1813				0:07:40	1.293e+001	1 of 2
X = 115.181				0:11:19	8.377e+000	2 of 2
19	30BDCTOP	13	4:46 p.m.	0:03:55	4.467e+002	REFERENCE
X = 79.99				0:07:41	4.467e+002	1 of 2
X = 108.99				0:11:13	1.983e+002	2 of 2
22	30BTOP0	13	4:57 p.m.	0:03:55	1.708e+001	REFERENCE
X = 79.99				0:07:41	1.708e+001	1 of 2
X = 108.99				0:11:15	7.458e+000	2 of 2
25	30BTOP10	13	5:08 p.m.	0:03:54	4.884e+001	REFERENCE
X = 79.99				0:07:40	4.884e+001	1 of 2
X = 108.99				0:11:12	2.169e+001	2 of 2
28	AREA	5	5:19 p.m.	0:00:15	4.677e+001	REFERENCE
X = 101				0:00:22	4.677e+001	1 of 2
X = 200				0:00:28	2.110e+001	2 of 2
31	AREA0	5	5:20 p.m.	0:00:15	1.734e+000	REFERENCE
X = 101				0:00:22	1.734e+000	1 of 2
X = 200				0:00:28	7.844e-001	2 of 2
34	AREA10	5	5:20 p.m.	0:00:15	5.324e+000	REFERENCE
X = 101				0:00:22	5.324e+000	1 of 2
X = 200				0:00:29	2.403e+000	2 of 2
37	BDC	13	5:21 p.m.	0:03:56	1.177e+002	REFERENCE
X = 86.1813				0:07:41	1.177e+002	1 of 2
X = 185.181				0:11:18	2.407e+001	2 of 2
40	BDC0	13	5:32 p.m.	0:03:55	5.409e+000	REFERENCE
X = 86.1813				0:07:41	5.409e+000	1 of 2
X = 185.181				0:11:17	1.011e+000	2 of 2
43	BDC10	13	5:43 p.m.	0:03:55	1.293e+001	REFERENCE
X = 86.1813				0:07:40	1.293e+001	1 of 2
X = 185.181				0:11:16	2.629e+000	2 of 2
46	BDCTOP	13	5:55 p.m.	0:03:55	4.467e+002	REFERENCE
X = 79.99				0:07:40	4.467e+002	1 of 2
X = 178.99				0:11:09	5.070e+001	2 of 2

49	BDCTOP0	13	6:06 p.m.	0:03:54	1.708e+001	REFERENCE
X = 79.99				0:07:40	1.708e+001	1 of 2
X = 178.99				0:11:08	1.918e+000	2 of 2
52	BDCTOP10	13	6:17 p.m.	0:03:55	4.884e+001	REFERENCE
X = 79.99				0:07:39	4.884e+001	1 of 2
X = 178.99				0:11:08	5.547e+000	2 of 2

Batch run completed on Tuesday, January 28, 1997 at 6:28 p.m.

TOTAL EXECUTION TIME: 2:18:05

MicroShield 4.21 - Serial #4.21-00915
Fermco

Page : 1
DOS File: 30AREA.MS4
Run Date: January 28, 1997
Run Time: 4:11 p.m. Tuesday
Duration: 0:00:28

File Ref: _____
Date: __/__/__
By: _____
Checked: _____

Case Title: Glass Spill (Area Source) 100% Eq

GEOMETRY 5 - Horizontal Rectangular Area

	centimeters	feet and inches	
Dose point coordinate X:	101.0	3.0	3.8
Dose point coordinate Y:	100.0	3.0	3.4
Dose point coordinate Z:	50.0	1.0	7.7
Rectangular area length:	100.0	3.0	3.4
Rectangular area width :	100.0	3.0	3.4
Air Gap:	1.0	0.0	.4

Source Area: 10000 sq cm 10.7639 sq ft. 1550. sq in.

MATERIAL DENSITIES (g/cm^3)

Material	Air Gap	Immersion Shield
Air	0.00122	0.00122

BUILDUP
Method: Buildup Factor Tables
The material reference is Immersion

INTEGRATION PARAMETERS

	Quadrature Order
Z Direction	20
X Direction	20

SOURCE NUCLIDES

Nuclide	curies	ÌCi/cm»	Nuclide	curies	ÌCi/cm»
Ac-227	2.6000e-003	2.6000e-001	Ac-228	3.7000e-004	3.7000e-002
Bi-210	6.7000e-002	6.7000e+000	Bi-211	2.6000e-003	2.6000e-001
Bi-212	2.4000e-003	2.4000e-001	Bi-214	1.6000e-001	1.6000e+001
Fr-223	3.5000e-005	3.5000e-003	Pa-231	1.3000e-003	1.3000e-001
Pa-234	6.0000e-007	6.0000e-005	Pa-234m	3.7000e-004	3.7000e-002
Pb-210	6.7000e-002	6.7000e+000	Pb-211	2.6000e-003	2.6000e-001
Pb-212	2.4000e-003	2.4000e-001	Pb-214	1.6000e-001	1.6000e+001
Po-210	9.3000e-002	9.3000e+000	Po-211	7.0000e-006	7.0000e-004
Po-212	1.6000e-003	1.6000e-001	Po-214	1.6000e-001	1.6000e+001
Po-215	2.6000e-003	2.6000e-001	Po-216	2.4000e-003	2.4000e-001
Po-218	1.6000e-001	1.6000e+001	Ra-223	2.6000e-003	2.6000e-001
Ra-224	2.4000e-003	2.4000e-001	Ra-226	1.6000e-001	1.6000e+001
Ra-228	3.7000e-004	3.7000e-002	Rn-219	2.6000e-003	2.6000e-001
Rn-220	2.4000e-003	2.4000e-001	Rn-222	1.6000e-001	1.6000e+001

DOS File: 30AREA.MS4
Run Date: January 28, 1997
Run Time: 4:11 p.m. Tuesday
Title : Glass Spill (Area Source) 100% Eq

Nuclide	curies	ìCi/cm»	Nuclide	curies	ìCi/cm»
Th-227	2.6000e-003	2.6000e-001	Th-228	2.4000e-003	2.4000e-001
Th-230	2.3000e-002	2.3000e+000	Th-231	3.1000e-005	3.1000e-003
Th-232	3.7000e-004	3.7000e-002	Th-234	3.7000e-004	3.7000e-002
Tl-207	2.6000e-003	2.6000e-001	Tl-208	2.4000e-003	2.4000e-001
U-234	3.1000e-004	3.1000e-002	U-235	3.1000e-005	3.1000e-003
U-238	3.7000e-004	3.7000e-002			

========== RESULTS FOR SENSITIVITY REFERENCE CASE (X = 101) ==========

Energy (MeV)	Activity (photons/sec)	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildup
0.015	3.700e+004	9.823e-004	1.059e-003	8.425e-005	9.084e-005
0.02	3.706e+005	1.563e-002	1.685e-002	5.413e-004	5.837e-004
0.03	4.738e+006	3.268e-001	3.541e-001	3.238e-003	3.509e-003
0.04	1.201e+006	1.127e-001	1.221e-001	4.985e-004	5.399e-004
0.05	1.746e+008	2.065e+001	2.232e+001	5.500e-002	5.947e-002
0.06	4.046e+006	5.762e-001	6.206e-001	1.144e-003	1.233e-003
0.08	1.455e+009	2.772e+002	2.959e+002	4.387e-001	4.683e-001
0.1	2.691e+007	6.425e+000	6.798e+000	9.830e-003	1.040e-002
0.15	1.162e+007	4.178e+000	4.371e+000	6.880e-003	7.198e-003
0.2	6.981e+008	3.355e+002	3.473e+002	5.922e-001	6.129e[illegible]
0.3	1.289e+009	9.323e+002	9.569e+002	1.769e+000	1.815e[illegible]0
0.4	2.291e+009	2.215e+003	2.262e+003	4.315e+000	4.408e+000
0.5	1.266e+008	1.532e+002	1.560e+002	3.008e-001	3.062e-001
0.6	2.929e+009	4.263e+003	4.330e+003	8.320e+000	8.451e+000
0.8	5.909e+008	1.149e+003	1.163e+003	2.185e+000	2.213e+000
1.0	1.863e+009	4.535e+003	4.584e+003	8.360e+000	8.450e+000
1.5	1.131e+009	4.142e+003	4.175e+003	6.969e+000	7.024e+000
2.0	1.584e+009	7.746e+003	7.795e+003	1.198e+001	1.205e+001
3.0	8.862e+007	6.511e+002	6.541e+002	8.834e-001	8.874e-001
TOTAL:	1.427e+010	2.643e+004	2.675e+004	4.619e+001	4.677e+001

Page : 3
DOS File: 30AREA.MS4
Run Date: January 28, 1997
Run Time: 4:11 p.m. Tuesday
Title : Glass Spill (Area Source) 100% Eq

SENSITIVITY RESULTS For: X (cm)

Case Number	Sensitivity Variable Value	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildup
1	101.0	2.643e+004	2.675e+004	4.619e+001	4.677e+001
2	130.0	2.040e+004	2.073e+004	3.564e+001	3.624e+001

Use the Display Menu For Energy Group Results For All Cases.

MicroShield 4.21 - Serial #4.21-00915
Fermco

Page : 1
DOS File: 30AREA0.MS4
Run Date: January 28, 1997
Run Time: 4:11 p.m. Tuesday
Duration: 0:00:28

File Ref: _____
Date: ___/___/___
By: _____
Checked: _____

Case Title: Glass Spill (Area Source) 0% Eq

GEOMETRY 5 - Horizontal Rectangular Area

	centimeters	feet and inches	
Dose point coordinate X:	101.0	3.0	3.8
Dose point coordinate Y:	100.0	3.0	3.4
Dose point coordinate Z:	50.0	1.0	7.7
Rectangular area length:	100.0	3.0	3.4
Rectangular area width :	100.0	3.0	3.4
Air Gap:	1.0	0.0	.4

Source Area: 10000 sq cm 10.7639 sq ft. 1550. sq in.

MATERIAL DENSITIES (g/cm^3)

Material	Air Gap	Immersion Shield
Air	0.00122	0.00122

BUILDUP
Method: Buildup Factor Tables
The material reference is Immersion

INTEGRATION PARAMETERS

	Quadrature Order
Z Direction	20
X Direction	20

SOURCE NUCLIDES

Nuclide	curies	ÌCi/cm»	Nuclide	curies	ÌCi/cm»
Ac-227	2.6000e-003	2.6000e-001	Ac-228	3.7000e-004	3.7000e-002
Bi-210	6.7000e-002	6.7000e+000	Bi-211	0.0000e+000	0.0000e+000
Bi-212	2.4000e-003	2.4000e-001	Bi-214	0.0000e+000	0.0000e+000
Fr-223	3.5000e-005	3.5000e-003	Pa-231	1.3000e-003	1.3000e-001
Pa-234	6.0000e-007	6.0000e-005	Pa-234m	3.7000e-004	3.7000e-002
Pb-210	6.7000e-002	6.7000e+000	Pb-211	0.0000e+000	0.0000e+000
Pb-212	2.4000e-003	2.4000e-001	Pb-214	0.0000e+000	0.0000e+000
Po-210	9.3000e-002	9.3000e+000	Po-211	0.0000e+000	0.0000e+000
Po-212	1.6000e-003	1.6000e-001	Po-214	0.0000e+000	0.0000e+000
Po-215	0.0000e+000	0.0000e+000	Po-216	0.0000e+000	0.0000e+000
Po-218	0.0000e+000	0.0000e+000	Ra-223	2.6000e-003	2.6000e-001
Ra-224	2.4000e-003	2.4000e-001	Ra-226	1.6000e-001	1.6000e+001
Ra-228	3.7000e-004	3.7000e-002	Rn-219	0.0000e+000	0.0000e+000
Rn-220	0.0000e+000	0.0000e+000	Rn-222	0.0000e+000	0.0000e+000

DOS File: 30AREA0.MS4
Run Date: January 28, 1997
Run Time: 4:11 p.m. Tuesday
Title : Glass Spill (Area Source) 0% Eq

Nuclide	curies	ÌCi/cm»	Nuclide	curies	ÌCi/cm»
Th-227	2.6000e-003	2.6000e-001	Th-228	2.4000e-003	2.4000e-001
Th-230	2.3000e-002	2.3000e+000	Th-231	3.1000e-005	3.1000e-003
Th-232	3.7000e-004	3.7000e-002	Th-234	3.7000e-004	3.7000e-002
Tl-207	0.0000e+000	0.0000e+000	Tl-208	2.4000e-003	2.4000e-001
U-234	3.1000e-004	3.1000e-002	U-235	3.1000e-005	3.1000e-003
U-238	3.7000e-004	3.7000e-002			

========== RESULTS FOR SENSITIVITY REFERENCE CASE (X = 101) ==========

Energy (MeV)	Activity (photons/sec)	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildup
0.015	3.700e+004	9.823e-004	1.059e-003	8.425e-005	9.084e-005
0.02	3.706e+005	1.563e-002	1.685e-002	5.413e-004	5.837e-004
0.03	4.738e+006	3.268e-001	3.541e-001	3.238e-003	3.509e-003
0.04	1.201e+006	1.127e-001	1.221e-001	4.985e-004	5.399e-004
0.05	1.092e+008	1.291e+001	1.396e+001	3.439e-002	3.718e-002
0.06	4.046e+006	5.762e-001	6.206e-001	1.144e-003	1.233e-003
0.08	1.135e+008	2.163e+001	2.308e+001	3.422e-002	3.653e-002
0.1	2.691e+007	6.425e+000	6.798e+000	9.830e-003	1.040e-002
0.15	1.151e+007	4.138e+000	4.329e+000	6.814e-003	7.129e-003
0.2	2.546e+008	1.224e+002	1.267e+002	2.160e-001	2.236e-001
0.3	5.723e+007	4.140e+001	4.250e+001	7.854e-002	8.061e-002
0.4	2.184e+006	2.111e+000	2.156e+000	4.114e-003	4.202e-003
0.5	2.026e+007	2.453e+001	2.498e+001	4.815e-002	4.902e-002
0.6	7.502e+007	1.092e+002	1.109e+002	2.131e-001	2.164e-001
0.8	2.759e+007	5.365e+001	5.432e+001	1.020e-001	1.033e-001
1.0	9.669e+006	2.354e+001	2.379e+001	4.338e-002	4.385e-002
1.5	4.414e+006	1.616e+001	1.629e+001	2.718e-002	2.740e-002
2.0	1.908e+005	9.329e-001	9.388e-001	1.443e-003	1.452e-003
3.0	8.862e+007	6.511e+002	6.541e+002	8.834e-001	8.874e-001
TOTAL:	8.113e+008	1.091e+003	1.106e+003	1.708e+000	1.734e+000

Page : 3
DOS File: 30AREA0.MS4
Run Date: January 28, 1997
Run Time: 4:11 p.m. Tuesday
Title : Glass Spill (Area Source) 0% Eq

SENSITIVITY RESULTS For: X (cm)

Case Number	Sensitivity Variable Value	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildup
1	101.0	1.091e+003	1.106e+003	1.708e+000	1.734e+000
2	130.0	8.421e+002	8.573e+002	1.318e+000	1.345e+000

Use the Display Menu For Energy Group Results For All Cases.

MicroShield 4.21 - Serial #4.21-00915
Fermco

Page : 1
DOS File: 30AREA10.MS4
Run Date: January 28, 1997
Run Time: 4:12 p.m. Tuesday
Duration: 0:00:28

File Ref: _____
Date: ___/___/___
By: _____
Checked: _____

Case Title: Glass Spill (Area Source) 10% Eq

GEOMETRY 5 - Horizontal Rectangular Area

	centimeters	feet and inches	
Dose point coordinate X:	101.0	3.0	3.8
Dose point coordinate Y:	100.0	3.0	3.4
Dose point coordinate Z:	50.0	1.0	7.7
Rectangular area length:	100.0	3.0	3.4
Rectangular area width :	100.0	3.0	3.4
Air Gap:	1.0	0.0	.4

Source Area: 10000 sq cm 10.7639 sq ft. 1550. sq in.

MATERIAL DENSITIES (g/cm^3)

Material	Air Gap	Immersion Shield
Air	0.00122	0.00122

BUILDUP
Method: Buildup Factor Tables
The material reference is Immersion

INTEGRATION PARAMETERS

	Quadrature Order
Z Direction	20
X Direction	20

SOURCE NUCLIDES

Nuclide	curies	ÌCi/cm»	Nuclide	curies	ÌCi/cm»
Ac-227	2.5999e-003	2.5999e-001	Ac-228	3.7000e-004	3.7000e-002
Bi-210	6.7000e-002	6.7000e+000	Bi-211	2.6012e-003	2.6012e-001
Bi-212	2.3985e-003	2.3985e-001	Bi-214	1.5182e-002	1.5182e+000
Fr-223	3.5879e-005	3.5879e-003	Pa-231	1.3000e-003	1.3000e-001
Pa-234	5.9380e-007	5.9380e-005	Pa-234m	3.7000e-004	3.7000e-002
Pb-210	6.6997e-002	6.6997e+000	Pb-211	2.6012e-003	2.6012e-001
Pb-212	2.3986e-003	2.3986e-001	Pb-214	1.5705e-002	1.5705e+000
Po-210	9.2922e-002	9.2922e+000	Po-211	7.1012e-006	7.1012e-004
Po-212	1.5367e-003	1.5367e-001	Po-214	1.5179e-002	1.5179e+000
Po-215	2.6013e-003	2.6013e-001	Po-216	2.3999e-003	2.3999e-001
Po-218	1.6411e-002	1.6411e+000	Ra-223	2.6013e-003	2.6013e-001
Ra-224	2.3999e-003	2.3999e-001	Ra-226	1.6000e-001	1.6000e+001
Ra-228	3.7000e-004	3.7000e-002	Rn-219	2.6013e-003	2.6013e-001
Rn-220	2.3999e-003	2.3999e-001	Rn-222	1.6490e-002	1.6490e+000

DOS File: 30AREA10.MS4
Run Date: January 28, 1997
Run Time: 4:12 p.m. Tuesday
Title : Glass Spill (Area Source) 10% Eq

Nuclide	curies	ÌCi/cm»	Nuclide	curies	ÌCi/cm»
Th-227	2.5992e-003	2.5992e-001	Th-228	2.3988e-003	2.3988e-001
Th-230	2.3000e-002	2.3000e+000	Th-231	3.1000e-005	3.1000e-003
Th-232	3.7000e-004	3.7000e-002	Th-234	3.7000e-004	3.7000e-002
Tl-207	2.5941e-003	2.5941e-001	Tl-208	8.6177e-004	8.6177e-002
U-234	3.1000e-004	3.1000e-002	U-235	3.1000e-005	3.1000e-003
U-238	3.7000e-004	3.7000e-002			

========== RESULTS FOR SENSITIVITY REFERENCE CASE (X = 101) ==========

Energy (MeV)	Activity (photons/sec)	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildup
0.015	3.700e+004	9.823e-004	1.059e-003	8.425e-005	9.084e-005
0.02	3.708e+005	1.564e-002	1.686e-002	5.416e-004	5.840e-004
0.03	4.738e+006	3.267e-001	3.541e-001	3.238e-003	3.509e-003
0.04	1.200e+006	1.127e-001	1.220e-001	4.983e-004	5.396e-004
0.05	1.156e+008	1.367e+001	1.478e+001	3.642e-002	3.937e-002
0.06	4.046e+006	5.762e-001	6.206e-001	1.144e-003	1.233e-003
0.08	2.450e+008	4.671e+001	4.986e+001	7.391e-002	7.890e-002
0.1	2.691e+007	6.427e+000	6.799e+000	9.832e-003	1.040e-002
0.15	1.162e+007	4.179e+000	4.373e+000	6.882e-003	7.200e-003
0.2	2.979e+008	1.432e+002	1.482e+002	2.527e-001	2.615e-001
0.3	1.830e+008	1.324e+002	1.359e+002	2.511e-001	2.577e-001
0.4	2.474e+008	2.392e+002	2.443e+002	4.661e-001	4.760e-001
0.5	1.883e+007	2.279e+001	2.321e+001	4.474e-002	4.555e-002
0.6	2.980e+008	4.336e+002	4.404e+002	8.463e-001	8.597e-001
0.8	7.651e+007	1.488e+002	1.506e+002	2.830e-001	2.865e-001
1.0	1.852e+008	4.507e+002	4.555e+002	8.308e-001	8.397e-001
1.5	1.114e+008	4.076e+002	4.109e+002	6.858e-001	6.913e-001
2.0	1.505e+008	7.359e+002	7.405e+002	1.138e+000	1.145e+000
3.0	3.182e+007	2.338e+002	2.349e+002	3.172e-001	3.186e-001
TOTAL:	2.010e+009	3.020e+003	3.061e+003	5.248e+000	5.324e+000

Page : 3
DOS File: 30AREA10.MS4
Run Date: January 28, 1997
Run Time: 4:12 p.m. Tuesday
Title : Glass Spill (Area Source) 10% Eq

SENSITIVITY RESULTS For: X (cm)

Case Number	Sensitivity Variable Value	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildup
1	101.0	3.020e+003	3.061e+003	5.248e+000	5.324e+000
2	130.0	2.330e+003	2.372e+003	4.048e+000	4.126e+000

Use the Display Menu For Energy Group Results For All Cases.

MicroShield 4.21 - Serial #4.21-00915
Fermco

Page : 1
DOS File: 30BDC.MS4
Run Date: January 28, 1997
Run Time: 4:23 p.m. Tuesday
Duration: 0:11:20

File Ref: _____
Date: ___/___/___
By: _____
Checked: _____

Case Title: Bottom Drn Cont w Silo 1 glass 100% eq

GEOMETRY 13 - Rectangular Volume

	centimeters	feet and inches	
Dose point coordinate X:	86.18125	2.0	9.9
Dose point coordinate Y:	39.495	1.0	3.5
Dose point coordinate Z:	72.515	2.0	4.5
Rectangular volume width :	145.03	4.0	9.1
Rectangular volume length:	78.99	2.0	7.1
Rectangular volume height:	72.01	2.0	4.4
Shield 1:	5.715	0.0	2.3
Shield 2:	0.47625	0.0	.2
Air Gap:	1.0	0.0	.4

Source Volume: 824941. cm^3 29.1325 cu ft. 50341. cu in.

MATERIAL DENSITIES (g/cm^3)

Material	Source Shield	Shield 1 Slab	Shield 2 Slab	Air Gap
Air				0.00122
Iron			7.86	
HI-ALUMINA		2.499		
Monolith	2.11			

BUILDUP
Method: Buildup Factor Tables
The material reference is Source

INTEGRATION PARAMETERS

	Quadrature Order
X Direction	20
Y Direction	20
Z Direction	20

SOURCE NUCLIDES

Nuclide	curies	ìCi/cm^3	Nuclide	curies	ìCi/cm^3
Ac-227	7.2000e-003	8.7279e-003	Ac-228	1.0000e-003	1.2122e-003
Bi-210	1.9000e-001	2.3032e-001	Bi-211	7.2000e-003	8.7279e-003
Bi-212	6.9000e-003	8.3642e-003	Bi-214	4.5000e-001	5.4549e-001
Fr-223	9.9000e-005	1.2001e-004	Pa-231	3.8000e-003	4.6064e-003
Pa-234	1.7000e-006	2.0608e-006	Pa-234m	1.1000e-003	1.3334e-003
Pb-210	1.9000e-001	2.3032e-001	Pb-211	7.2000e-003	8.7279e-003
Pb-212	6.9000e-003	8.3642e-003	Pb-214	4.5000e-001	5.4549e-001
Po-210	2.6000e-001	3.1517e-001	Po-211	2.0000e-005	2.4244e-005

DOS File: 30BDC.MS4
Run Date: January 28, 1997
Run Time: 4:23 p.m. Tuesday
Title : Bottom Drn Cont w Silo 1 glass 100% eq

Nuclide	curies	ìCi/cm^3	Nuclide	curies	ìCi/cm^3
Po-212	4.4000e-003	5.3337e-003	Po-214	4.5000e-001	5.4549e-001
Po-215	7.2000e-003	8.7279e-003	Po-216	6.9000e-003	8.3642e-003
Po-218	4.5000e-001	5.4549e-001	Ra-223	7.2000e-003	8.7279e-003
Ra-224	6.9000e-003	8.3642e-003	Ra-226	4.5000e-001	5.4549e-001
Ra-228	1.0000e-003	1.2122e-003	Rn-219	7.2000e-003	8.7279e-003
Rn-220	6.9000e-003	8.3642e-003	Rn-222	4.5000e-001	5.4549e-001
Th-227	7.2000e-003	8.7279e-003	Th-228	6.9000e-003	8.3642e-003
Th-230	6.5000e-002	7.8794e-002	Th-231	8.8000e-005	1.0667e-004
Th-232	1.0000e-003	1.2122e-003	Th-234	1.1000e-003	1.3334e-003
Tl-207	7.2000e-003	8.7279e-003	Tl-208	6.9000e-003	8.3642e-003
U-234	8.7000e-004	1.0546e-003	U-235	8.8000e-005	1.0667e-004
U-238	1.1000e-003	1.3334e-003			

========= RESULTS FOR SENSITIVITY REFERENCE CASE (X = 86.1813) =========

Energy (MeV)	Activity (photons/sec)	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildu
0.015	1.026e+005	4.684e-141	3.941e-028	4.018e-142	3.380e-029
0.02	1.053e+006	1.274e-065	5.865e-027	4.412e-067	2.032e-028
0.03	1.382e+007	1.405e-022	1.602e-022	1.392e-024	1.587e-024
0.04	3.433e+006	1.116e-012	1.414e-012	4.936e-015	6.25[illegible]15
0.05	4.932e+008	2.854e-006	4.101e-006	7.603e-009	1.093e-008
0.06	1.149e+007	7.135e-006	1.162e-005	1.417e-008	2.308e-008
0.08	4.091e+009	1.903e-001	3.952e-001	3.011e-004	6.254e-004
0.1	7.515e+007	1.126e-002	2.926e-002	1.723e-005	4.476e-005
0.15	3.232e+007	5.616e-002	2.127e-001	9.248e-005	3.503e-004
0.2	1.966e+009	1.196e+001	5.655e+001	2.111e-002	9.981e-002
0.3	3.623e+009	8.984e+001	4.804e+002	1.704e-001	9.114e-001
0.4	6.441e+009	3.695e+002	1.953e+003	7.199e-001	3.805e+00
0.5	3.571e+008	3.710e+001	1.854e+002	7.282e-002	3.639e-00
0.6	8.243e+009	1.358e+003	6.350e+003	2.651e+000	1.239e+00
0.8	1.663e+009	5.518e+002	2.275e+003	1.049e+000	4.326e+00
1.0	5.240e+009	2.945e+003	1.090e+004	5.429e+000	2.009e+00
1.5	3.182e+009	4.503e+003	1.365e+004	7.576e+000	2.297e+00
2.0	4.456e+009	1.162e+004	3.120e+004	1.797e+001	4.824e+00
3.0	2.548e+008	1.469e+003	3.348e+003	1.993e+000	4.542e+00
TOTAL:	4.015e+010	2.295e+004	7.040e+004	3.765e+001	1.177e+002

DOS File: 30BDC.MS4
Run Date: January 28, 1997
Run Time: 4:23 p.m. Tuesday
Title : Bottom Drn Cont w Silo 1 glass 100% eq

607

SENSITIVITY RESULTS For: X (cm)

Case Number	Sensitivity Variable Value	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildup
1	86.18125	2.295e+004	7.040e+004	3.765e+001	1.177e+002
2	115.1813	1.608e+004	4.553e+004	2.653e+001	7.665e+001

Use the Display Menu For Energy Group Results For All Cases.

MicroShield 4.21 - Serial #4.21-00915
Fermco

Page : 1
DOS File: 30BDC0.MS4
Run Date: January 28, 1997
Run Time: 4:34 p.m. Tuesday
Duration: 0:11:19

File Ref: _____
Date: ___/___/___
By: _____
Checked: _____

Case Title: Bottom Drn Cont w Silo 1 glass 0% eq

GEOMETRY 13 - Rectangular Volume

	centimeters	feet and inches	
Dose point coordinate X:	86.18125	2.0	9.9
Dose point coordinate Y:	39.495	1.0	3.5
Dose point coordinate Z:	72.515	2.0	4.5
Rectangular volume width :	145.03	4.0	9.1
Rectangular volume length:	78.99	2.0	7.1
Rectangular volume height:	72.01	2.0	4.4
Shield 1:	5.715	0.0	2.3
Shield 2:	0.47625	0.0	.2
Air Gap:	1.0	0.0	.4

Source Volume: 824941. cm^3 29.1325 cu ft. 50341. cu in.

MATERIAL DENSITIES (g/cm^3)

Material	Source Shield	Shield 1 Slab	Shield 2 Slab	Air Gap
Air				0.00122
Iron			7.86	
HI-ALUMINA		2.499		
Monolith	2.11			

BUILDUP
Method: Buildup Factor Tables
The material reference is Source

INTEGRATION PARAMETERS

	Quadrature Order
X Direction	20
Y Direction	20
Z Direction	20

SOURCE NUCLIDES

Nuclide	curies	ìCi/cm^3	Nuclide	curies	ìCi/cm^3
Ac-227	7.2000e-003	8.7279e-003	Ac-228	1.0000e-003	1.2122e-003
Bi-210	1.9000e-001	2.3032e-001	Bi-211	0.0000e+000	0.0000e+000
Bi-212	6.9000e-003	8.3642e-003	Bi-214	0.0000e+000	0.0000e+000
Fr-223	9.9000e-005	1.2001e-004	Pa-231	3.8000e-003	4.6064e-003
Pa-234	1.7000e-006	2.0608e-006	Pa-234m	1.1000e-003	1.3334e-003
Pb-210	1.9000e-001	2.3032e-001	Pb-211	0.0000e+000	0.0000e+000
Pb-212	6.9000e-003	8.3642e-003	Pb-214	0.0000e+000	0.0000e+000
Po-210	2.6000e-001	3.1517e-001	Po-211	0.0000e+000	0.0000e+000

DOS File: 30BDC0.MS4
Run Date: January 28, 1997
Run Time: 4:34 p.m. Tuesday
Title : Bottom Drn Cont w Silo 1 glass 0% eq

Nuclide	curies	ÌCi/cm^3	Nuclide	curies	ÌCi/cm^3
Po-212	4.4000e-003	5.3337e-003	Po-214	0.0000e+000	0.0000e+000
Po-215	0.0000e+000	0.0000e+000	Po-216	0.0000e+000	0.0000e+000
Po-218	0.0000e+000	0.0000e+000	Ra-223	7.2000e-003	8.7279e-003
Ra-224	6.9000e-003	8.3642e-003	Ra-226	4.5000e-001	5.4549e-001
Ra-228	1.0000e-003	1.2122e-003	Rn-219	0.0000e+000	0.0000e+000
Rn-220	0.0000e+000	0.0000e+000	Rn-222	0.0000e+000	0.0000e+000
Th-227	7.2000e-003	8.7279e-003	Th-228	6.9000e-003	8.3642e-003
Th-230	6.5000e-002	7.8794e-002	Th-231	8.8000e-005	1.0667e-004
Th-232	1.0000e-003	1.2122e-003	Th-234	1.1000e-003	1.3334e-003
Tl-207	0.0000e+000	0.0000e+000	Tl-208	6.9000e-003	8.3642e-003
U-234	8.7000e-004	1.0546e-003	U-235	8.8000e-005	1.0667e-004
U-238	1.1000e-003	1.3334e-003			

========= RESULTS FOR SENSITIVITY REFERENCE CASE (X = 86.1813) =========

Energy (MeV)	Activity (photons/sec)	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildup
0.015	1.026e+005	4.684e-141	3.941e-028	4.018e-142	3.380e-029
0.02	1.053e+006	1.274e-065	5.865e-027	4.412e-067	2.032e-028
0.03	1.382e+007	1.405e-022	1.602e-022	1.392e-024	1.587[redacted]4
0.04	3.433e+006	1.116e-012	1.414e-012	4.936e-015	6.253[redacted]5
0.05	3.091e+008	1.789e-006	2.571e-006	4.766e-009	6.848e-009
0.06	1.149e+007	7.135e-006	1.162e-005	1.417e-008	2.308e-008
0.08	3.197e+008	1.487e-002	3.089e-002	2.353e-005	4.888e-005
0.1	7.515e+007	1.126e-002	2.926e-002	1.723e-005	4.476e-005
0.15	3.201e+007	5.562e-002	2.107e-001	9.159e-005	3.469e-004
0.2	7.182e+008	4.370e+000	2.066e+001	7.713e-003	3.647e-002
0.3	1.600e+008	3.967e+000	2.122e+001	7.525e-003	4.025e-002
0.4	6.048e+006	3.469e-001	1.834e+000	6.759e-004	3.573e-003
0.5	5.811e+007	6.038e+000	3.017e+001	1.185e-002	5.923e-002
0.6	2.156e+008	3.553e+001	1.661e+002	6.935e-002	3.242e-001
0.8	7.904e+007	2.622e+001	1.081e+002	4.988e-002	2.056e-001
1.0	2.647e+007	1.488e+001	5.508e+001	2.743e-002	1.015e-001
1.5	1.245e+007	1.762e+001	5.342e+001	2.965e-002	8.987e-002
2.0	5.461e+005	1.424e+000	3.823e+000	2.202e-003	5.912e-003
3.0	2.548e+008	1.469e+003	3.348e+003	1.993e+000	4.542e+000
TOTAL:	2.297e+009	1.580e+003	3.808e+003	2.200e+000	5.409e+000

Page : 3
DOS File: 30BDC0.MS4
Run Date: January 28, 1997
Run Time: 4:34 p.m. Tuesday
Title : Bottom Drn Cont w Silo 1 glass 0% eq

SENSITIVITY RESULTS For: X (cm)

Case Number	Sensitivity Variable Value	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildup
1	86.18125	1.580e+003	3.808e+003	2.200e+000	5.409e+000
2	115.1813	1.027e+003	2.298e+003	1.437e+000	3.291e+000

Use the Display Menu For Energy Group Results For All Cases.

MicroShield 4.21 - Serial #4.21-00915
Fermco

Page : 1
DOS File: 30BDC10.MS4
Run Date: January 28, 1997
Run Time: 4:46 p.m. Tuesday
Duration: 0:11:19

File Ref: _____
Date: ___/___/___
By: _____
Checked: _____

Case Title: Bottom Drn Cont w Silo 1 glass 10% eq

GEOMETRY 13 - Rectangular Volume

	centimeters	feet and inches	
Dose point coordinate X:	86.18125	2.0	9.9
Dose point coordinate Y:	39.495	1.0	3.5
Dose point coordinate Z:	72.515	2.0	4.5
Rectangular volume width :	145.03	4.0	9.1
Rectangular volume length:	78.99	2.0	7.1
Rectangular volume height:	72.01	2.0	4.4
Shield 1:	5.715	0.0	2.3
Shield 2:	0.47625	0.0	.2
Air Gap:	1.0	0.0	.4

Source Volume: 824941. cm^3 29.1325 cu ft. 50341. cu in.

MATERIAL DENSITIES (g/cm^3)

Material	Source Shield	Shield 1 Slab	Shield 2 Slab	Air Gap
Air				0.00122
Iron			7.86	
HI-ALUMINA		2.499		
Monolith	2.11			

BUILDUP
Method: Buildup Factor Tables
The material reference is Source

INTEGRATION PARAMETERS

	Quadrature Order
X Direction	20
Y Direction	20
Z Direction	20

SOURCE NUCLIDES

Nuclide	curies	µCi/cm^3	Nuclide	curies	µCi/cm^3
Ac-227	7.1998e-003	8.7277e-003	Ac-228	1.0000e-003	1.2122e-003
Bi-210	1.9000e-001	2.3032e-001	Bi-211	7.2033e-003	8.7319e-003
Bi-212	6.8956e-003	8.3589e-003	Bi-214	4.2699e-002	5.1761e-002
Fr-223	9.9358e-005	1.2044e-004	Pa-231	3.8000e-003	4.6064e-003
Pa-234	1.7465e-006	2.1171e-006	Pa-234m	1.1000e-003	1.3334e-003
Pb-210	1.8999e-001	2.3031e-001	Pb-211	7.2033e-003	8.7319e-003
Pb-212	6.8960e-003	8.3594e-003	Pb-214	4.4171e-002	5.3545e-002
Po-210	2.5979e-001	3.1492e-001	Po-211	1.9665e-005	2.3838e-005

DOS File: 30BDC10.MS4
Run Date: January 28, 1997
Run Time: 4:46 p.m. Tuesday
Title : Bottom Drn Cont w Silo 1 glass 10% eq

Nuclide	curies	ìCi/cm^3	Nuclide	curies	ìCi/cm^3
Po-212	4.4180e-003	5.3556e-003	Po-214	4.2690e-002	5.1750e-002
Po-215	7.2035e-003	8.7322e-003	Po-216	6.8998e-003	8.3640e-003
Po-218	4.6155e-002	5.5950e-002	Ra-223	7.2035e-003	8.7322e-003
Ra-224	6.8998e-003	8.3640e-003	Ra-226	4.5000e-001	5.4549e-001
Ra-228	1.0000e-003	1.2122e-003	Rn-219	7.2035e-003	8.7322e-003
Rn-220	6.8998e-003	8.3640e-003	Rn-222	4.6379e-002	5.6221e-002
Th-227	7.1978e-003	8.7252e-003	Th-228	6.8965e-003	8.3600e-003
Th-230	6.5000e-002	7.8794e-002	Th-231	8.8000e-005	1.0667e-004
Th-232	1.0000e-003	1.2122e-003	Th-234	1.1000e-003	1.3334e-003
Tl-207	7.1836e-003	8.7080e-003	Tl-208	2.4776e-003	3.0034e-003
U-234	8.7000e-004	1.0546e-003	U-235	8.8000e-005	1.0667e-004
U-238	1.1000e-003	1.3334e-003			

========= RESULTS FOR SENSITIVITY REFERENCE CASE (X = 86.1813) =========

Energy (MeV)	Activity (photons/sec)	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildup
0.015	1.026e+005	4.684e-141	3.941e-028	4.018e-142	3.380e-029
0.02	1.053e+006	1.274e-065	5.865e-027	4.412e-067	2.032e-028
0.03	1.382e+007	1.405e-022	1.602e-022	1.392e-024	1.587e-024
0.04	3.431e+006	1.115e-012	1.413e-012	4.933e-015	6.250e-015
0.05	3.272e+008	1.893e-006	2.721e-006	5.044e-009	7.248e-009
0.06	1.149e+007	7.135e-006	1.162e-005	1.417e-008	2.308e-008
0.08	6.894e+008	3.206e-002	6.660e-002	5.074e-005	1.054e-004
0.1	7.517e+007	1.126e-002	2.926e-002	1.723e-005	4.477e-005
0.15	3.233e+007	5.618e-002	2.128e-001	9.251e-005	3.504e-004
0.2	8.398e+008	5.110e+000	2.416e+001	9.018e-003	4.264e-002
0.3	5.129e+008	1.272e+001	6.802e+001	2.412e-002	1.290e-001
0.4	6.947e+008	3.985e+001	2.106e+002	7.764e-002	4.104e-001
0.5	5.330e+007	5.538e+000	2.767e+001	1.087e-002	5.432e-002
0.6	8.397e+008	1.384e+002	6.468e+002	2.701e-001	1.263e+000
0.8	2.159e+008	7.164e+001	2.954e+002	1.363e-001	5.618e-001
1.0	5.200e+008	2.923e+002	1.082e+003	5.388e-001	1.994e+000
1.5	3.132e+008	4.432e+002	1.344e+003	7.456e-001	2.260e+000
2.0	4.233e+008	1.104e+003	2.964e+003	1.707e+000	4.583e+000
3.0	9.149e+007	5.275e+002	1.202e+003	7.157e-001	1.631e+000
TOTAL:	5.658e+009	2.640e+003	7.864e+003	4.235e+000	1.293e+001

Page : 3
DOS File: 30BDC10.MS4
Run Date: January 28, 1997
Run Time: 4:46 p.m. Tuesday
Title : Bottom Drn Cont w Silo 1 glass 10% eq

SENSITIVITY RESULTS For: X (cm)

Case Number	Sensitivity Variable Value	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildup
1	86.18125	2.640e+003	7.864e+003	4.235e+000	1.293e+001
2	115.1813	1.832e+003	5.054e+003	2.960e+000	8.377e+000

Use the Display Menu For Energy Group Results For All Cases.

MicroShield 4.21 - Serial #4.21-00915
Fermco

Page : 1
DOS File: 30BDCTOP.MS4
Run Date: January 28, 1997
Run Time: 4:57 p.m. Tuesday
Duration: 0:11:13

File Ref: _____
Date: __/__/__
By: _____
Checked: _____

Case Title: Bottom Drn Cont w Silo 1 glass 100% eq (TOP)

GEOMETRY 13 - Rectangular Volume

	centimeters	feet and inches	
Dose point coordinate X:	79.99	2.0	7.5
Dose point coordinate Y:	72.515	2.0	4.5
Dose point coordinate Z:	36.005	1.0	2.2
Rectangular volume width :	72.01	2.0	4.4
Rectangular volume length:	78.99	2.0	7.1
Rectangular volume height:	145.03	4.0	9.1
Air Gap:	1.0	0.0	.4

Source Volume: 824941. cm^3 29.1325 cu ft. 50341. cu in.

MATERIAL DENSITIES (g/cm^3)

Material	Source Shield	Air Gap
Air		0.00122
Monolith	2.11	

BUILDUP
Method: Buildup Factor Tables
The material reference is Source

INTEGRATION PARAMETERS

	Quadrature Order
X Direction	20
Y Direction	20
Z Direction	20

SOURCE NUCLIDES

Nuclide	curies	ìCi/cm^3	Nuclide	curies	ìCi/cm^3
Ac-227	7.2000e-003	8.7279e-003	Ac-228	1.0000e-003	1.2122e-003
Bi-210	1.9000e-001	2.3032e-001	Bi-211	7.2000e-003	8.7279e-003
Bi-212	6.9000e-003	8.3642e-003	Bi-214	4.5000e-001	5.4549e-001
Fr-223	9.9000e-005	1.2001e-004	Pa-231	3.8000e-003	4.6064e-003
Pa-234	1.7000e-006	2.0608e-006	Pa-234m	1.1000e-003	1.3334e-003
Pb-210	1.9000e-001	2.3032e-001	Pb-211	7.2000e-003	8.7279e-003
Pb-212	6.9000e-003	8.3642e-003	Pb-214	4.5000e-001	5.4549e-001
Po-210	2.6000e-001	3.1517e-001	Po-211	2.0000e-005	2.4244e-005
Po-212	4.4000e-003	5.3337e-003	Po-214	4.5000e-001	5.4549e-001
Po-215	7.2000e-003	8.7279e-003	Po-216	6.9000e-003	8.3642e-003
Po-218	4.5000e-001	5.4549e-001	Ra-223	7.2000e-003	8.7279e-003
Ra-224	6.9000e-003	8.3642e-003	Ra-226	4.5000e-001	5.4549e-001

DOS File: 30BDCTOP.MS4
Run Date: January 28, 1997
Run Time: 4:57 p.m. Tuesday
Title : Bottom Drn Cont w Silo 1 glass 100% eq (TOP)

Nuclide	curies	ÌCi/cm^3	Nuclide	curies	ÌCi/cm^3
Ra-228	1.0000e-003	1.2122e-003	Rn-219	7.2000e-003	8.7279e-003
Rn-220	6.9000e-003	8.3642e-003	Rn-222	4.5000e-001	5.4549e-001
Th-227	7.2000e-003	8.7279e-003	Th-228	6.9000e-003	8.3642e-003
Th-230	6.5000e-002	7.8794e-002	Th-231	8.8000e-005	1.0667e-004
Th-232	1.0000e-003	1.2122e-003	Th-234	1.1000e-003	1.3334e-003
Tl-207	7.2000e-003	8.7279e-003	Tl-208	6.9000e-003	8.3642e-003
U-234	8.7000e-004	1.0546e-003	U-235	8.8000e-005	1.0667e-004
U-238	1.1000e-003	1.3334e-003			

========== RESULTS FOR SENSITIVITY REFERENCE CASE (X = 79.99) ==========

Energy (MeV)	Activity (photons/sec)	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildu
0.015	1.026e+005	4.855e-012	4.933e-012	4.164e-013	4.231e-013
0.02	1.053e+006	4.605e-008	4.761e-008	1.595e-009	1.649e-009
0.03	1.382e+007	2.223e-003	2.382e-003	2.203e-005	2.361e-005
0.04	3.433e+006	2.316e-003	2.625e-003	1.024e-005	1.161e-005
0.05	4.932e+008	1.681e+000	2.012e+000	4.478e-003	5.360e-003
0.06	1.149e+007	1.011e-001	1.268e-001	2.009e-004	2.519e-004
0.08	4.091e+009	1.158e+002	1.602e+002	1.833e-001	2.53[illegible]01
0.1	7.515e+007	1.730e+000	2.757e+000	2.647e-003	4.21[illegible]03
0.15	3.232e+007	3.063e+000	5.582e+000	5.045e-003	9.192e-003
0.2	1.966e+009	4.277e+002	8.552e+002	7.549e-001	1.509e+000
0.3	3.623e+009	2.050e+003	4.395e+003	3.889e+000	8.336e+000
0.4	6.441e+009	6.374e+003	1.379e+004	1.242e+001	2.686e+001
0.5	3.571e+008	5.218e+002	1.112e+003	1.024e+000	2.184e+000
0.6	8.243e+009	1.627e+004	3.399e+004	3.176e+001	6.634e+001
0.8	1.663e+009	5.183e+003	1.039e+004	9.859e+000	1.976e+00
1.0	5.240e+009	2.311e+004	4.463e+004	4.259e+001	8.227e+00
1.5	3.182e+009	2.608e+004	4.679e+004	4.388e+001	7.872e+00
2.0	4.456e+009	5.584e+004	9.579e+004	8.636e+001	1.481e+00
3.0	2.548e+008	5.674e+003	9.089e+003	7.698e+000	1.233e+00
TOTAL:	4.015e+010	1.417e+005	2.610e+005	2.404e+002	4.467e+00

DOS File: 30BDCTOP.MS4
Run Date: January 28, 1997
Run Time: 4:57 p.m. Tuesday
Title : Bottom Drn Cont w Silo 1 glass 100% eq (TOP)

SENSITIVITY RESULTS For: X (cm)

Case Number	Sensitivity Variable Value	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildup
1	79.99	1.417e+005	2.610e+005	2.404e+002	4.467e+002
2	108.99	6.539e+004	1.156e+005	1.113e+002	1.983e+002

Use the Display Menu For Energy Group Results For All Cases.

MicroShield 4.21 - Serial #4.21-00915
Fermco

Page : 1
DOS File: 30BTOP0.MS4
Run Date: January 28, 1997
Run Time: 5:08 p.m. Tuesday
Duration: 0:11:15

File Ref: _____
Date: ___/___/___
By: _____
Checked: _____

Case Title: Bottom Drn Cont w Silo 1 glass 0% eq (TOP)

GEOMETRY 13 - Rectangular Volume

	centimeters	feet and inches	
Dose point coordinate X:	79.99	2.0	7.5
Dose point coordinate Y:	72.515	2.0	4.5
Dose point coordinate Z:	36.005	1.0	2.2
Rectangular volume width :	72.01	2.0	4.4
Rectangular volume length:	78.99	2.0	7.1
Rectangular volume height:	145.03	4.0	9.1
Air Gap:	1.0	0.0	.4

Source Volume: 824941. cm^3 29.1325 cu ft. 50341. cu in.

MATERIAL DENSITIES (g/cm^3)

Material	Source Shield	Air Gap
Air		0.00122
Monolith	2.11	

BUILDUP
Method: Buildup Factor Tables
The material reference is Source

INTEGRATION PARAMETERS

	Quadrature Order
X Direction	20
Y Direction	20
Z Direction	20

SOURCE NUCLIDES

Nuclide	curies	ìCi/cm^3	Nuclide	curies	ìCi/cm^3
Ac-227	7.2000e-003	8.7279e-003	Ac-228	1.0000e-003	1.2122e-003
Bi-210	1.9000e-001	2.3032e-001	Bi-211	0.0000e+000	0.0000e+000
Bi-212	6.9000e-003	8.3642e-003	Bi-214	0.0000e+000	0.0000e+000
Fr-223	9.9000e-005	1.2001e-004	Pa-231	3.8000e-003	4.6064e-003
Pa-234	1.7000e-006	2.0608e-006	Pa-234m	1.1000e-003	1.3334e-003
Pb-210	1.9000e-001	2.3032e-001	Pb-211	0.0000e+000	0.0000e+000
Pb-212	6.9000e-003	8.3642e-003	Pb-214	0.0000e+000	0.0000e+000
Po-210	2.6000e-001	3.1517e-001	Po-211	0.0000e+000	0.0000e+000
Po-212	4.4000e-003	5.3337e-003	Po-214	0.0000e+000	0.0000e+000
Po-215	0.0000e+000	0.0000e+000	Po-216	0.0000e+000	0.0000e+000
Po-218	0.0000e+000	0.0000e+000	Ra-223	7.2000e-003	8.7279e-003
Ra-224	6.9000e-003	8.3642e-003	Ra-226	4.5000e-001	5.4549e-001

DOS File: 30BTOP0.MS4
Run Date: January 28, 1997
Run Time: 5:08 p.m. Tuesday
Title : Bottom Drn Cont w Silo 1 glass 0% eq (TOP)

Nuclide	curies	ÌCi/cm^3	Nuclide	curies	ÌCi/cm^3
Ra-228	1.0000e-003	1.2122e-003	Rn-219	0.0000e+000	0.0000e+000
Rn-220	0.0000e+000	0.0000e+000	Rn-222	0.0000e+000	0.0000e+000
Th-227	7.2000e-003	8.7279e-003	Th-228	6.9000e-003	8.3642e-003
Th-230	6.5000e-002	7.8794e-002	Th-231	8.8000e-005	1.0667e-004
Th-232	1.0000e-003	1.2122e-003	Th-234	1.1000e-003	1.3334e-003
Tl-207	0.0000e+000	0.0000e+000	Tl-208	6.9000e-003	8.3642e-003
U-234	8.7000e-004	1.0546e-003	U-235	8.8000e-005	1.0667e-004
U-238	1.1000e-003	1.3334e-003			

========== RESULTS FOR SENSITIVITY REFERENCE CASE (X = 79.99) ==========

Energy (MeV)	Activity (photons/sec)	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildup
0.015	1.026e+005	4.855e-012	4.933e-012	4.164e-013	4.231e-013
0.02	1.053e+006	4.605e-008	4.761e-008	1.595e-009	1.649e-009
0.03	1.382e+007	2.223e-003	2.382e-003	2.203e-005	2.361e-005
0.04	3.433e+006	2.316e-003	2.625e-003	1.024e-005	1.161e-005
0.05	3.091e+008	1.054e+000	1.261e+000	2.807e-003	3.360e-003
0.06	1.149e+007	1.011e-001	1.268e-001	2.009e-004	2.519e-004
0.08	3.197e+008	9.051e+000	1.252e+001	1.432e-002	1.981e[redacted]
0.1	7.515e+007	1.730e+000	2.757e+000	2.647e-003	4.219e[redacted]
0.15	3.201e+007	3.034e+000	5.529e+000	4.996e-003	9.104e-003
0.2	7.182e+008	1.563e+002	3.125e+002	2.759e-001	5.515e-001
0.3	1.600e+008	9.054e+001	1.941e+002	1.717e-001	3.681e-001
0.4	6.048e+006	5.985e+000	1.294e+001	1.166e-002	2.522e-002
0.5	5.811e+007	8.492e+001	1.811e+002	1.667e-001	3.554e-001
0.6	2.156e+008	4.256e+002	8.891e+002	8.308e-001	1.735e+000
0.8	7.904e+007	2.464e+002	4.937e+002	4.686e-001	9.391e-001
1.0	2.647e+007	1.167e+002	2.255e+002	2.152e-001	4.157e-001
1.5	1.245e+007	1.021e+002	1.831e+002	1.717e-001	3.081e-001
2.0	5.461e+005	6.844e+000	1.174e+001	1.058e-002	1.815e-002
3.0	2.548e+008	5.674e+003	9.089e+003	7.698e+000	1.233e+001
TOTAL:	2.297e+009	6.925e+003	1.161e+004	1.005e+001	1.708e+001

Page : 3
DOS File: 30BTOP0.MS4
Run Date: January 28, 1997
Run Time: 5:08 p.m. Tuesday
Title : Bottom Drn Cont w Silo 1 glass 0% eq (TOP)

SENSITIVITY RESULTS For: X (cm)

Case Number	Sensitivity Variable Value	Energy Fluence Rate (MeV/sq cm/sec) No Buildup	Energy Fluence Rate (MeV/sq cm/sec) With Buildup	Exposure Rate In Air (mR/hr) No Buildup	Exposure Rate In Air (mR/hr) With Buildup
1	79.99	6.925e+003	1.161e+004	1.005e+001	1.708e+001
2	108.99	3.124e+003	5.047e+003	4.552e+000	7.458e+000

Use the Display Menu For Energy Group Results For All Cases.

MicroShield 4.21 - Serial #4.21-00915
Fermco

Page : 1 File Ref: _____
DOS File: 30BTOP10.MS4 Date: ___/___/___
Run Date: January 28, 1997 By: _____
Run Time: 5:19 p.m. Tuesday Checked: _____
Duration: 0:11:12

Case Title: Bottom Drn Cont w Silo 1 glass 10% eq (TOP)

GEOMETRY 13 - Rectangular Volume

	centimeters	feet and inches	
Dose point coordinate X:	79.99	2.0	7.5
Dose point coordinate Y:	72.515	2.0	4.5
Dose point coordinate Z:	36.005	1.0	2.2
Rectangular volume width :	72.01	2.0	4.4
Rectangular volume length:	78.99	2.0	7.1
Rectangular volume height:	145.03	4.0	9.1
Air Gap:	1.0	0.0	.4

Source Volume: 824941. cm^3 29.1325 cu ft. 50341. cu in.

MATERIAL DENSITIES (g/cm^3)

Material	Source Shield	Air Gap
Air		0.00122
Monolith	2.11	

BUILDUP
Method: Buildup Factor Tables
The material reference is Source

INTEGRATION PARAMETERS

	Quadrature Order
X Direction	20
Y Direction	20
Z Direction	20

SOURCE NUCLIDES

Nuclide	curies	ìCi/cm^3	Nuclide	curies	ìCi/cm^3
Ac-227	7.1998e-003	8.7277e-003	Ac-228	1.0000e-003	1.2122e-003
Bi-210	1.9000e-001	2.3032e-001	Bi-211	7.2033e-003	8.7319e-003
Bi-212	6.8956e-003	8.3589e-003	Bi-214	4.2699e-002	5.1761e-002
Fr-223	9.9358e-005	1.2044e-004	Pa-231	3.8000e-003	4.6064e-003
Pa-234	1.7465e-006	2.1171e-006	Pa-234m	1.1000e-003	1.3334e-003
Pb-210	1.8999e-001	2.3031e-001	Pb-211	7.2033e-003	8.7319e-003
Pb-212	6.8960e-003	8.3594e-003	Pb-214	4.4171e-002	5.3545e-002
Po-210	2.5979e-001	3.1492e-001	Po-211	1.9665e-005	2.3838e-005
Po-212	4.4180e-003	5.3556e-003	Po-214	4.2690e-002	5.1750e-002
Po-215	7.2035e-003	8.7322e-003	Po-216	6.8998e-003	8.3640e-003
Po-218	4.6155e-002	5.5950e-002	Ra-223	7.2035e-003	8.7322e-003
Ra-224	6.8998e-003	8.3640e-003	Ra-226	4.5000e-001	5.4549e-001

DOS File: 30BTOP10.MS4
Run Date: January 28, 1997
Run Time: 5:19 p.m. Tuesday
Title : Bottom Drn Cont w Silo 1 glass 10% eq (TOP)

Nuclide	curies	ÌCi/cm^3	Nuclide	curies	ÌCi/cm^3
Ra-228	1.0000e-003	1.2122e-003	Rn-219	7.2035e-003	8.7322e-003
Rn-220	6.8998e-003	8.3640e-003	Rn-222	4.6379e-002	5.6221e-002
Th-227	7.1978e-003	8.7252e-003	Th-228	6.8965e-003	8.3600e-003
Th-230	6.5000e-002	7.8794e-002	Th-231	8.8000e-005	1.0667e-004
Th-232	1.0000e-003	1.2122e-003	Th-234	1.1000e-003	1.3334e-003
Tl-207	7.1836e-003	8.7080e-003	Tl-208	2.4776e-003	3.0034e-003
U-234	8.7000e-004	1.0546e-003	U-235	8.8000e-005	1.0667e-004
U-238	1.1000e-003	1.3334e-003			

========== RESULTS FOR SENSITIVITY REFERENCE CASE (X = 79.99) ==========

Energy (MeV)	Activity (photons/sec)	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildup
0.015	1.026e+005	4.855e-012	4.933e-012	4.164e-013	4.231e-013
0.02	1.053e+006	4.604e-008	4.761e-008	1.595e-009	1.649e-009
0.03	1.382e+007	2.222e-003	2.382e-003	2.203e-005	2.361e-005
0.04	3.431e+006	2.315e-003	2.623e-003	1.024e-005	1.160e-005
0.05	3.272e+008	1.115e+000	1.335e+000	2.971e-003	3.556e-003
0.06	1.149e+007	1.011e-001	1.268e-001	2.009e-004	2.519e-004
0.08	6.894e+008	1.952e+001	2.699e+001	3.088e-002	4.270e-002
0.1	7.517e+007	1.731e+000	2.758e+000	2.648e-003	4.219e-003
0.15	3.233e+007	3.065e+000	5.584e+000	5.047e-003	9.196e-003
0.2	8.398e+008	1.828e+002	3.654e+002	3.225e-001	6.449e-001
0.3	5.129e+008	2.902e+002	6.221e+002	5.506e-001	1.180e+000
0.4	6.947e+008	6.874e+002	1.487e+003	1.339e+000	2.897e+000
0.5	5.330e+007	7.788e+001	1.661e+002	1.529e-001	3.259e-001
0.6	8.397e+008	1.658e+003	3.462e+003	3.235e+000	6.758e+000
0.8	2.159e+008	6.731e+002	1.349e+003	1.280e+000	2.566e+000
1.0	5.200e+008	2.293e+003	4.430e+003	4.227e+000	8.165e+000
1.5	3.132e+008	2.567e+003	4.605e+003	4.319e+000	7.748e+000
2.0	4.233e+008	5.305e+003	9.100e+003	8.204e+000	1.407e+001
3.0	9.149e+007	2.037e+003	3.263e+003	2.764e+000	4.428e+000
TOTAL:	5.658e+009	1.580e+004	2.889e+004	2.644e+001	4.884e+001

Page : 3
DOS File: 30BTOP10.MS4
Run Date: January 28, 1997
Run Time: 5:19 p.m. Tuesday
Title : Bottom Drn Cont w Silo 1 glass 10% eq (TOP)

SENSITIVITY RESULTS For: X (cm)

Case Number	Sensitivity Variable Value	Energy Fluence Rate (MeV/sq cm/sec) No Buildup	Energy Fluence Rate (MeV/sq cm/sec) With Buildup	Exposure Rate In Air (mR/hr) No Buildup	Exposure Rate In Air (mR/hr) With Buildup
1	79.99	1.580e+004	2.889e+004	2.644e+001	4.884e+001
2	108.99	7.285e+003	1.279e+004	1.223e+001	2.169e+001

Use the Display Menu For Energy Group Results For All Cases.

MicroShield 4.21 - Serial #4.21-00915
Fermco

Page : 1
DOS File: AREA.MS4
Run Date: January 28, 1997
Run Time: 5:20 p.m. Tuesday
Duration: 0:00:29

File Ref: _____
Date: ___/___/___
By: _____
Checked: _____

Case Title: Glass Spill (Area Source) 100% Eq

GEOMETRY 5 - Horizontal Rectangular Area

	centimeters	feet and inches	
Dose point coordinate X:	101.0	3.0	3.8
Dose point coordinate Y:	100.0	3.0	3.4
Dose point coordinate Z:	50.0	1.0	7.7
Rectangular area length:	100.0	3.0	3.4
Rectangular area width :	100.0	3.0	3.4
Air Gap:	1.0	0.0	.4

Source Area: 10000 sq cm 10.7639 sq ft. 1550. sq in.

MATERIAL DENSITIES (g/cm^3)

Material	Air Gap	Immersion Shield
Air	0.00122	0.00122

BUILDUP
Method: Buildup Factor Tables
The material reference is Immersion

INTEGRATION PARAMETERS

	Quadrature Order
Z Direction	20
X Direction	20

SOURCE NUCLIDES

Nuclide	curies	ÌCi/cm»	Nuclide	curies	ÌCi/cm»
Ac-227	2.6000e-003	2.6000e-001	Ac-228	3.7000e-004	3.7000e-002
Bi-210	6.7000e-002	6.7000e+000	Bi-211	2.6000e-003	2.6000e-001
Bi-212	2.4000e-003	2.4000e-001	Bi-214	1.6000e-001	1.6000e+001
Fr-223	3.5000e-005	3.5000e-003	Pa-231	1.3000e-003	1.3000e-001
Pa-234	6.0000e-007	6.0000e-005	Pa-234m	3.7000e-004	3.7000e-002
Pb-210	6.7000e-002	6.7000e+000	Pb-211	2.6000e-003	2.6000e-001
Pb-212	2.4000e-003	2.4000e-001	Pb-214	1.6000e-001	1.6000e+001
Po-210	9.3000e-002	9.3000e+000	Po-211	7.0000e-006	7.0000e-004
Po-212	1.6000e-003	1.6000e-001	Po-214	1.6000e-001	1.6000e+001
Po-215	2.6000e-003	2.6000e-001	Po-216	2.4000e-003	2.4000e-001
Po-218	1.6000e-001	1.6000e+001	Ra-223	2.6000e-003	2.6000e-001
Ra-224	2.4000e-003	2.4000e-001	Ra-226	1.6000e-001	1.6000e+001
Ra-228	3.7000e-004	3.7000e-002	Rn-219	2.6000e-003	2.6000e-001
Rn-220	2.4000e-003	2.4000e-001	Rn-222	1.6000e-001	1.6000e+001

Page : 2
DOS File: AREA.MS4
Run Date: January 28, 1997
Run Time: 5:20 p.m. Tuesday
Title : Glass Spill (Area Source) 100% Eq

Nuclide	curies	ÌCi/cm»	Nuclide	curies	ÌCi/cm»
Th-227	2.6000e-003	2.6000e-001	Th-228	2.4000e-003	2.4000e-001
Th-230	2.3000e-002	2.3000e+000	Th-231	3.1000e-005	3.1000e-003
Th-232	3.7000e-004	3.7000e-002	Th-234	3.7000e-004	3.7000e-002
Tl-207	2.6000e-003	2.6000e-001	Tl-208	2.4000e-003	2.4000e-001
U-234	3.1000e-004	3.1000e-002	U-235	3.1000e-005	3.1000e-003
U-238	3.7000e-004	3.7000e-002			

========== RESULTS FOR SENSITIVITY REFERENCE CASE (X = 101) ==========

Energy (MeV)	Activity (photons/sec)	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildup
0.015	3.700e+004	9.823e-004	1.059e-003	8.425e-005	9.084e-005
0.02	3.706e+005	1.563e-002	1.685e-002	5.413e-004	5.837e-004
0.03	4.738e+006	3.268e-001	3.541e-001	3.238e-003	3.509e-003
0.04	1.201e+006	1.127e-001	1.221e-001	4.985e-004	5.399e-004
0.05	1.746e+008	2.065e+001	2.232e+001	5.500e-002	5.947e-002
0.06	4.046e+006	5.762e-001	6.206e-001	1.144e-003	1.233e-003
0.08	1.455e+009	2.772e+002	2.959e+002	4.387e-001	4.683e-001
0.1	2.691e+007	6.425e+000	6.798e+000	9.830e-003	1.040e-002
0.15	1.162e+007	4.178e+000	4.371e+000	6.880e-003	7.198e[illegible]
0.2	6.981e+008	3.355e+002	3.473e+002	5.922e-001	6.129e[illegible]
0.3	1.289e+009	9.323e+002	9.569e+002	1.769e+000	1.815e+000
0.4	2.291e+009	2.215e+003	2.262e+003	4.315e+000	4.408e+000
0.5	1.266e+008	1.532e+002	1.560e+002	3.008e-001	3.062e-001
0.6	2.929e+009	4.263e+003	4.330e+003	8.320e+000	8.451e+000
0.8	5.909e+008	1.149e+003	1.163e+003	2.185e+000	2.213e+000
1.0	1.863e+009	4.535e+003	4.584e+003	8.360e+000	8.450e+000
1.5	1.131e+009	4.142e+003	4.175e+003	6.969e+000	7.024e+000
2.0	1.584e+009	7.746e+003	7.795e+003	1.198e+001	1.205e+001
3.0	8.862e+007	6.511e+002	6.541e+002	8.834e-001	8.874e-001
TOTAL:	1.427e+010	2.643e+004	2.675e+004	4.619e+001	4.677e+001

Page : 3
DOS File: AREA.MS4
Run Date: January 28, 1997
Run Time: 5:20 p.m. Tuesday
Title : Glass Spill (Area Source) 100% Eq

SENSITIVITY RESULTS For: X (cm)

Case Number	Sensitivity Variable Value	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildup
1	101.0	2.643e+004	2.675e+004	4.619e+001	4.677e+001
2	200.0	1.176e+004	1.207e+004	2.055e+001	2.110e+001

Use the Display Menu For Energy Group Results For All Cases.

MicroShield 4.21 - Serial #4.21-00915
Fermco

Page : 1
DOS File: AREA0.MS4
Run Date: January 28, 1997
Run Time: 5:20 p.m. Tuesday
Duration: 0:00:29

File Ref: _____
Date: ___/___/___
By: _____
Checked: _____

Case Title: Glass Spill (Area Source) 0% Eq

GEOMETRY 5 - Horizontal Rectangular Area

	centimeters	feet and inches	
Dose point coordinate X:	101.0	3.0	3.8
Dose point coordinate Y:	100.0	3.0	3.4
Dose point coordinate Z:	50.0	1.0	7.7
Rectangular area length:	100.0	3.0	3.4
Rectangular area width :	100.0	3.0	3.4
Air Gap:	1.0	0.0	.4

Source Area: 10000 sq cm 10.7639 sq ft. 1550. sq in.

MATERIAL DENSITIES (g/cm^3)

Material	Air Gap	Immersion Shield
Air	0.00122	0.00122

BUILDUP
Method: Buildup Factor Tables
The material reference is Immersion

INTEGRATION PARAMETERS

	Quadrature Order
Z Direction	20
X Direction	20

SOURCE NUCLIDES

Nuclide	curies	ÌCi/cm»	Nuclide	curies	ÌCi/cm»
Ac-227	2.6000e-003	2.6000e-001	Ac-228	3.7000e-004	3.7000e-002
Bi-210	6.7000e-002	6.7000e+000	Bi-211	0.0000e+000	0.0000e+000
Bi-212	2.4000e-003	2.4000e-001	Bi-214	0.0000e+000	0.0000e+000
Fr-223	3.5000e-005	3.5000e-003	Pa-231	1.3000e-003	1.3000e-001
Pa-234	6.0000e-007	6.0000e-005	Pa-234m	3.7000e-004	3.7000e-002
Pb-210	6.7000e-002	6.7000e+000	Pb-211	0.0000e+000	0.0000e+000
Pb-212	2.4000e-003	2.4000e-001	Pb-214	0.0000e+000	0.0000e+000
Po-210	9.3000e-002	9.3000e+000	Po-211	0.0000e+000	0.0000e+000
Po-212	1.6000e-003	1.6000e-001	Po-214	0.0000e+000	0.0000e+000
Po-215	0.0000e+000	0.0000e+000	Po-216	0.0000e+000	0.0000e+000
Po-218	0.0000e+000	0.0000e+000	Ra-223	2.6000e-003	2.6000e-001
Ra-224	2.4000e-003	2.4000e-001	Ra-226	1.6000e-001	1.6000e+001
Ra-228	3.7000e-004	3.7000e-002	Rn-219	0.0000e+000	0.0000e+000
Rn-220	0.0000e+000	0.0000e+000	Rn-222	0.0000e+000	0.0000e+000

DOS File: AREA0.MS4
Run Date: January 28, 1997
Run Time: 5:20 p.m. Tuesday
Title : Glass Spill (Area Source) 0% Eq

Nuclide	curies	ÌCi/cm»	Nuclide	curies	ÌCi/cm»
Th-227	2.6000e-003	2.6000e-001	Th-228	2.4000e-003	2.4000e-001
Th-230	2.3000e-002	2.3000e+000	Th-231	3.1000e-005	3.1000e-003
Th-232	3.7000e-004	3.7000e-002	Th-234	3.7000e-004	3.7000e-002
Tl-207	0.0000e+000	0.0000e+000	Tl-208	2.4000e-003	2.4000e-001
U-234	3.1000e-004	3.1000e-002	U-235	3.1000e-005	3.1000e-003
U-238	3.7000e-004	3.7000e-002			

========== RESULTS FOR SENSITIVITY REFERENCE CASE (X = 101) ==========

Energy (MeV)	Activity (photons/sec)	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildup
0.015	3.700e+004	9.823e-004	1.059e-003	8.425e-005	9.084e-005
0.02	3.706e+005	1.563e-002	1.685e-002	5.413e-004	5.837e-004
0.03	4.738e+006	3.268e-001	3.541e-001	3.238e-003	3.509e-003
0.04	1.201e+006	1.127e-001	1.221e-001	4.985e-004	5.399e-004
0.05	1.092e+008	1.291e+001	1.396e+001	3.439e-002	3.718e-002
0.06	4.046e+006	5.762e-001	6.206e-001	1.144e-003	1.233e-003
0.08	1.135e+008	2.163e+001	2.308e+001	3.422e-002	3.653e-002
0.1	2.691e+007	6.425e+000	6.798e+000	9.830e-003	1.040e-002
0.15	1.151e+007	4.138e+000	4.329e+000	6.814e-003	7.129e-003
0.2	2.546e+008	1.224e+002	1.267e+002	2.160e-001	2.236e-001
0.3	5.723e+007	4.140e+001	4.250e+001	7.854e-002	8.061e-002
0.4	2.184e+006	2.111e+000	2.156e+000	4.114e-003	4.202e-003
0.5	2.026e+007	2.453e+001	2.498e+001	4.815e-002	4.902e-002
0.6	7.502e+007	1.092e+002	1.109e+002	2.131e-001	2.164e-001
0.8	2.759e+007	5.365e+001	5.432e+001	1.020e-001	1.033e-001
1.0	9.669e+006	2.354e+001	2.379e+001	4.338e-002	4.385e-002
1.5	4.414e+006	1.616e+001	1.629e+001	2.718e-002	2.740e-002
2.0	1.908e+005	9.329e-001	9.388e-001	1.443e-003	1.452e-003
3.0	8.862e+007	6.511e+002	6.541e+002	8.834e-001	8.874e-001
TOTAL:	8.113e+008	1.091e+003	1.106e+003	1.708e+000	1.734e+000

Page : 3
DOS File: AREA0.MS4
Run Date: January 28, 1997
Run Time: 5:20 p.m. Tuesday
Title : Glass Spill (Area Source) 0% Eq

SENSITIVITY RESULTS For: X (cm)

Case Number	Sensitivity Variable Value	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildup
1	101.0	1.091e+003	1.106e+003	1.708e+000	1.734e+000
2	200.0	4.859e+002	4.998e+002	7.596e-001	7.844e-001

Use the Display Menu For Energy Group Results For All Cases.

MicroShield 4.21 - Serial #4.21-00915
Fermco

Page : 1
DOS File: AREA10.MS4
Run Date: January 28, 1997
Run Time: 5:21 p.m. Tuesday
Duration: 0:00:29

File Ref: _____
Date: ___/___/___
By: _____
Checked: _____

Case Title: Glass Spill (Area Source) 10% Eq

GEOMETRY 5 - Horizontal Rectangular Area

	centimeters	feet and inches	
Dose point coordinate X:	101.0	3.0	3.8
Dose point coordinate Y:	100.0	3.0	3.4
Dose point coordinate Z:	50.0	1.0	7.7
Rectangular area length:	100.0	3.0	3.4
Rectangular area width :	100.0	3.0	3.4
Air Gap:	1.0	0.0	.4

Source Area: 10000 sq cm 10.7639 sq ft. 1550. sq in.

MATERIAL DENSITIES (g/cm^3)

Material	Air Gap	Immersion Shield
Air	0.00122	0.00122

BUILDUP
Method: Buildup Factor Tables
The material reference is Immersion

INTEGRATION PARAMETERS

	Quadrature Order
Z Direction	20
X Direction	20

SOURCE NUCLIDES

Nuclide	curies	ÌCi/cm»	Nuclide	curies	ÌCi/cm»
Ac-227	2.5999e-003	2.5999e-001	Ac-228	3.7000e-004	3.7000e-002
Bi-210	6.7000e-002	6.7000e+000	Bi-211	2.6012e-003	2.6012e-001
Bi-212	2.3985e-003	2.3985e-001	Bi-214	1.5182e-002	1.5182e+000
Fr-223	3.5879e-005	3.5879e-003	Pa-231	1.3000e-003	1.3000e-001
Pa-234	5.9380e-007	5.9380e-005	Pa-234m	3.7000e-004	3.7000e-002
Pb-210	6.6997e-002	6.6997e+000	Pb-211	2.6012e-003	2.6012e-001
Pb-212	2.3986e-003	2.3986e-001	Pb-214	1.5705e-002	1.5705e+000
Po-210	9.2922e-002	9.2922e+000	Po-211	7.1012e-006	7.1012e-004
Po-212	1.5367e-003	1.5367e-001	Po-214	1.5179e-002	1.5179e+000
Po-215	2.6013e-003	2.6013e-001	Po-216	2.3999e-003	2.3999e-001
Po-218	1.6411e-002	1.6411e+000	Ra-223	2.6013e-003	2.6013e-001
Ra-224	2.3999e-003	2.3999e-001	Ra-226	1.6000e-001	1.6000e+001
Ra-228	3.7000e-004	3.7000e-002	Rn-219	2.6013e-003	2.6013e-001
Rn-220	2.3999e-003	2.3999e-001	Rn-222	1.6490e-002	1.6490e+000

DOS File: AREA10.MS4
Run Date: January 28, 1997
Run Time: 5:21 p.m. Tuesday
Title : Glass Spill (Area Source) 10% Eq

Nuclide	curies	ÌCi/cm»	Nuclide	curies	ÌCi/cm»
Th-227	2.5992e-003	2.5992e-001	Th-228	2.3988e-003	2.3988e-001
Th-230	2.3000e-002	2.3000e+000	Th-231	3.1000e-005	3.1000e-003
Th-232	3.7000e-004	3.7000e-002	Th-234	3.7000e-004	3.7000e-002
Tl-207	2.5941e-003	2.5941e-001	Tl-208	8.6177e-004	8.6177e-002
U-234	3.1000e-004	3.1000e-002	U-235	3.1000e-005	3.1000e-003
U-238	3.7000e-004	3.7000e-002			

========== RESULTS FOR SENSITIVITY REFERENCE CASE (X = 101) ==========

Energy (MeV)	Activity (photons/sec)	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildup
0.015	3.700e+004	9.823e-004	1.059e-003	8.425e-005	9.084e-005
0.02	3.708e+005	1.564e-002	1.686e-002	5.416e-004	5.840e-004
0.03	4.738e+006	3.267e-001	3.541e-001	3.238e-003	3.509e-003
0.04	1.200e+006	1.127e-001	1.220e-001	4.983e-004	5.396e-004
0.05	1.156e+008	1.367e+001	1.478e+001	3.642e-002	3.937e-002
0.06	4.046e+006	5.762e-001	6.206e-001	1.144e-003	1.233e-003
0.08	2.450e+008	4.671e+001	4.986e+001	7.391e-002	7.890e-002
0.1	2.691e+007	6.427e+000	6.799e+000	9.832e-003	1.040e-002
0.15	1.162e+007	4.179e+000	4.373e+000	6.882e-003	7.200[redacted]
0.2	2.979e+008	1.432e+002	1.482e+002	2.527e-001	2.615[redacted]
0.3	1.830e+008	1.324e+002	1.359e+002	2.511e-001	2.577e-001
0.4	2.474e+008	2.392e+002	2.443e+002	4.661e-001	4.760e-001
0.5	1.883e+007	2.279e+001	2.321e+001	4.474e-002	4.555e-002
0.6	2.980e+008	4.336e+002	4.404e+002	8.463e-001	8.597e-001
0.8	7.651e+007	1.488e+002	1.506e+002	2.830e-001	2.865e-001
1.0	1.852e+008	4.507e+002	4.555e+002	8.308e-001	8.397e-001
1.5	1.114e+008	4.076e+002	4.109e+002	6.858e-001	6.913e-001
2.0	1.505e+008	7.359e+002	7.405e+002	1.138e+000	1.145e+00
3.0	3.182e+007	2.338e+002	2.349e+002	3.172e-001	3.186e-00
TOTAL:	2.010e+009	3.020e+003	3.061e+003	5.248e+000	5.324e+00

607
DOS File: AREA10.MS4
Run Date: January 28, 1997
Run Time: 5:21 p.m. Tuesday
Title : Glass Spill (Area Source) 10% Eq

SENSITIVITY RESULTS For: X (cm)

Case Number	Sensitivity Variable Value	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildup
1	101.0	3.020e+003	3.061e+003	5.248e+000	5.324e+000
2	200.0	1.343e+003	1.382e+003	2.333e+000	2.403e+000

Use the Display Menu For Energy Group Results For All Cases.

MicroShield 4.21 - Serial #4.21-00915
Fermco

Page : 1
DOS File: BDC.MS4
Run Date: January 28, 1997
Run Time: 5:32 p.m. Tuesday
Duration: 0:11:18

File Ref: _____
Date: __/__/__
By: _____
Checked: _____

Case Title: Bottom Drn Cont w Silo 1 glass 100% eq

GEOMETRY 13 - Rectangular Volume

	centimeters	feet and inches	
Dose point coordinate X:	86.18125	2.0	9.9
Dose point coordinate Y:	39.495	1.0	3.5
Dose point coordinate Z:	72.515	2.0	4.5
Rectangular volume width :	145.03	4.0	9.1
Rectangular volume length:	78.99	2.0	7.1
Rectangular volume height:	72.01	2.0	4.4
Shield 1:	5.715	0.0	2.3
Shield 2:	0.47625	0.0	.2
Air Gap:	1.0	0.0	.4

Source Volume: 824941. cm^3 29.1325 cu ft. 50341. cu in.

MATERIAL DENSITIES (g/cm^3)

Material	Source Shield	Shield 1 Slab	Shield 2 Slab	Air Gap
Air				0.00122
Iron			7.86	
HI-ALUMINA		2.499		
Monolith	2.11			

BUILDUP
Method: Buildup Factor Tables
The material reference is Source

INTEGRATION PARAMETERS

	Quadrature Order
X Direction	20
Y Direction	20
Z Direction	20

SOURCE NUCLIDES

Nuclide	curies	ìCi/cm^3	Nuclide	curies	ìCi/cm^3
Ac-227	7.2000e-003	8.7279e-003	Ac-228	1.0000e-003	1.2122e-003
Bi-210	1.9000e-001	2.3032e-001	Bi-211	7.2000e-003	8.7279e-003
Bi-212	6.9000e-003	8.3642e-003	Bi-214	4.5000e-001	5.4549e-001
Fr-223	9.9000e-005	1.2001e-004	Pa-231	3.8000e-003	4.6064e-003
Pa-234	1.7000e-006	2.0608e-006	Pa-234m	1.1000e-003	1.3334e-003
Pb-210	1.9000e-001	2.3032e-001	Pb-211	7.2000e-003	8.7279e-003
Pb-212	6.9000e-003	8.3642e-003	Pb-214	4.5000e-001	5.4549e-001
Po-210	2.6000e-001	3.1517e-001	Po-211	2.0000e-005	2.4244e-005

DOS File: BDC.MS4
Run Date: January 28, 1997
Run Time: 5:32 p.m. Tuesday
Title : Bottom Drn Cont w Silo 1 glass 100% eq

Nuclide	curies	µCi/cm^3	Nuclide	curies	µCi/cm^3
Po-212	4.4000e-003	5.3337e-003	Po-214	4.5000e-001	5.4549e-001
Po-215	7.2000e-003	8.7279e-003	Po-216	6.9000e-003	8.3642e-003
Po-218	4.5000e-001	5.4549e-001	Ra-223	7.2000e-003	8.7279e-003
Ra-224	6.9000e-003	8.3642e-003	Ra-226	4.5000e-001	5.4549e-001
Ra-228	1.0000e-003	1.2122e-003	Rn-219	7.2000e-003	8.7279e-003
Rn-220	6.9000e-003	8.3642e-003	Rn-222	4.5000e-001	5.4549e-001
Th-227	7.2000e-003	8.7279e-003	Th-228	6.9000e-003	8.3642e-003
Th-230	6.5000e-002	7.8794e-002	Th-231	8.8000e-005	1.0667e-004
Th-232	1.0000e-003	1.2122e-003	Th-234	1.1000e-003	1.3334e-003
Tl-207	7.2000e-003	8.7279e-003	Tl-208	6.9000e-003	8.3642e-003
U-234	8.7000e-004	1.0546e-003	U-235	8.8000e-005	1.0667e-004
U-238	1.1000e-003	1.3334e-003			

========= RESULTS FOR SENSITIVITY REFERENCE CASE (X = 86.1813) =========

Energy (MeV)	Activity (photons/sec)	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildup
0.015	1.026e+005	4.684e-141	3.941e-028	4.018e-142	3.380e-029
0.02	1.053e+006	1.274e-065	5.865e-027	4.412e-067	2.032e-028
0.03	1.382e+007	1.405e-022	1.602e-022	1.392e-024	1.587e-024
0.04	3.433e+006	1.116e-012	1.414e-012	4.936e-015	6.253e-015
0.05	4.932e+008	2.854e-006	4.101e-006	7.603e-009	1.093e-008
0.06	1.149e+007	7.135e-006	1.162e-005	1.417e-008	2.308e-008
0.08	4.091e+009	1.903e-001	3.952e-001	3.011e-004	6.254e-004
0.1	7.515e+007	1.126e-002	2.926e-002	1.723e-005	4.476e-005
0.15	3.232e+007	5.616e-002	2.127e-001	9.248e-005	3.503e-004
0.2	1.966e+009	1.196e+001	5.655e+001	2.111e-002	9.981e-002
0.3	3.623e+009	8.984e+001	4.804e+002	1.704e-001	9.114e-001
0.4	6.441e+009	3.695e+002	1.953e+003	7.199e-001	3.805e+000
0.5	3.571e+008	3.710e+001	1.854e+002	7.282e-002	3.639e-001
0.6	8.243e+009	1.358e+003	6.350e+003	2.651e+000	1.239e+001
0.8	1.663e+009	5.518e+002	2.275e+003	1.049e+000	4.326e+000
1.0	5.240e+009	2.945e+003	1.090e+004	5.429e+000	2.009e+001
1.5	3.182e+009	4.503e+003	1.365e+004	7.576e+000	2.297e+001
2.0	4.456e+009	1.162e+004	3.120e+004	1.797e+001	4.824e+001
3.0	2.548e+008	1.469e+003	3.348e+003	1.993e+000	4.542e+000
TOTAL:	4.015e+010	2.295e+004	7.040e+004	3.765e+001	1.177e+002

Page : 3
DOS File: BDC.MS4
Run Date: January 28, 1997
Run Time: 5:32 p.m. Tuesday
Title : Bottom Drn Cont w Silo 1 glass 100% eq

SENSITIVITY RESULTS For: X (cm)

Case Number	Sensitivity Variable Value	Energy Fluence Rate (MeV/sq cm/sec) No Buildup	Energy Fluence Rate (MeV/sq cm/sec) With Buildup	Exposure Rate In Air (mR/hr) No Buildup	Exposure Rate In Air (mR/hr) With Buildup
1	86.18125	2.295e+004	7.040e+004	3.765e+001	1.177e+002
2	185.1813	5.304e+003	1.426e+004	8.783e+000	2.407e+001

Use the Display Menu For Energy Group Results For All Cases.

MicroShield 4.21 - Serial #4.21-00915
Fermco

Page : 1
DOS File: BDC0.MS4
Run Date: January 28, 1997
Run Time: 5:43 p.m. Tuesday
Duration: 0:11:17

File Ref: _____
Date: ___/___/___
By: _____
Checked: _____

Case Title: Bottom Drn Cont w Silo 1 glass 0% eq

GEOMETRY 13 - Rectangular Volume

	centimeters	feet and inches	
Dose point coordinate X:	86.18125	2.0	9.9
Dose point coordinate Y:	39.495	1.0	3.5
Dose point coordinate Z:	72.515	2.0	4.5
Rectangular volume width :	145.03	4.0	9.1
Rectangular volume length:	78.99	2.0	7.1
Rectangular volume height:	72.01	2.0	4.4
Shield 1:	5.715	0.0	2.3
Shield 2:	0.47625	0.0	.2
Air Gap:	1.0	0.0	.4

Source Volume: 824941. cm^3 29.1325 cu ft. 50341. cu in.

MATERIAL DENSITIES (g/cm^3)

Material	Source Shield	Shield 1 Slab	Shield 2 Slab	Air Gap
Air				0.00122
Iron			7.86	
HI-ALUMINA		2.499		
Monolith	2.11			

BUILDUP
Method: Buildup Factor Tables
The material reference is Source

INTEGRATION PARAMETERS

	Quadrature Order
X Direction	20
Y Direction	20
Z Direction	20

SOURCE NUCLIDES

Nuclide	curies	ìCi/cm^3	Nuclide	curies	ìCi/cm^3
Ac-227	7.2000e-003	8.7279e-003	Ac-228	1.0000e-003	1.2122e-003
Bi-210	1.9000e-001	2.3032e-001	Bi-211	0.0000e+000	0.0000e+000
Bi-212	6.9000e-003	8.3642e-003	Bi-214	0.0000e+000	0.0000e+000
Fr-223	9.9000e-005	1.2001e-004	Pa-231	3.8000e-003	4.6064e-003
Pa-234	1.7000e-006	2.0608e-006	Pa-234m	1.1000e-003	1.3334e-003
Pb-210	1.9000e-001	2.3032e-001	Pb-211	0.0000e+000	0.0000e+000
Pb-212	6.9000e-003	8.3642e-003	Pb-214	0.0000e+000	0.0000e+000
Po-210	2.6000e-001	3.1517e-001	Po-211	0.0000e+000	0.0000e+000

Page : 2
DOS File: BDC0.MS4
Run Date: January 28, 1997
Run Time: 5:43 p.m. Tuesday
Title : Bottom Drn Cont w Silo 1 glass 0% eq

Nuclide	curies	ÌCi/cm^3	Nuclide	curies	ÌCi/cm^3
Po-212	4.4000e-003	5.3337e-003	Po-214	0.0000e+000	0.0000e+000
Po-215	0.0000e+000	0.0000e+000	Po-216	0.0000e+000	0.0000e+000
Po-218	0.0000e+000	0.0000e+000	Ra-223	7.2000e-003	8.7279e-003
Ra-224	6.9000e-003	8.3642e-003	Ra-226	4.5000e-001	5.4549e-001
Ra-228	1.0000e-003	1.2122e-003	Rn-219	0.0000e+000	0.0000e+000
Rn-220	0.0000e+000	0.0000e+000	Rn-222	0.0000e+000	0.0000e+000
Th-227	7.2000e-003	8.7279e-003	Th-228	6.9000e-003	8.3642e-003
Th-230	6.5000e-002	7.8794e-002	Th-231	8.8000e-005	1.0667e-004
Th-232	1.0000e-003	1.2122e-003	Th-234	1.1000e-003	1.3334e-003
Tl-207	0.0000e+000	0.0000e+000	Tl-208	6.9000e-003	8.3642e-003
U-234	8.7000e-004	1.0546e-003	U-235	8.8000e-005	1.0667e-004
U-238	1.1000e-003	1.3334e-003			

========== RESULTS FOR SENSITIVITY REFERENCE CASE (X = 86.1813) ==========

Energy (MeV)	Activity (photons/sec)	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildup
0.015	1.026e+005	4.684e-141	3.941e-028	4.018e-142	3.380e-029
0.02	1.053e+006	1.274e-065	5.865e-027	4.412e-067	2.032e-028
0.03	1.382e+007	1.405e-022	1.602e-022	1.392e-024	1.587[illegible]
0.04	3.433e+006	1.116e-012	1.414e-012	4.936e-015	6.253e[illegible]5
0.05	3.091e+008	1.789e-006	2.571e-006	4.766e-009	6.848e-009
0.06	1.149e+007	7.135e-006	1.162e-005	1.417e-008	2.308e-008
0.08	3.197e+008	1.487e-002	3.089e-002	2.353e-005	4.888e-005
0.1	7.515e+007	1.126e-002	2.926e-002	1.723e-005	4.476e-005
0.15	3.201e+007	5.562e-002	2.107e-001	9.159e-005	3.469e-004
0.2	7.182e+008	4.370e+000	2.066e+001	7.713e-003	3.647e-002
0.3	1.600e+008	3.967e+000	2.122e+001	7.525e-003	4.025e-002
0.4	6.048e+006	3.469e-001	1.834e+000	6.759e-004	3.573e-003
0.5	5.811e+007	6.038e+000	3.017e+001	1.185e-002	5.923e-002
0.6	2.156e+008	3.553e+001	1.661e+002	6.935e-002	3.242e-001
0.8	7.904e+007	2.622e+001	1.081e+002	4.988e-002	2.056e-001
1.0	2.647e+007	1.488e+001	5.508e+001	2.743e-002	1.015e-001
1.5	1.245e+007	1.762e+001	5.342e+001	2.965e-002	8.987e-002
2.0	5.461e+005	1.424e+000	3.823e+000	2.202e-003	5.912e-003
3.0	2.548e+008	1.469e+003	3.348e+003	1.993e+000	4.542e+000
TOTAL:	2.297e+009	1.580e+003	3.808e+003	2.200e+000	5.409e+000

Page : 3
DOS File: BDC0.MS4
Run Date: January 28, 1997
Run Time: 5:43 p.m. Tuesday
Title : Bottom Drn Cont w Silo 1 glass 0% eq

SENSITIVITY RESULTS For: X (cm)

Case Number	Sensitivity Variable Value	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildup
1	86.18125	1.580e+003	3.808e+003	2.200e+000	5.409e+000
2	185.1813	3.249e+002	7.029e+002	4.565e-001	1.011e+000

Use the Display Menu For Energy Group Results For All Cases.

MicroShield 4.21 - Serial #4.21-00915
Fermco

Page : 1
DOS File: BDC10.MS4
Run Date: January 28, 1997
Run Time: 5:55 p.m. Tuesday
Duration: 0:11:16

File Ref: _____
Date: __/__/__
By: _____
Checked: _____

Case Title: Bottom Drn Cont w Silo 1 glass 10% eq

GEOMETRY 13 - Rectangular Volume

	centimeters	feet and inches	
Dose point coordinate X:	86.18125	2.0	9.9
Dose point coordinate Y:	39.495	1.0	3.5
Dose point coordinate Z:	72.515	2.0	4.5
Rectangular volume width :	145.03	4.0	9.1
Rectangular volume length:	78.99	2.0	7.1
Rectangular volume height:	72.01	2.0	4.4
Shield 1:	5.715	0.0	2.3
Shield 2:	0.47625	0.0	.2
Air Gap:	1.0	0.0	.4

Source Volume: 824941. cm^3 29.1325 cu ft. 50341. cu in.

MATERIAL DENSITIES (g/cm^3)

Material	Source Shield	Shield 1 Slab	Shield 2 Slab	Air Gap
Air				0.00122
Iron			7.86	
HI-ALUMINA		2.499		
Monolith	2.11			

BUILDUP
Method: Buildup Factor Tables
The material reference is Source

INTEGRATION PARAMETERS

	Quadrature Order
X Direction	20
Y Direction	20
Z Direction	20

SOURCE NUCLIDES

Nuclide	curies	ìCi/cm^3	Nuclide	curies	ìCi/cm^3
Ac-227	7.1998e-003	8.7277e-003	Ac-228	1.0000e-003	1.2122e-003
Bi-210	1.9000e-001	2.3032e-001	Bi-211	7.2033e-003	8.7319e-003
Bi-212	6.8956e-003	8.3589e-003	Bi-214	4.2699e-002	5.1761e-002
Fr-223	9.9358e-005	1.2044e-004	Pa-231	3.8000e-003	4.6064e-003
Pa-234	1.7465e-006	2.1171e-006	Pa-234m	1.1000e-003	1.3334e-003
Pb-210	1.8999e-001	2.3031e-001	Pb-211	7.2033e-003	8.7319e-003
Pb-212	6.8960e-003	8.3594e-003	Pb-214	4.4171e-002	5.3545e-002
Po-210	2.5979e-001	3.1492e-001	Po-211	1.9665e-005	2.3838e-005

Page : 2
DOS File: BDC10.MS4
Run Date: January 28, 1997
Run Time: 5:55 p.m. Tuesday
Title : Bottom Drn Cont w Silo 1 glass 10% eq

Nuclide	curies	ÌCi/cm^3	Nuclide	curies	ÌCi/cm^3
Po-212	4.4180e-003	5.3556e-003	Po-214	4.2690e-002	5.1750e-002
Po-215	7.2035e-003	8.7322e-003	Po-216	6.8998e-003	8.3640e-003
Po-218	4.6155e-002	5.5950e-002	Ra-223	7.2035e-003	8.7322e-003
Ra-224	6.8998e-003	8.3640e-003	Ra-226	4.5000e-001	5.4549e-001
Ra-228	1.0000e-003	1.2122e-003	Rn-219	7.2035e-003	8.7322e-003
Rn-220	6.8998e-003	8.3640e-003	Rn-222	4.6379e-002	5.6221e-002
Th-227	7.1978e-003	8.7252e-003	Th-228	6.8965e-003	8.3600e-003
Th-230	6.5000e-002	7.8794e-002	Th-231	8.8000e-005	1.0667e-004
Th-232	1.0000e-003	1.2122e-003	Th-234	1.1000e-003	1.3334e-003
Tl-207	7.1836e-003	8.7080e-003	Tl-208	2.4776e-003	3.0034e-003
U-234	8.7000e-004	1.0546e-003	U-235	8.8000e-005	1.0667e-004
U-238	1.1000e-003	1.3334e-003			

========= RESULTS FOR SENSITIVITY REFERENCE CASE (X = 86.1813) =========

Energy (MeV)	Activity (photons/sec)	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildup
0.015	1.026e+005	4.684e-141	3.941e-028	4.018e-142	3.380e-029
0.02	1.053e+006	1.274e-065	5.865e-027	4.412e-067	2.032e-028
0.03	1.382e+007	1.405e-022	1.602e-022	1.392e-024	1.587e-024
0.04	3.431e+006	1.115e-012	1.413e-012	4.933e-015	6.250e-015
0.05	3.272e+008	1.893e-006	2.721e-006	5.044e-009	7.248e-009
0.06	1.149e+007	7.135e-006	1.162e-005	1.417e-008	2.308e-008
0.08	6.894e+008	3.206e-002	6.660e-002	5.074e-005	1.054e-004
0.1	7.517e+007	1.126e-002	2.926e-002	1.723e-005	4.477e-005
0.15	3.233e+007	5.618e-002	2.128e-001	9.251e-005	3.504e-004
0.2	8.398e+008	5.110e+000	2.416e+001	9.018e-003	4.264e-002
0.3	5.129e+008	1.272e+001	6.802e+001	2.412e-002	1.290e-001
0.4	6.947e+008	3.985e+001	2.106e+002	7.764e-002	4.104e-001
0.5	5.330e+007	5.538e+000	2.767e+001	1.087e-002	5.432e-002
0.6	8.397e+008	1.384e+002	6.468e+002	2.701e-001	1.263e+000
0.8	2.159e+008	7.164e+001	2.954e+002	1.363e-001	5.618e-001
1.0	5.200e+008	2.923e+002	1.082e+003	5.388e-001	1.994e+000
1.5	3.132e+008	4.432e+002	1.344e+003	7.456e-001	2.260e+000
2.0	4.233e+008	1.104e+003	2.964e+003	1.707e+000	4.583e+000
3.0	9.149e+007	5.275e+002	1.202e+003	7.157e-001	1.631e+000
TOTAL:	5.658e+009	2.640e+003	7.864e+003	4.235e+000	1.293e+001

Page : 3
DOS File: BDC10.MS4
Run Date: January 28, 1997
Run Time: 5:55 p.m. Tuesday
Title : Bottom Drn Cont w Silo 1 glass 10% eq

SENSITIVITY RESULTS For: X (cm)

Case Number	Sensitivity Variable Value	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildup
1	86.18125	2.640e+003	7.864e+003	4.235e+000	1.293e+001
2	185.1813	6.012e+002	1.581e+003	9.762e-001	2.629e+000

Use the Display Menu For Energy Group Results For All Cases.

MicroShield 4.21 - Serial #4.21-00915
Fermco

Page : 1
DOS File: BDCTOP.MS4
Run Date: January 29, 1997
Run Time: 7:55 a.m. Wednesday
Duration: 0:17:49

File Ref: _____
Date: ___/___/___
By: _____
Checked: _____

Case Title: Bottom Drn Cont w Silo 1 glass 100% eq (TOP)

GEOMETRY 13 - Rectangular Volume

	centimeters	feet and inches	
Dose point coordinate X:	79.99	2.0	7.5
Dose point coordinate Y:	72.515	2.0	4.5
Dose point coordinate Z:	36.005	1.0	2.2
Rectangular volume width :	72.01	2.0	4.4
Rectangular volume length:	78.99	2.0	7.1
Rectangular volume height:	145.03	4.0	9.1
Air Gap:	1.0	0.0	.4

Source Volume: 824941. cm^3 29.1325 cu ft. 50341. cu in.

MATERIAL DENSITIES (g/cm^3)

Material	Source Shield	Air Gap
Air		0.00122
Monolith	2.11	

BUILDUP
Method: Buildup Factor Tables
The material reference is Source

INTEGRATION PARAMETERS

	Quadrature Order
X Direction	20
Y Direction	20
Z Direction	20

SOURCE NUCLIDES

Nuclide	curies	ÌCi/cm^3	Nuclide	curies	ÌCi/cm^3
Ac-227	7.2000e-003	8.7279e-003	Ac-228	1.0000e-003	1.2122e-003
Bi-210	1.9000e-001	2.3032e-001	Bi-211	7.2000e-003	8.7279e-003
Bi-212	6.9000e-003	8.3642e-003	Bi-214	4.5000e-001	5.4549e-001
Fr-223	9.9000e-005	1.2001e-004	Pa-231	3.8000e-003	4.6064e-003
Pa-234	1.7000e-006	2.0608e-006	Pa-234m	1.1000e-003	1.3334e-003
Pb-210	1.9000e-001	2.3032e-001	Pb-211	7.2000e-003	8.7279e-003
Pb-212	6.9000e-003	8.3642e-003	Pb-214	4.5000e-001	5.4549e-001
Po-210	2.6000e-001	3.1517e-001	Po-211	2.0000e-005	2.4244e-005
Po-212	4.4000e-003	5.3337e-003	Po-214	4.5000e-001	5.4549e-001
Po-215	7.2000e-003	8.7279e-003	Po-216	6.9000e-003	8.3642e-003
Po-218	4.5000e-001	5.4549e-001	Ra-223	7.2000e-003	8.7279e-003
Ra-224	6.9000e-003	8.3642e-003	Ra-226	4.5000e-001	5.4549e-001

DOS File: BDCTOP.MS4
Run Date: January 29, 1997
Run Time: 7:55 a.m. Wednesday
Title : Bottom Drn Cont w Silo 1 glass 100% eq (TOP)

Nuclide	curies	ÌCi/cm^3	Nuclide	curies	ÌCi/cm^3
Ra-228	1.0000e-003	1.2122e-003	Rn-219	7.2000e-003	8.7279e-003
Rn-220	6.9000e-003	8.3642e-003	Rn-222	4.5000e-001	5.4549e-001
Th-227	7.2000e-003	8.7279e-003	Th-228	6.9000e-003	8.3642e-003
Th-230	6.5000e-002	7.8794e-002	Th-231	8.8000e-005	1.0667e-004
Th-232	1.0000e-003	1.2122e-003	Th-234	1.1000e-003	1.3334e-003
Tl-207	7.2000e-003	8.7279e-003	Tl-208	6.9000e-003	8.3642e-003
U-234	8.7000e-004	1.0546e-003	U-235	8.8000e-005	1.0667e-004
U-238	1.1000e-003	1.3334e-003			

========== RESULTS FOR SENSITIVITY REFERENCE CASE (X = 79.99) ==========

Energy (MeV)	Activity (photons/sec)	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildup
0.015	1.026e+005	4.855e-012	4.933e-012	4.164e-013	4.231e-013
0.02	1.053e+006	4.605e-008	4.761e-008	1.595e-009	1.649e-009
0.03	1.382e+007	2.223e-003	2.382e-003	2.203e-005	2.361e-005
0.04	3.433e+006	2.316e-003	2.625e-003	1.024e-005	1.161e-005
0.05	4.932e+008	1.681e+000	2.012e+000	4.478e-003	5.360e-003
0.06	1.149e+007	1.011e-001	1.268e-001	2.009e-004	2.519e-004
0.08	4.091e+009	1.158e+002	1.602e+002	1.833e-001	2.534e-001
0.1	7.515e+007	1.730e+000	2.757e+000	2.647e-003	4.219e-003
0.15	3.232e+007	3.063e+000	5.582e+000	5.045e-003	9.192e-003
0.2	1.966e+009	4.277e+002	8.552e+002	7.549e-001	1.509e+000
0.3	3.623e+009	2.050e+003	4.395e+003	3.889e+000	8.336e+000
0.4	6.441e+009	6.374e+003	1.379e+004	1.242e+001	2.686e+001
0.5	3.571e+008	5.218e+002	1.112e+003	1.024e+000	2.184e+000
0.6	8.243e+009	1.627e+004	3.399e+004	3.176e+001	6.634e+001
0.8	1.663e+009	5.183e+003	1.039e+004	9.859e+000	1.976e+001
1.0	5.240e+009	2.311e+004	4.463e+004	4.259e+001	8.227e+00
1.5	3.182e+009	2.608e+004	4.679e+004	4.388e+001	7.872e+00
2.0	4.456e+009	5.584e+004	9.579e+004	8.636e+001	1.481e+00
3.0	2.548e+008	5.674e+003	9.089e+003	7.698e+000	1.233e+00
TOTAL:	4.015e+010	1.417e+005	2.610e+005	2.404e+002	4.467e+00

Page : 3
DOS File: BDCTOP.MS4
Run Date: January 29, 1997
Run Time: 7:55 a.m. Wednesday
Title : Bottom Drn Cont w Silo 1 glass 100% eq (TOP)

SENSITIVITY RESULTS For: X (cm)

Case Number	Sensitivity Variable Value	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildup
1	79.99	1.417e+005	2.610e+005	2.404e+002	4.467e+002
2	178.99	1.658e+004	2.957e+004	2.819e+001	5.070e+001

Use the Display Menu For Energy Group Results For All Cases.

MicroShield 4.21 - Serial #4.21-00915
Fermco

Page : 1
DOS File: BDCTOP0.MS4
Run Date: January 28, 1997
Run Time: 6:17 p.m. Tuesday
Duration: 0:11:09

File Ref: _____
Date: ___/___/___
By: _____
Checked: _____

Case Title: Bottom Drn Cont w Silo 1 glass 0% eq (TOP)

GEOMETRY 13 - Rectangular Volume

	centimeters	feet and inches	
Dose point coordinate X:	79.99	2.0	7.5
Dose point coordinate Y:	72.515	2.0	4.5
Dose point coordinate Z:	36.005	1.0	2.2
Rectangular volume width :	72.01	2.0	4.4
Rectangular volume length:	78.99	2.0	7.1
Rectangular volume height:	145.03	4.0	9.1
Air Gap:	1.0	0.0	.4

Source Volume: 824941. cm^3 29.1325 cu ft. 50341. cu in.

MATERIAL DENSITIES (g/cm^3)

Material	Source Shield	Air Gap
Air		0.00122
Monolith	2.11	

BUILDUP
Method: Buildup Factor Tables
The material reference is Source

INTEGRATION PARAMETERS

	Quadrature Order
X Direction	20
Y Direction	20
Z Direction	20

SOURCE NUCLIDES

Nuclide	curies	ìCi/cm^3	Nuclide	curies	ìCi/cm^3
Ac-227	7.2000e-003	8.7279e-003	Ac-228	1.0000e-003	1.2122e-003
Bi-210	1.9000e-001	2.3032e-001	Bi-211	0.0000e+000	0.0000e+000
Bi-212	6.9000e-003	8.3642e-003	Bi-214	0.0000e+000	0.0000e+000
Fr-223	9.9000e-005	1.2001e-004	Pa-231	3.8000e-003	4.6064e-003
Pa-234	1.7000e-006	2.0608e-006	Pa-234m	1.1000e-003	1.3334e-003
Pb-210	1.9000e-001	2.3032e-001	Pb-211	0.0000e+000	0.0000e+000
Pb-212	6.9000e-003	8.3642e-003	Pb-214	0.0000e+000	0.0000e+000
Po-210	2.6000e-001	3.1517e-001	Po-211	0.0000e+000	0.0000e+000
Po-212	4.4000e-003	5.3337e-003	Po-214	0.0000e+000	0.0000e+000
Po-215	0.0000e+000	0.0000e+000	Po-216	0.0000e+000	0.0000e+000
Po-218	0.0000e+000	0.0000e+000	Ra-223	7.2000e-003	8.7279e-003
Ra-224	6.9000e-003	8.3642e-003	Ra-226	4.5000e-001	5.4549e-001

DOS File: BDCTOP0.MS4
Run Date: January 28, 1997
Run Time: 6:17 p.m. Tuesday
Title : Bottom Drn Cont w Silo 1 glass 0% eq (TOP)

Nuclide	curies	ÌCi/cm^3	Nuclide	curies	ÌCi/cm^3
Ra-228	1.0000e-003	1.2122e-003	Rn-219	0.0000e+000	0.0000e+000
Rn-220	0.0000e+000	0.0000e+000	Rn-222	0.0000e+000	0.0000e+000
Th-227	7.2000e-003	8.7279e-003	Th-228	6.9000e-003	8.3642e-003
Th-230	6.5000e-002	7.8794e-002	Th-231	8.8000e-005	1.0667e-004
Th-232	1.0000e-003	1.2122e-003	Th-234	1.1000e-003	1.3334e-003
Tl-207	0.0000e+000	0.0000e+000	Tl-208	6.9000e-003	8.3642e-003
U-234	8.7000e-004	1.0546e-003	U-235	8.8000e-005	1.0667e-004
U-238	1.1000e-003	1.3334e-003			

========== RESULTS FOR SENSITIVITY REFERENCE CASE (X = 79.99) ==========

Energy (MeV)	Activity (photons/sec)	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildup
0.015	1.026e+005	4.855e-012	4.933e-012	4.164e-013	4.231e-013
0.02	1.053e+006	4.605e-008	4.761e-008	1.595e-009	1.649e-009
0.03	1.382e+007	2.223e-003	2.382e-003	2.203e-005	2.361e-005
0.04	3.433e+006	2.316e-003	2.625e-003	1.024e-005	1.161e-005
0.05	3.091e+008	1.054e+000	1.261e+000	2.807e-003	3.360e-003
0.06	1.149e+007	1.011e-001	1.268e-001	2.009e-004	2.519e-004
0.08	3.197e+008	9.051e+000	1.252e+001	1.432e-002	1.981e-002
0.1	7.515e+007	1.730e+000	2.757e+000	2.647e-003	4.219e-003
0.15	3.201e+007	3.034e+000	5.529e+000	4.996e-003	9.104e-003
0.2	7.182e+008	1.563e+002	3.125e+002	2.759e-001	5.515e-001
0.3	1.600e+008	9.054e+001	1.941e+002	1.717e-001	3.681e-001
0.4	6.048e+006	5.985e+000	1.294e+001	1.166e-002	2.522e-002
0.5	5.811e+007	8.492e+001	1.811e+002	1.667e-001	3.554e-001
0.6	2.156e+008	4.256e+002	8.891e+002	8.308e-001	1.735e+000
0.8	7.904e+007	2.464e+002	4.937e+002	4.686e-001	9.391e-001
1.0	2.647e+007	1.167e+002	2.255e+002	2.152e-001	4.157e-001
1.5	1.245e+007	1.021e+002	1.831e+002	1.717e-001	3.081e-001
2.0	5.461e+005	6.844e+000	1.174e+001	1.058e-002	1.815e-002
3.0	2.548e+008	5.674e+003	9.089e+003	7.698e+000	1.233e+001
TOTAL:	2.297e+009	6.925e+003	1.161e+004	1.005e+001	1.708e+001

Page : 3
DOS File: BDCTOP0.MS4
Run Date: January 28, 1997
Run Time: 6:17 p.m. Tuesday
Title : Bottom Drn Cont w Silo 1 glass 0% eq (TOP)

SENSITIVITY RESULTS For: X (cm)

Case Number	Sensitivity Variable Value	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildup
1	79.99	6.925e+003	1.161e+004	1.005e+001	1.708e+001
2	178.99	7.965e+002	1.300e+003	1.159e+000	1.918e+000

Use the Display Menu For Energy Group Results For All Cases.

MicroShield 4.21 - Serial #4.21-00915
Fermco

Page : 1
DOS File: BDCTOP10.MS4
Run Date: January 28, 1997
Run Time: 6:28 p.m. Tuesday
Duration: 0:11:08

File Ref: _____
Date: __/__/__
By: _____
Checked: _____

Case Title: Bottom Drn Cont w Silo 1 glass 10% eq (TOP)

GEOMETRY 13 - Rectangular Volume

	centimeters	feet and inches	
Dose point coordinate X:	79.99	2.0	7.5
Dose point coordinate Y:	72.515	2.0	4.5
Dose point coordinate Z:	36.005	1.0	2.2
Rectangular volume width :	72.01	2.0	4.4
Rectangular volume length:	78.99	2.0	7.1
Rectangular volume height:	145.03	4.0	9.1
Air Gap:	1.0	0.0	.4

Source Volume: 824941. cm^3 29.1325 cu ft. 50341. cu in.

MATERIAL DENSITIES (g/cm^3)

Material	Source Shield	Air Gap
Air		0.00122
Monolith	2.11	

BUILDUP
Method: Buildup Factor Tables
The material reference is Source

INTEGRATION PARAMETERS

	Quadrature Order
X Direction	20
Y Direction	20
Z Direction	20

SOURCE NUCLIDES

Nuclide	curies	µCi/cm^3	Nuclide	curies	µCi/cm^3
Ac-227	7.1998e-003	8.7277e-003	Ac-228	1.0000e-003	1.2122e-003
Bi-210	1.9000e-001	2.3032e-001	Bi-211	7.2033e-003	8.7319e-003
Bi-212	6.8956e-003	8.3589e-003	Bi-214	4.2699e-002	5.1761e-002
Fr-223	9.9358e-005	1.2044e-004	Pa-231	3.8000e-003	4.6064e-003
Pa-234	1.7465e-006	2.1171e-006	Pa-234m	1.1000e-003	1.3334e-003
Pb-210	1.8999e-001	2.3031e-001	Pb-211	7.2033e-003	8.7319e-003
Pb-212	6.8960e-003	8.3594e-003	Pb-214	4.4171e-002	5.3545e-002
Po-210	2.5979e-001	3.1492e-001	Po-211	1.9665e-005	2.3838e-005
Po-212	4.4180e-003	5.3556e-003	Po-214	4.2690e-002	5.1750e-002
Po-215	7.2035e-003	8.7322e-003	Po-216	6.8998e-003	8.3640e-003
Po-218	4.6155e-002	5.5950e-002	Ra-223	7.2035e-003	8.7322e-003
Ra-224	6.8998e-003	8.3640e-003	Ra-226	4.5000e-001	5.4549e-001

Page : 2
DOS File: BDCTOP10.MS4
Run Date: January 28, 1997
Run Time: 6:28 p.m. Tuesday
Title : Bottom Drn Cont w Silo 1 glass 10% eq (TOP)

Nuclide	curies	ÌCi/cm^3	Nuclide	curies	ÌCi/cm^3
Ra-228	1.0000e-003	1.2122e-003	Rn-219	7.2035e-003	8.7322e-003
Rn-220	6.8998e-003	8.3640e-003	Rn-222	4.6379e-002	5.6221e-002
Th-227	7.1978e-003	8.7252e-003	Th-228	6.8965e-003	8.3600e-003
Th-230	6.5000e-002	7.8794e-002	Th-231	8.8000e-005	1.0667e-004
Th-232	1.0000e-003	1.2122e-003	Th-234	1.1000e-003	1.3334e-003
Tl-207	7.1836e-003	8.7080e-003	Tl-208	2.4776e-003	3.0034e-003
U-234	8.7000e-004	1.0546e-003	U-235	8.8000e-005	1.0667e-004
U-238	1.1000e-003	1.3334e-003			

========== RESULTS FOR SENSITIVITY REFERENCE CASE (X = 79.99) ==========

Energy (MeV)	Activity (photons/sec)	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildup
0.015	1.026e+005	4.855e-012	4.933e-012	4.164e-013	4.231e-013
0.02	1.053e+006	4.604e-008	4.761e-008	1.595e-009	1.649e-009
0.03	1.382e+007	2.222e-003	2.382e-003	2.203e-005	2.361e-005
0.04	3.431e+006	2.315e-003	2.623e-003	1.024e-005	1.160e-005
0.05	3.272e+008	1.115e+000	1.335e+000	2.971e-003	3.556e-003
0.06	1.149e+007	1.011e-001	1.268e-001	2.009e-004	2.519e-004
0.08	6.894e+008	1.952e+001	2.699e+001	3.088e-002	4.270e-002
0.1	7.517e+007	1.731e+000	2.758e+000	2.648e-003	4.219e-003
0.15	3.233e+007	3.065e+000	5.584e+000	5.047e-003	9.196e-003
0.2	8.398e+008	1.828e+002	3.654e+002	3.225e-001	6.449e-001
0.3	5.129e+008	2.902e+002	6.221e+002	5.506e-001	1.180e+000
0.4	6.947e+008	6.874e+002	1.487e+003	1.339e+000	2.897e+000
0.5	5.330e+007	7.788e+001	1.661e+002	1.529e-001	3.259e-001
0.6	8.397e+008	1.658e+003	3.462e+003	3.235e+000	6.758e+000
0.8	2.159e+008	6.731e+002	1.349e+003	1.280e+000	2.566e+000
1.0	5.200e+008	2.293e+003	4.430e+003	4.227e+000	8.165e+000
1.5	3.132e+008	2.567e+003	4.605e+003	4.319e+000	7.748e+000
2.0	4.233e+008	5.305e+003	9.100e+003	8.204e+000	1.407e+001
3.0	9.149e+007	2.037e+003	3.263e+003	2.764e+000	4.428e+000
TOTAL:	5.658e+009	1.580e+004	2.889e+004	2.644e+001	4.884e+001

Page : 3
DOS File: BDCTOP10.MS4
Run Date: January 28, 1997
Run Time: 6:28 p.m. Tuesday
Title : Bottom Drn Cont w Silo 1 glass 10% eq (TOP)

SENSITIVITY RESULTS For: X (cm)

Case Number	Sensitivity Variable Value	Energy Fluence Rate (MeV/sq cm/sec)		Exposure Rate In Air (mR/hr)	
		No Buildup	With Buildup	No Buildup	With Buildup
1	79.99	1.580e+004	2.889e+004	2.644e+001	4.884e+001
2	178.99	1.848e+003	3.273e+003	3.099e+000	5.547e+000

Use the Display Menu For Energy Group Results For All Cases.

Attachment 5: Organization

FERNALD

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INTEROFFICE MEMORANDUM

To:	Distribution	**Date:**	October 11, 1996
Location:	Various	**Reference:**	N/A
From:	Yvonne Gale, YG Silos Project Administration	**FD Fernald#:**	M:WMTSP(SP):96-0058
Location:	Fernald, MS52-4	**Client:**	DOE DE-AC24-92OR21972
Extension:	648-4858	**Subject:**	**Silos Project Organization Charts**

c: File Record Storage Copy 106.4.35.9
Nina Akgunduz, DOE-FN, MS45
Bob Heck, MS52-5
Jerry Lester, MS52-3
Lynn Macenko, MS52-3
Dennis Nix, MS52-3

Attached are the Silos Project organization charts listing the functional responsibilities as well as team members.

If you have any questions, please contact me at 648-4858.

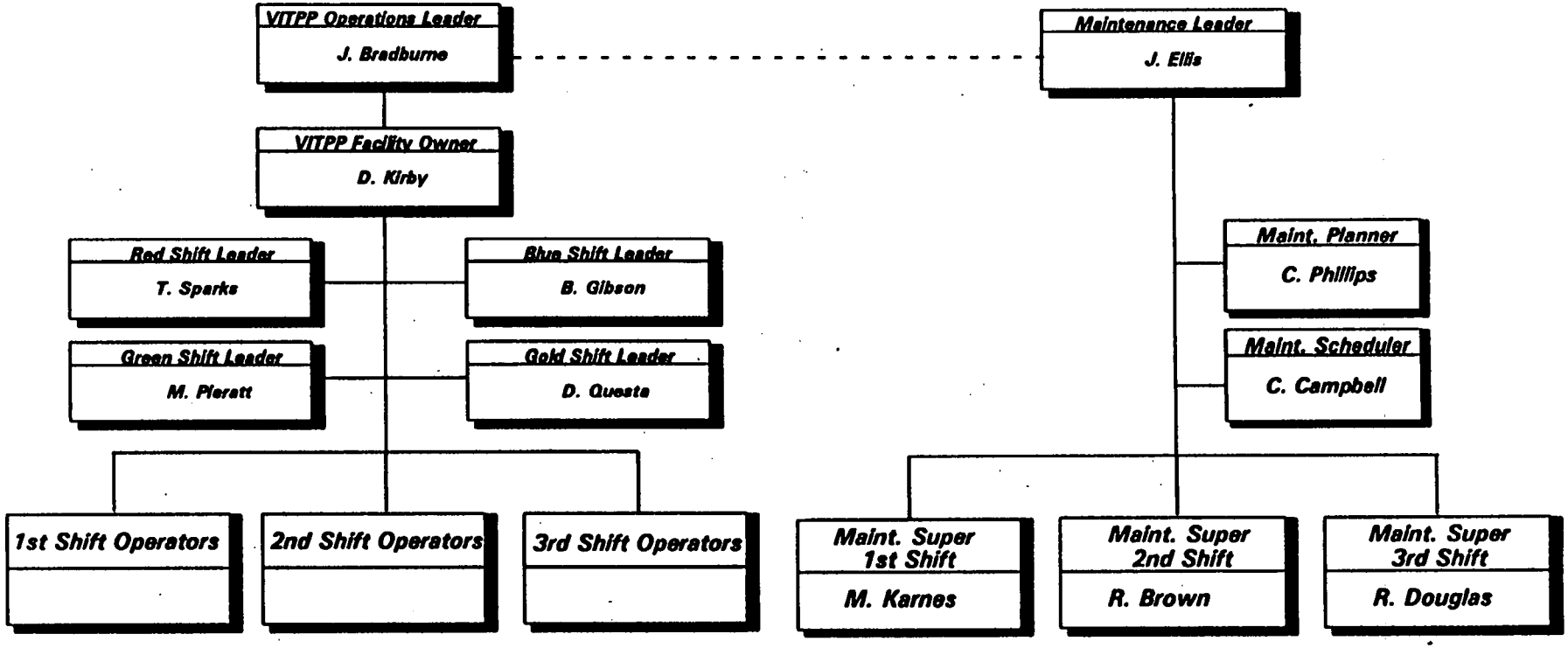
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Attachment

FS-F-3934 (8/31/95)

000534

VITPP OPERATIONS & MAINTENANCE



S. Chaney
T. Collins
R. Cranert
D. Ebel
R. Henze
L. Larkin
M. Lee
R. Muncie
T. Roberts
D. Scott
J. Sexton

V. Butler
S. Flack
W. Hawkins
D. Nunemaker
C. Smith
G. Stafford
R. Stewart
R. Sutton

Z. Black
N. Dudley
J. Grace
D. Johnson
J. Miller
W. Pettit
P. Webb
M. Wilson

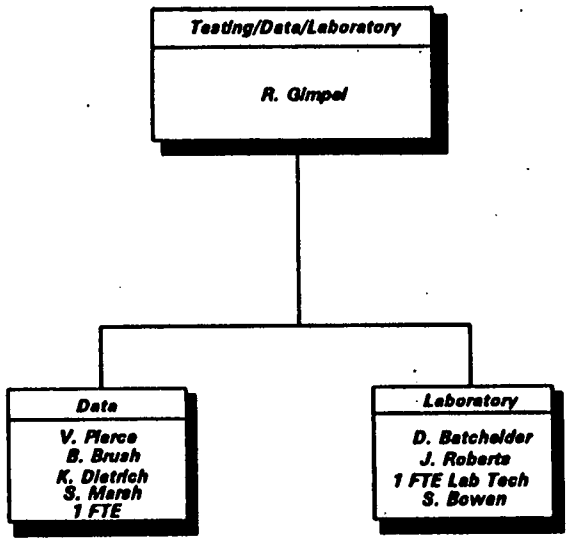
C. Bradbury
L. Cash
C. Clark
R. Fisher
R. Kelly
S. Lukacs
C. McGoodin
J. Metzler
C. Shouse, Sr.
C. Shouse, Jr.
J. Siciliano
A. Windler

M. Baker
G. Eagle
J. Plavsic
D. Seiller
C. Taylor
C. Varns

M. Caudill
S. Craig
F. Goble
J. Hunter
G. Reid
A. Roberts

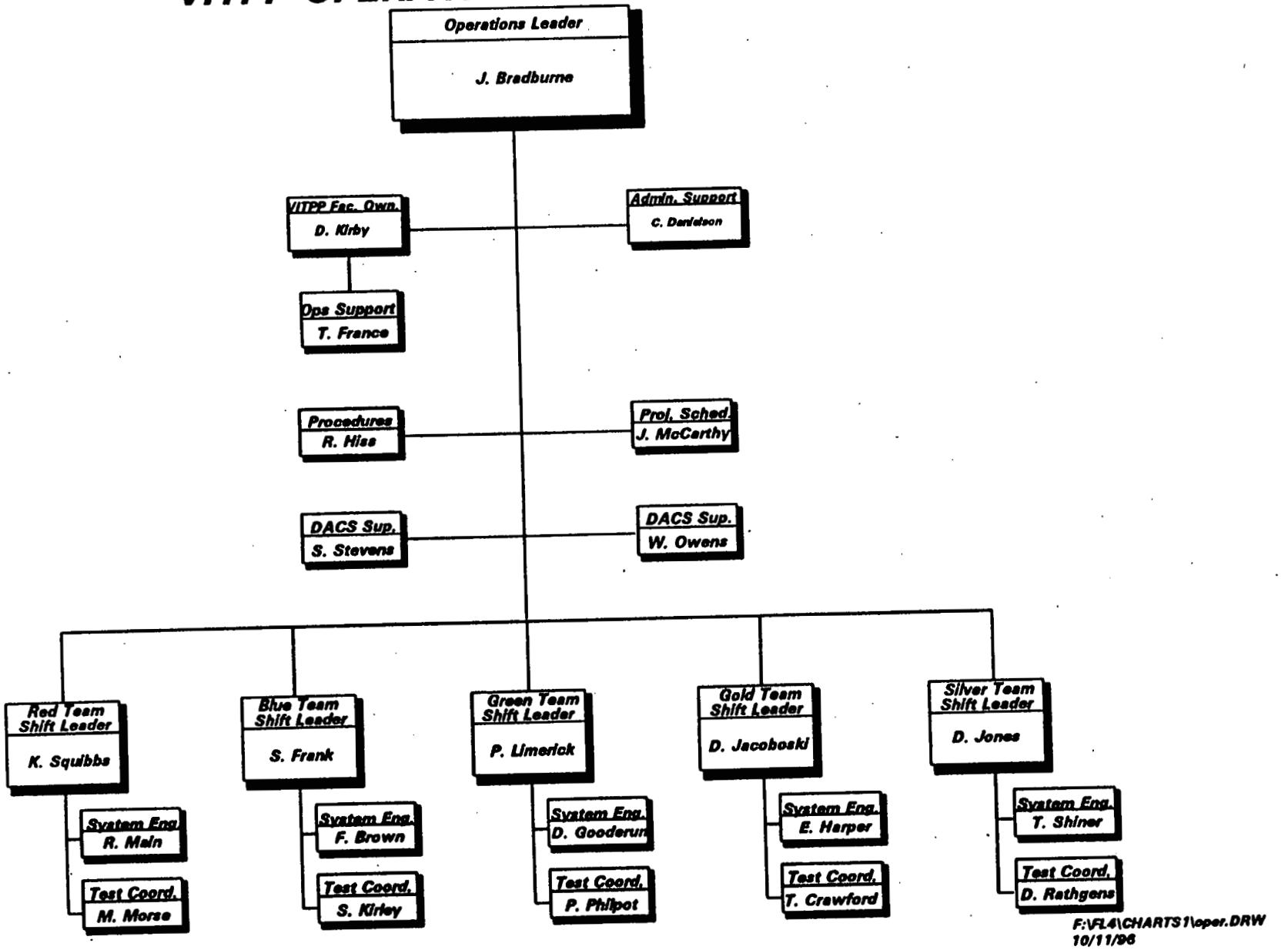
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10/14/96

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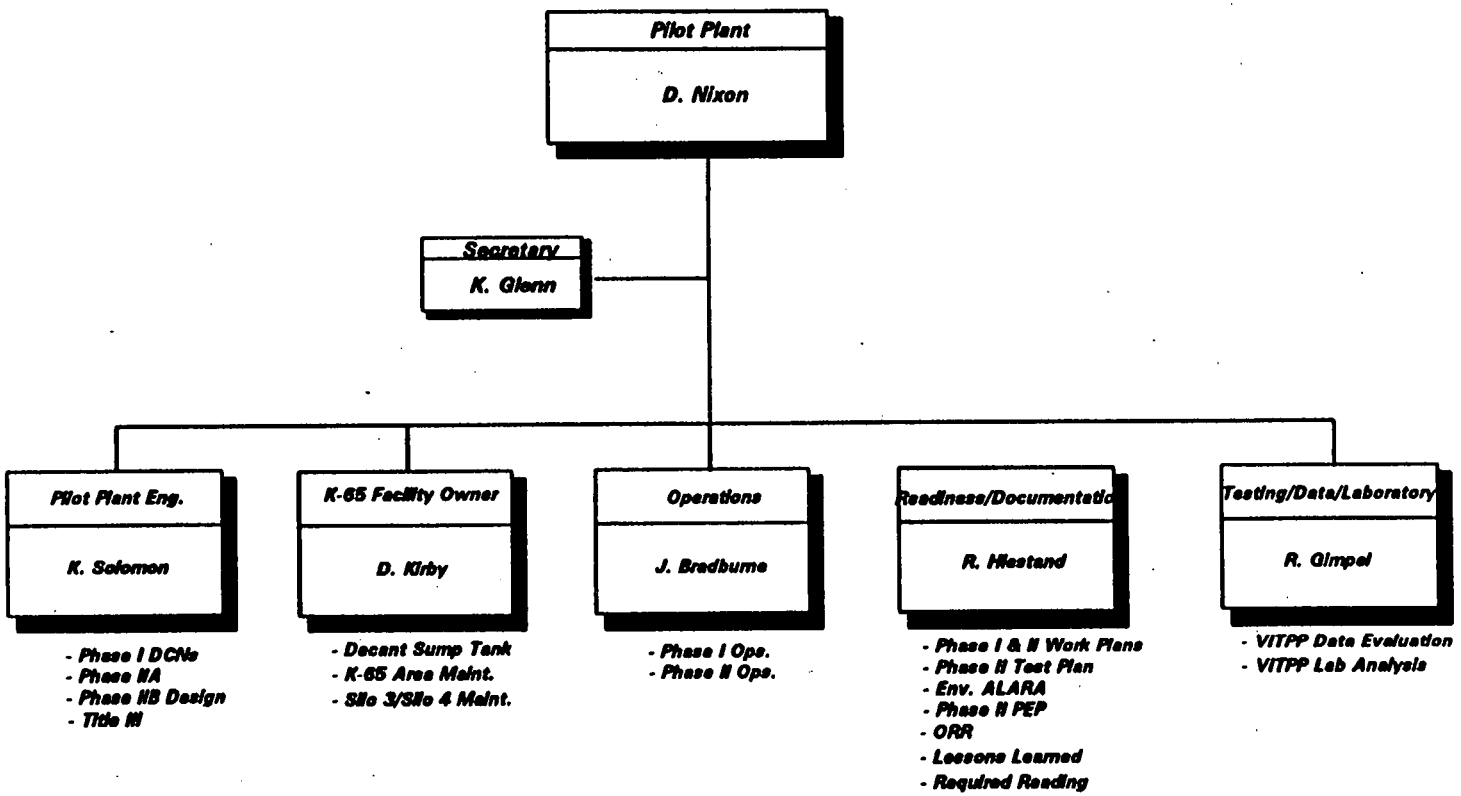
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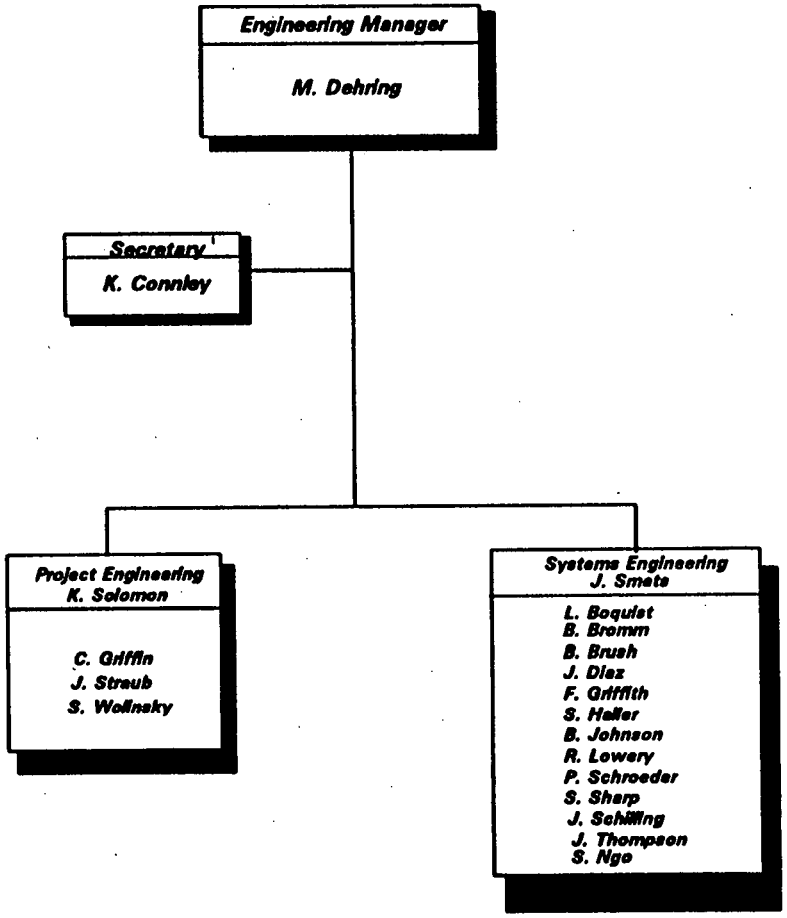
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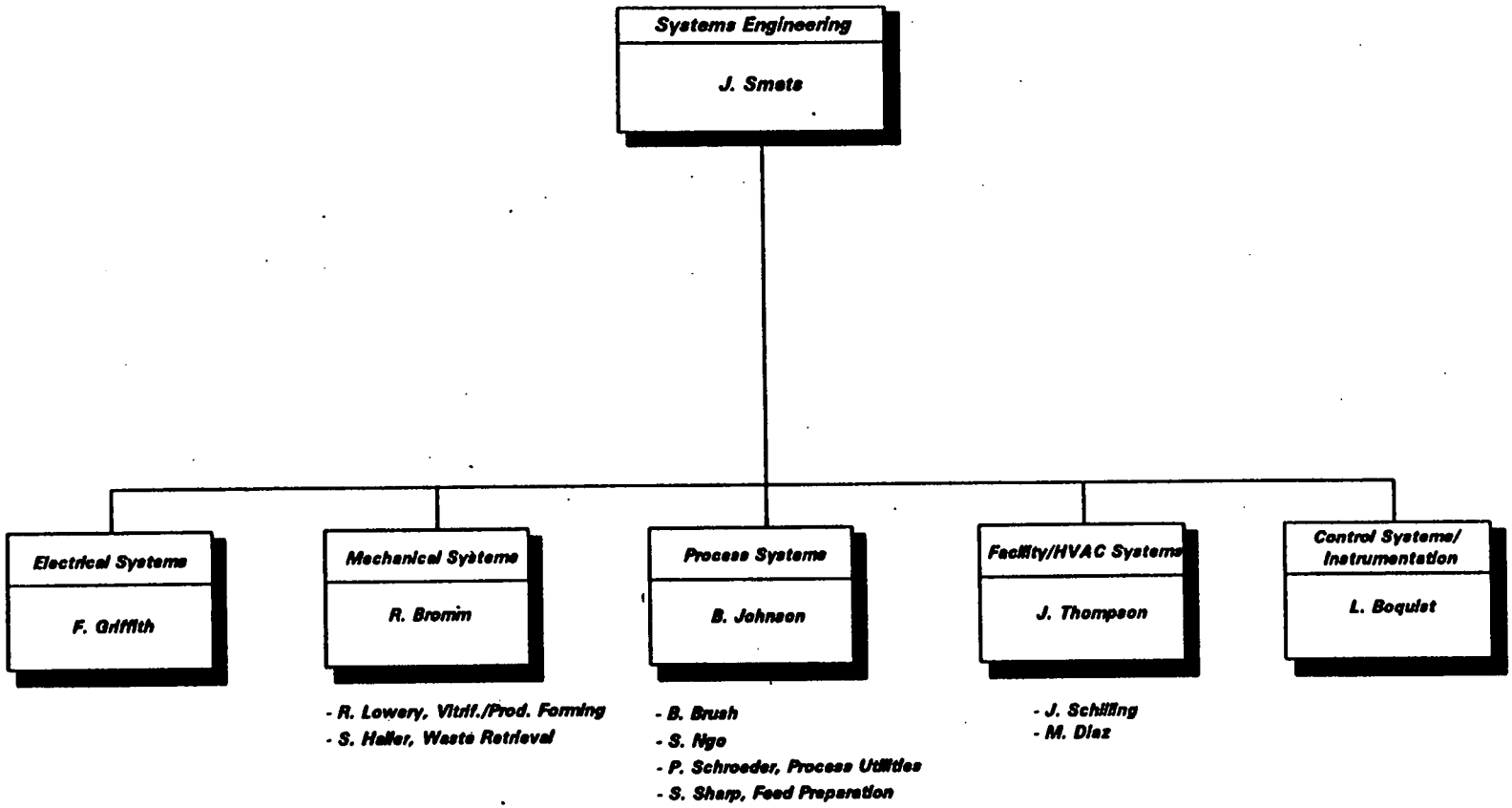
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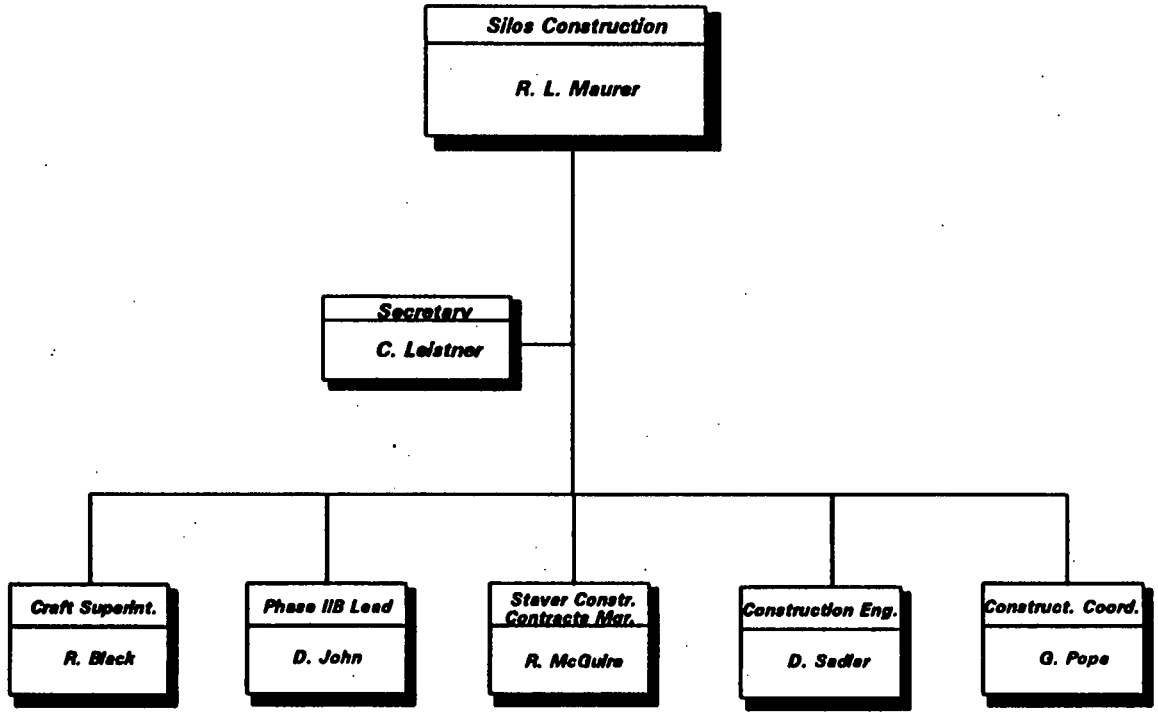
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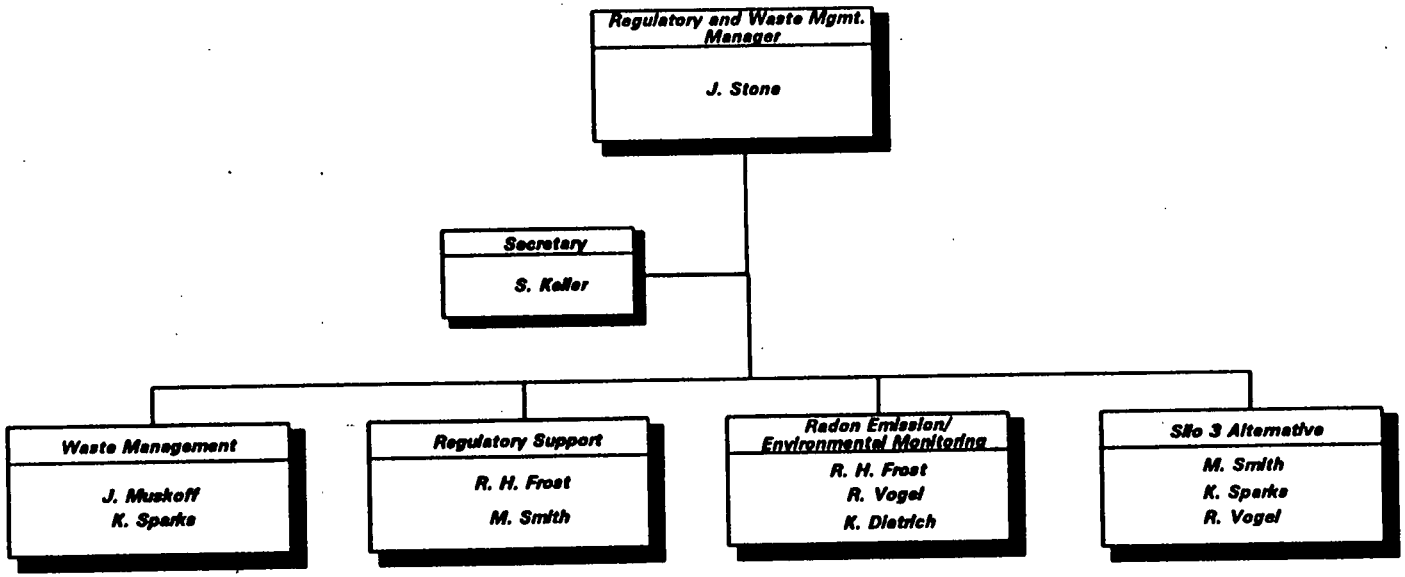
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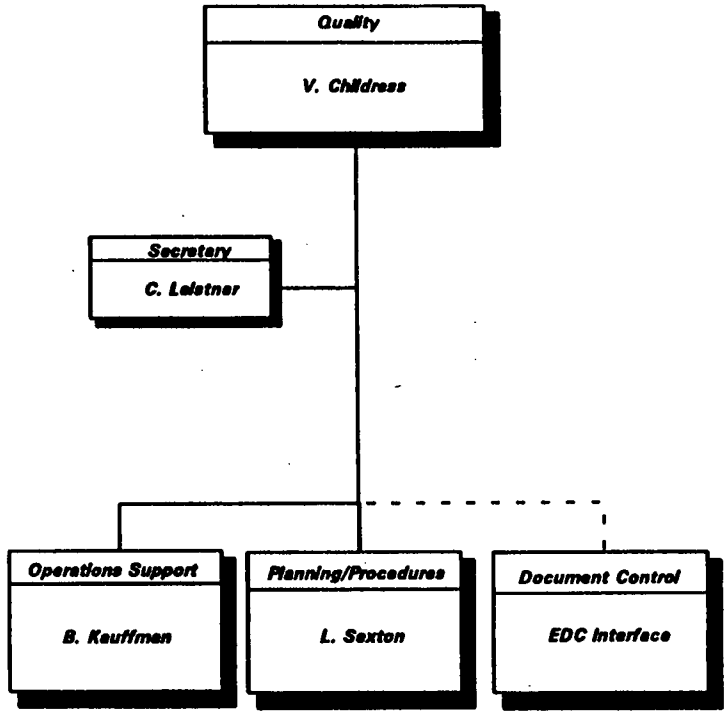
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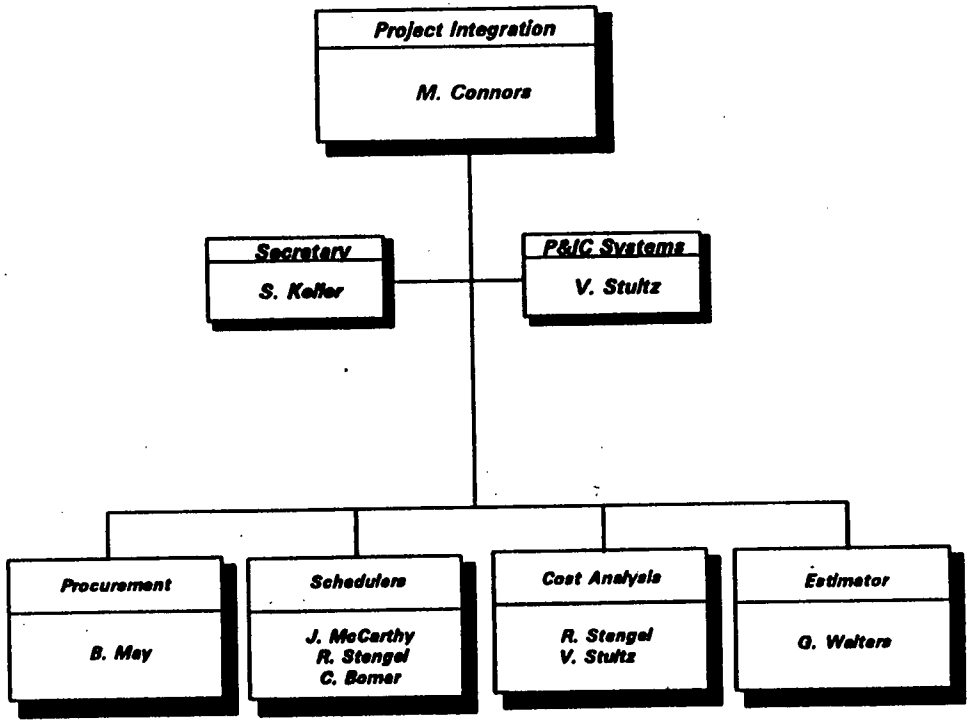
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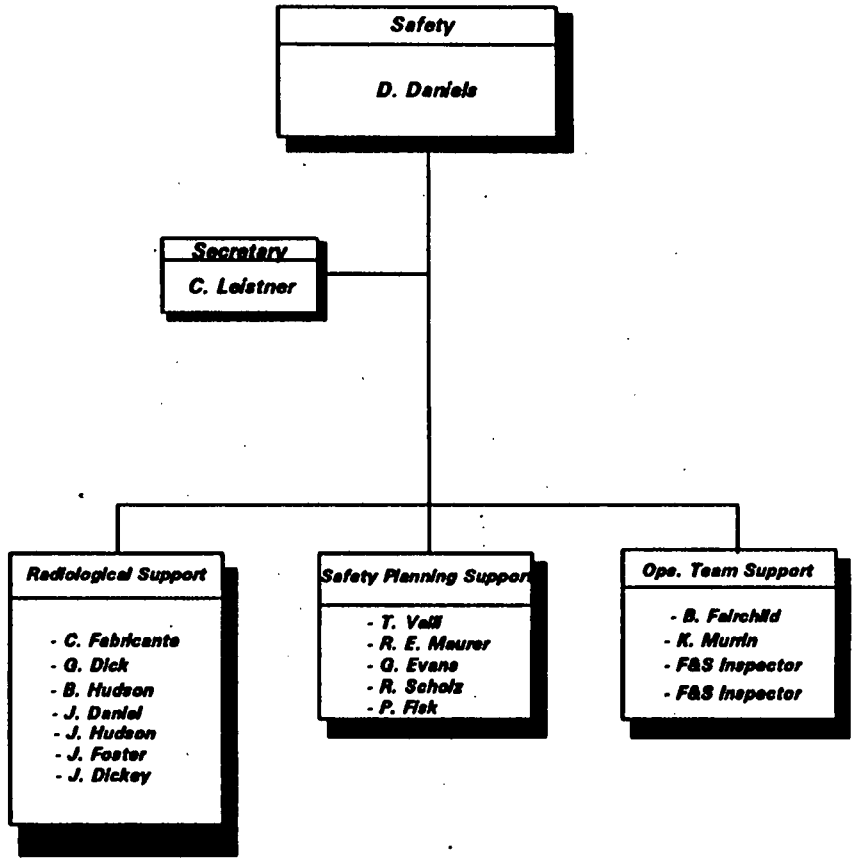
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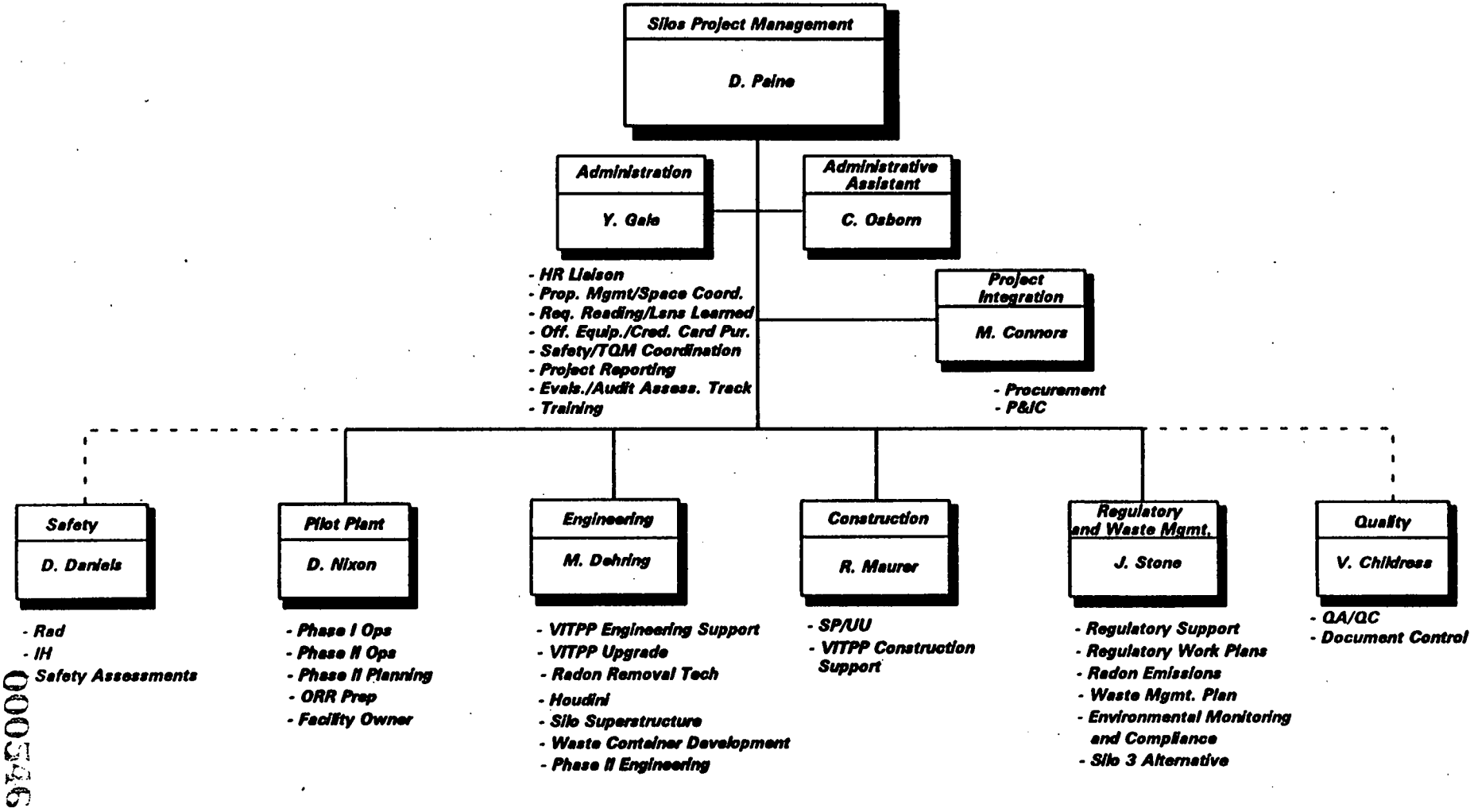
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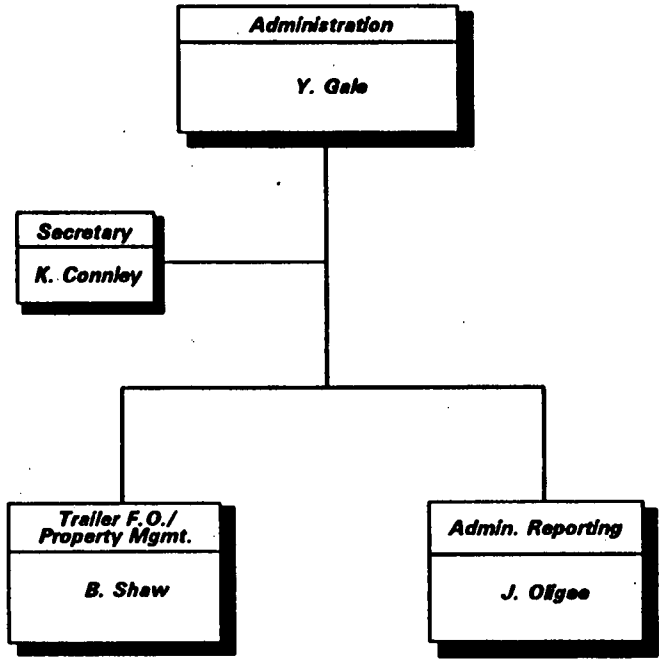
SILOS PROJECT
FUNCTIONAL RESPONSIBILITIES



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ADMINISTRATION



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Attachment 6: Team Member Qualifications

Team I: Review of Safety Basis Documentation

Raymond Crawford, Team Co-Leader
Qualifications: Provided independent assessments of Engineering Operations, Nuclear Plant Operations, Fire Protection, Quality Assurance, Emergency Preparedness, Environmental Monitoring, and Radioactive Waste Disposal Programs for Senior Management. Twenty seven years experience in the Nuclear Industry including responsibilities for Engineering Management, Systems Safety, Regulatory Affairs, Systems Testing, and Decommissioning of Nuclear Facilities. B.S.Ch.E. and M.S.Ch.E. Wayne State University; Ph.D. Engineering, University of California; Registered Professional Engineer since 1982.

R. Douglas Daniels, Team Member
Qualifications: A Health Physicist with thirteen years of experience in the nuclear industry. Robert has been assigned in support of FDF OU4 activities for the past five years. He is currently acting as the VITPP Safety Project Manager responsible for six functional areas within the FDF Vitrification Pilot Plant Project; Emergency Preparedness, Fire Protection, Occupational Safety and Health, Industrial Hygiene, Radiological Protection, and Nuclear Safety. He has a Bachelor of Science Degree in Applied Science and Technology, Radiation Protection.

Team 2: Off-Normal Scenarios and Test Corrections

Donald Nordquist, Team Member
Qualifications: A senior level manager with twenty two years experience in the commercial nuclear utility industry. He has been a director of the quality assurance function for two nuclear utilities. In these positions, he was responsible for identifying and resolving quality issues and interface with the Nuclear Regulatory Commission. Don chaired a management level oversight team to identify and resolve engineering and testing issues associated with motor operated valves.
At Fernald, he assisted the Vitrification Pilot Plant personnel in their preparation for operational readiness. He is currently working in Conduct of Operations and performing Pre-Operational Assessments. He is a member of the Independent Safety Review Committee (ISRC) at Fernald. He has a Bachelor of Science Degree in Industrial Engineering.

Team 3: Response to Incident

William Previty, Team Member

Qualifications: A consultant engineer with over 30 years management, operations, and engineering experience in the nuclear programs in the private sector and naval nuclear power. He has held several senior management positions for supervision and operation of nuclear reactors and facilities with concurrent management responsibilities for nuclear safety, maintenance, training, quality assurance, environmental, and assessment programs. He was a nuclear submarine commander and was a former Navy Deputy Director of Test and Evaluation, and Technology Requirements. In over two and one-half years at Fernald, he has worked as a consultant for conduct of operations and readiness programs supporting major projects and has served as Team Leader for Operational Readiness Assessments and as an ad hoc member of the Independent Safety Review Committee. Mr. Previty has a B.S. in Engineering, U.S. Naval Academy, and completed the Naval Reactors Senior Officer Nuclear Power Course.

Lou Bogar, Team Co-Leader

Qualifications:

Louis C. Bogar has over 40 years experience in nuclear science and technology. He graduated from the Massachusetts Institute of Technology where he studied Nuclear Chemistry and Engineering. For 28 years he was at the Westinghouse Bettis Atomic Power Laboratory where he held several senior management positions among which were management of nuclear safety, radiological controls, and reactor materials performance for the Laboratory and oversight of chemistry and radiological controls at the Naval Reactors Facility prototype reactor plants and the Expended Core Facility. In addition, he was assigned by Naval Reactors as Radiological Controls Supervisor for the Duquesne Light Co. At the Shippingport Atomic Power Station during defueling of PWR Core 2 and fueling with the Light Water Breeder core. More recently, he was the senior manager for ES&H at the Feed Materials Production Center with the Westinghouse Materials Co. Of Ohio. During his six years at Fernald he also managed the development of a Westinghouse Radiological Controls Manual for use by all Westinghouse GOCOs which later became the model for the DOE Radiological Controls Manual. Mr. Bogar was a member of the Westinghouse Nuclear Safety and Environmental Oversight Committee which was responsible for senior management oversight of activities and programs at all Westinghouse GOCOs. He retired from Westinghouse in 1992.

000549

Attachment 7: Documents Reviewed

Abriam, Rosemary O.; Baskett, Ronald L.; Crew, Daryl; Moore, Miki; Spruiell, Mark E.; and Vogt, Phillip J.; *ARAC Site Workstation System User's Guide*; Lawrence Livermore National Laboratory; University of California, Livermore, CA (February 1995)

Auditable Safety Record 95-0006 for Phase I Vitrification Pilot Plant Operations

Building Emergency Procedure, EM-0020

Chronology of Failure Considerations for the Air Bubbler Tubes

Code of Federal Regulations, Energy 10 Parts 500 to End, *Occupational Radiation Protection*, 10 CFR 835, U.S. Government Printing Office, Washington (December, 1993)

Configuration Management, CM-0001, (April 1996)

Daily Operations Report, Unusual Occurrence OH-FN-FDF-FEMP-1996-0075, Vitrification Pilot Plant Melter Breach, December 26, 1996

Design Change Notice DCN CONS\1636: 068 Rev. 0 (Approved 10/11/95)

Design Change Notice DCN CONS\1636: 068 Rev. 1 (Approved 1/11/96)

Design Change Notice DCN CONS\1636: 068 Rev. 2 (Approved 3/12/96)

Design Change Notice DCN CONS\1636: 068 Rev. 3 (Approved 4/9/96)

DURATEK Letter (DURATEK to FDF), Response to PARSONS ES&H Questions Concerning OU-4 Pilot Plant Furnace Area, L-95388, (1/23/94)

DURATEK Letter (DURATEK to FDF), RFP:109420: Joule-Heated Glass Furnace and Amendment 1, therto, L-94845, (6/2/94)

DURATEK Letter (DURATEK to FDF), RFP:109420: Joule-Heated Glass Furnace-Questions, L-94910, (6/22/94)

Engineering Design Change Process, 12-5002 (September 1996)

ENGINEERING, INC., *MicroShield®*, Version 4.21 Rockville, MD (February, 1995)

Environmental Impacts Due to Melter Bottom Drain Event, Robert Frost

Event Notification and Occurrence Reporting, EM-0010

Fact Sheet, Unusual Occurrence ON-FN-FDF-FEMP-1996-0075, Vitrification Pilot Plant Melter Breach, December 26, 1996

FERMCO Configuration Management Plan, PL-3035 (September 1994)

FERMCO (FDF), Technical Basis for Internal Dosimetry at the Fernald Environmental Management Project, SD-IRST-BAS-3008, (December 1995)

FERMCO, (FS), Fire Hazards Analysis Update Building 94 Vitrification Pilot Plant (February 1996)

FERMCO Letter (FDF to DOE), Responses to the VITPP Melter/Gem Former Value Engineering Review, C:P:(FRVP):96-0012, (1/26/96)

FERMCO Letter (FDF to DURATEK), CRU4 Melter/Gem Machine Design Review Resolution, C:CRUP:(CRU4/pp):95-0020, (5/5/95)

FERMCO Letter (FDF to DURATEK), Melter/Gem Maker Off-Normal Scenarios, C:CRUP:(CRU4/PP):95-0009, (2/9/95)

FERMCO Memorandum, Closeout of Technical Review Board Issues from the November 29, 1995 Meeting, M:P:(FRVP):96-0035 (1/25/96)

FERMCO Memorandum, CRU4 Vitrification Pilot Plant Design Review, M:CRUP:(CRU4/PP):95-0061, (5/16/95)

FERMCO Memorandum, Design Changes to VITPP PG-3 Components, M:P:(FRVP):96-0207, (4/19/96)

FERMCO Memorandum, Melter/Gem Maker Off-Normal Scenarios, M:CRU4(OR):94-038 (1/9/94)

FERMCO Memorandum, Site Power Outage-Recommendations for the Vitrification Pilot Plant, M:P:(FRVP):96-0190, (4/3/96)

FERMCO Memorandum, Structures, Systems, and Components, Performance Grade Evaluation, M:P:(FRVP):96-0246, (5/2/96)

FERMCO Memorandum, Structures, Systems, and Components, Performance Grade Evaluation, M:P:(FRVP):96-0311, (6/6/96)

FERMCO Memorandum, VITPP Safety Basis and Schedule, M:P:(FRVP):96-0244, (5/2/96)

FERMCO, Operable Unit 4 Vitrification Pilot Plant Phase II Test Plan, 18-SU-0004, Rev 0, (April 1985)

FERMCO, (OS), Operating Facility Health & Safety Briefing (Operational Safety Guidance), Rev. 3, (December 1996)

FERMCO, (OU4), Request for Safety Assessment, Operable Unit 4, Phase I (February 1995)

FERMCO, (SA), Final Hazard Analysis Report for Operable Unit 4, 25-HS-0004, (September 1995)

FERMCO, (SA), Final Human Factors Evaluation for Design Review of CRU4 VITPP, SA 95-0045, (March 1996)

FERMCO, (SA), Hazard and Operability Analysis Report for the Operable Unit 4 Vitrification Pilot Plant, 25-HS-0005, (September 1995)

FERMCO, (SA), Integrated Hazard Analysis: Preliminary Hazard Analysis Plan (July 1994)

FERMCO, (SA), Preliminary Integrated Hazard Analysis (PHA) for the CRU4 Pilot Plant, (July 1994)

FERMCO Technical Proposal Review, Joule-Heated Vitrification Furnace, Revision Date June 14, 1994

Final Hazard Analysis Report for Operable Unit 4, October 12, 1995

Fire Hazard Analysis, April 5, 1995

Glass Furnaces, Rakow Library Corning Museum of Glass, Society of Glass Technology, Sheffield, 1987

Homann, Steven G., Hotspot Health Physics Codes for the PC, Lawrence Livermore National Laboratory, University of California, Livermore, CA (March 1994)

Integrated Hazard Analysis: Preliminary Hazard Analysis Plan, July 7, 1994

Melter Specifications

Occurrence Report, Update Report, January 14, 1997

OE Weekly Summary 97-01

Operable Unit (OU4) Vitrification Pilot Plant Project Phase I Training and Qualification Program Description 25-TP-001

OU4 VITPP Phase I Test Plan, 18-SU-0003, Rev. 2 (May, 1996)

OU4 VITPP Phase II Test Plan, 18-SU-0004, Rev. 0 (April, 1994)

PARSONS Letter to FDF, Bottom Drain Capacities, PARSONS ID# 04:085:144:0001-97,(January 1997)

Performance Grading, ED-12-4015, Rev. 1

Project Execution Plan for Operable Unit 4 Vitrification Pilot Plant (PEP), 25-WP-0017 (May 1996)

Spill Incident Reporting and Clean-up, EP-0004

Standing Orders for Vitrification Pilot Plant Operations, M-134

U.S. DOE, *Airborne Release Fractions/Rates and Respirable Fractions for Nonreactor Nuclear Facilities*, DOE Handbook 3010-94, Vol. I, Table 3-8, Washington, D.C. (December 1994)

U.S. DOE, *Operable Unit 4 Treatability Study Report for the Vitrification of Residues from Silos 1, 2, and 3*, Fernald Environmental Management Project, Fernald, OH (May 1993)

U.S. DOE, Preliminary Safety Analysis Report (PSAR) for Operable Unit 4, FEMP-2337, Rev. 0, (April 1994)

U.S. DOE, *Remedial Investigation Report for Operable Unit 4, Final, Volume 1 of 3*, Fernald Environmental Management Project, Fernald, OH (November 1993)

USQD and Safety Evaluation System, NS-0002 (February 1994)

Vitrification Pilot Plant Data Acquisition and Control System Alarms, 11-A-001

Vitrification Pilot Plant Emergency Operating Procedure Loss of Electrical Power, 11-E-001

Vitrification Pilot Plant Emergency Operating Procedure Plant Emergency Shutdown, 11-E-002

Vitrification Pilot Plant Melter Bottom Draining, 11-C-281

Vitrification Pilot Plant Melter Control Panel Alarms and Gem Machine Control Panel Alarms, 11-A-002

Vitrification Pilot Plant Melter Operation, 11-C-269

Vitrification Pilot Plant Operational Safety Guidance Document

WVDP, *Safety Analysis for Vitrification System Operations and High Level Waste Interim Storage*, WVNS-SAR-003, Rev. 2, West Valley Nuclear Service GROVE Co., Inc., West Valley, NY (June 1995)

Signature Page

Prepared By: Louis C. Bogar — 2/13/97
Louis Bogar, Team Co-Leader — Date

Prepared By: Raymond Crawford — 02/13/97
Raymond Crawford, Team Co-Leader — Date

Prepared By: R Douglas Daniels — 2/13/97
R. Douglas Daniels, Team Member — Date

Prepared By: [signature] — 2/13/97
Donald Nordquist, Team Member — Date

Prepared By: William Previty — 2/13/97
William Previty, Team Member — Date

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ATTACHMENT C

VITRIFICATION PILOT PLANT

DATA ANALYSIS AND PATH FORWARD TEAM REPORT

FERNALD ENVIRONMENTAL MANAGEMENT PROJECT
VITRIFICATION PILOT PLANT
MELTER INCIDENT:
DATA ANALYSIS AND PATH FORWARD TEAM

FINAL REPORT
40100-RP-0020
REVISION 0
FEBRUARY 1997

PREPARED FOR:
VITRIFICATION PILOT PLANT MELTER INCIDENT INVESTIGATION

BY:
THE DATA ANALYSIS AND PATH FORWARD TEAM

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1.3 Assumptions 1-5
1.4 Team 1-6

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ACRONYMS

A/cm^2	amperes per square centimeter
ASTM	American Society of Testing and Materials
C	Celsius
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
DOE	U.S. Department of Energy
DOE-FEMP	U.S. Department of Energy - Fernald Environmental Management Project
EPA	U.S. Environmental Protection Agency
FDF	Fluor Daniel Fernald
Fe	iron
FEMP	Fernald Environmental Management Project
FY	fiscal year
HLW	high-level waste
in.	inch
IRT	Independent Review Team
kg	kilogram
kg/d	kilogram per day
Kg/hr	kilogram per hour
LAW	low activity waste
lbs./hr	pounds per hour
lbs.	pounds
lbs./d	pounds per day
m	meter
MAWS	Minimum Additive Waste Stabilization (facility)
MT/d	metric ton per day
OEPA	Ohio Environmental Protection Agency
pH	potential of hydrogen
PNNL	Pacific Northwest National Laboratory
PNNL HTM	Pacific Northwest National Laboratory High-Temperature Melter
ppm	part per million
RCRA	Resource Conservation and Recovery Act
RMI	Reactive Metals, Inc.
SRS	Savannah River Site
TCLP	Toxicity Characteristic Leaching Procedure
TVS	Transportable Vitrification System
VITPP	Vitrification Pilot Plant
VSL	Vitreous State Laboratory

1.0 INTRODUCTION

This report recommends the path forward for the Vitrification Pilot Plant (VITPP) project at the Fernald Environmental Management Project (FEMP) located northwest of Cincinnati, Ohio. This report is one of three reports prepared by three teams that investigated a non-radioactive leak in the vitrification melter at the U.S. Department of Energy-owned (DOE) facility.

The Data Analysis and Path Forward Team for the investigation of the VITPP incident was chartered to evaluate data and determine deficiencies for detailed design and operation, determine the need for low-temperature melts and alternatives to VITPP restart, determine melter "post-incident" data needs, and determine "path forward" for the VITPP Phase I Test Program. The team's output is:

- A data matrix of outstanding data requirements and operations experience that would have been gained from the balance of Campaign 4 to support full-scale detailed design
- Recommendations for acquiring outstanding Phase I data and operating experience
- Options for the path forward for the VITPP Phase I Test Program
- Cost/benefit analysis for path forward options.

1.1 Background

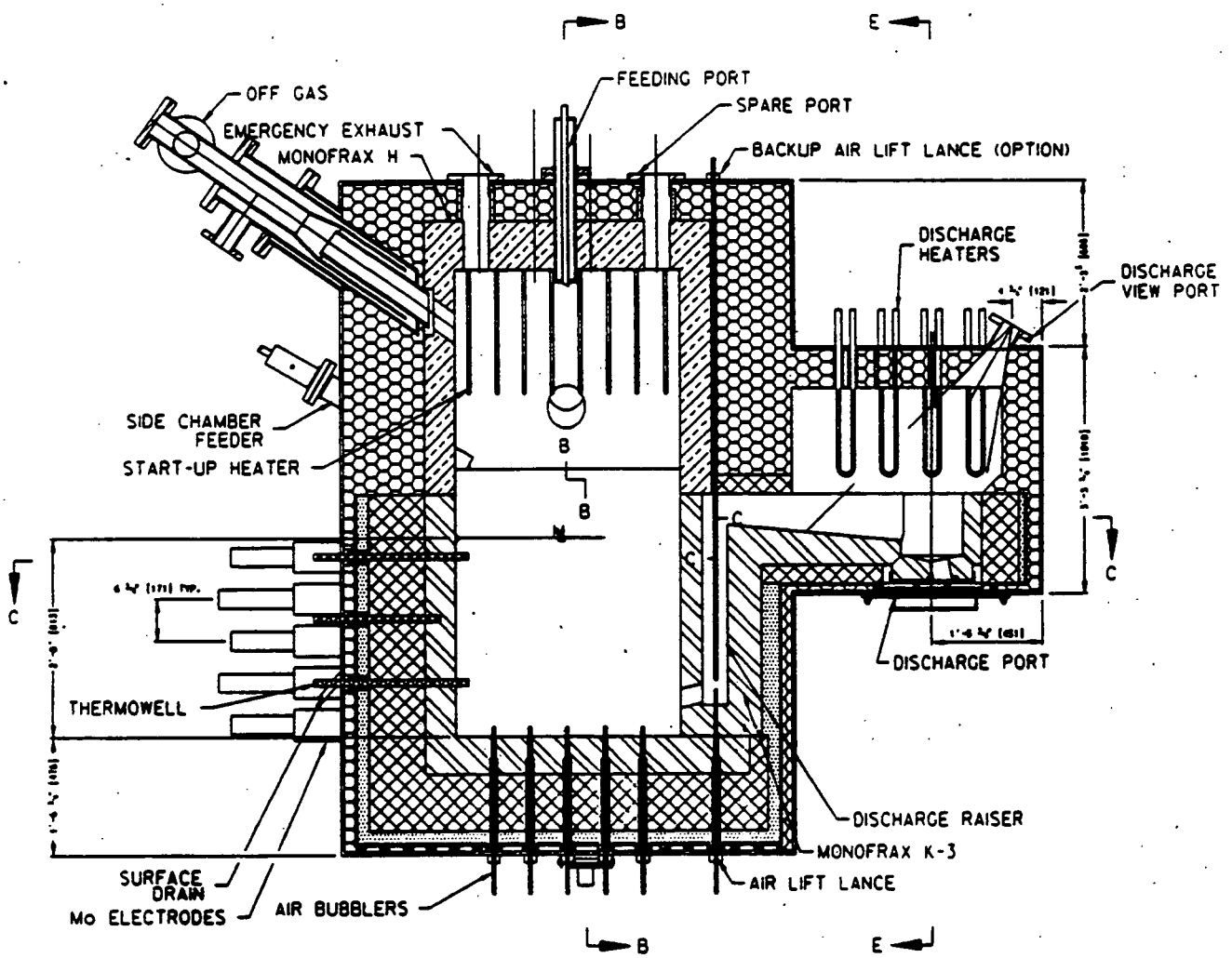
The VITPP is located on the west perimeter of the FEMP and is the administrative responsibility of the Silos Project. The VITPP is a two-phase treatability study designed to provide remedial design information for four waste storage silos, collectively known as Operable Unit 4. The silos are being remediated under the Comprehensive Environmental

Response Compensation and Liability Act (CERCLA) and related agreements between the DOE and the United States Environmental Protection Agency (EPA) and the Ohio EPA (OEPA). Fluor Daniel Fernald (FDF) manages FEMP remediation for the DOE. The area includes four waste storage silos, their ancillary structures and the surrounding soils, and the VITPP. Silos 1 and 2, also known as the K-65 silos, contain radium-bearing, low-level radioactive waste. Silo 3 contains dry, radioactive, metal oxide waste. Silo 4 was never used and is empty except for rainwater seepage. The VITPP contains the vitrification furnace and systems for product-forming and handling, off-gas treatment, furnace feed preparation and transfer, wastewater treatment, process support auxiliaries, and a control room.

The melter and facilities were designed for a production rate of one metric ton per day for vitrification of K-65 (Silos 1 and 2) and Silo 3 residues. The VITPP is the third and final tier of an exhaustive treatability program, culminating in the design of facilities and equipment for the final remediation of Operable Unit 4. The pilot plant program is being conducted in two phases. Phase I, which is currently in progress, will process surrogate materials to demonstrate the vitrification technology. Phase II, which is planned to follow Phase I testing, would utilize actual silo residues for confirmation of the surrogate testing.

The VITPP primary component is a unique joule-heated melter (see Figure 1-1) designed for high-temperature service with waste residues that contain significant concentrations of lead and sulfates. The melter is unique due to its three-chamber design. The center, or main chamber, contains the corrosive waste slurries, while the melter electrodes are housed in electrode chambers on each side of the main bath. The electrode chambers contain benign glass and are designed to isolate the 10 molybdenum electrodes from the corrosive/oxidizing main glass pool.

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SIDE VIEW (SECTION A-A)

Figure 1-1

On December 26, 1996, at approximately 2222 hours, a breach in the bottom of the melter resulted in the non-radioactive molten glass draining to the bottom drain container. At this point in the test program, Phase I was nearing completion with nine batches of slurry feed remaining and was scheduled for completion in January 1997. To date, the VITPP had produced over 30 tons of high-quality glass. The knowledge and experience gained through the operation and safe shutdown of the pilot plant has provided invaluable insight and lessons learned required for the design, construction, and operations of the full-scale facility.

In response to this event, FDF and DOE-FEMP have formed the following three teams:

- Data Analysis and Path Forward Team
- Incident Analysis Team
- Safety Review Team.

Each team has a specific charter and deliverables. This report deals only with the findings of the Data Analysis and Path Forward Team.

1.2 Objectives

The primary objectives of the Data Analysis and Path Forward Team were to evaluate all of the existing data to date (especially from Phase I of the VITPP) and to determine what additional data are needed for preparation of a high-quality melter performance specification and for detailed design of the rest of the facility. While focus was placed on addressing the data that would have been provided by completing the Phase I testing, further evaluation was given to the data needs for Phase II testing. Once data needs were identified, the team developed options to acquire the data. Both on-site (VITPP) and off-site sources were evaluated, and a recommendation for data acquisition was made.

The third objective of the team was to recommend the near-term path forward of the VITPP.

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The following deliverables were identified and are addressed in this report:

- A data matrix of outstanding data requirements and operations experience that would have been gained from the balance of Campaign 4 to support full-scale detailed design (Table 2-1).
- Options and recommendations for acquiring outstanding Phase I data, additional data and operating experience, as well as additional data required for full-scale design (Table 4-1).
- A recommendation for the path forward for the VITPP Phase I Test Program (Section 5.0).
- A comparison of cost, schedule, and risk analysis for path forward options (Section 4.0).

1.3 Assumptions

The Data Analysis and Path Forward Team developed the following assumptions in the determination and evaluation of data needs and the options to provide the needed data:

- Evaluation of data needs addresses Silos 1 and 2 materials only, due to the fact the remaining Phase I testing addressed only those residues.
- Melter(s) design, fabrication, and installation will be procured under a performance specification.
- FDF will design/build/operate the full-scale vitrification facility.
- Toxicity characteristics leaching procedure (TCLP) will be met with low-temperature operations; an acceptable glass formula can be produced. (The low-temperature formula planned for VITPP testing was developed at FDF as a low-viscosity alternative to the formula developed by The Catholic University Vitreous State Laboratory [VSL]). TCLP results from crucible tests at FDF, for the lower viscosity glass, are well within allowable limits.)
- Laboratory testing is always required prior to any scale of melter testing.

- Radioactive testing or testing of actual silo residues will be performed. That is, further glass chemistry development with radioactive residues will be performed to support the preparation of design documents. Therefore, some Phase I data requirements are deleted, primarily in the area of off-gas composition.

- A waste retrieval demonstration will be performed. Slurry testing will be included in the waste retrieval demonstration, which will supplement the Phase I slurry hydraulics data.

- Minimal scale-up results in decreased risk.

- Evaluation of melter options was confined to joule-heated melters only, due to time constraints.

1.4 Team

The Data Analysis and Path Forward Team is comprised of a diverse group of professionals from the Fernald site, as well as external experts from the vitrification industry. Team members, with biographies, are identified in Appendix C.

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2.0 METHODOLOGY

This section documents the steps taken to develop data needs and the path forward for meeting those needs.

The first activity was to review the previously identified data needs required to support remedial design as provided by planned Phases I and II operations. An original data matrix for each system was issued on February 7, 1996, in a letter entitled, VITPP Physical Sample Points for Phases I and II Operations. This data matrix (provided in Appendix A) was very detailed, including specific data needs, as well as the instrument to be used, frequency of recording, expected values, etc. However, the lack of definition of systems available for testing led the team to conclude that providing the same level of detail in a subsequent matrix would have little value. Specific information and data requirements will be identified when the procurement documentation is developed for contracting this work. For clarification, a simplified data matrix was developed to provide an understanding of the type of information that is needed, rather than the specific data (Table 2-1).

The data needs identified in Table 2-1 (Column 4) are grouped into four categories:

- Phase I
- Phase II
- Part of waste retrieval demonstration
- New (identified as a result of the melter incident).

Options for acquisition of needed data were then evaluated and categorized by the minimum scale of facility needed to develop sufficient data that would provide confidence for use in the design of the final remediation facility for Silos 1 and 2. In many cases, if the data were obtained in a larger-scale facility, the confidence level regarding potential "scale-up" would be higher. The options were categorized into four categories (Column 3 of Table 2-1):

A. Laboratory scale - crucible

B. Mini-melter - ~10 kilogram (kg) - 100 kg/day

C. Pilot-scale melter - ~1 metric ton per day (MTD)

D. Full-scale melter - ~6 MTD

Next, specific facilities (located both on and off site) that could provide the required data were identified for each category (Table 2-1). Then, cost, schedule, and risk analyses were compared for each facility. During evaluation of the facility options, emphasis was placed on schedule impact and the technical risk of each specific option providing the needed data. The scope of data gaps was also considered.

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TABLE 2-1
DATA NEEDS SUMMARY

System: Melter

Data Need	Reason for Data	Data Acquisition Options	Original VITPP Phase(a)	Remarks
1. Series A - Silos 1 and 2 low temperature (1,150°C) steady state operations at 1 MT/d	Determine feasibility of low temperature design	C. PILOT-SCALE MELTER	I	Validation of laboratory scale glass formulation at larger scale
2. Series B - Silos 1 and 2 w/bentonite steady state operation at 1 MT/d	Determine melter impacts from processing bentonite	C. PILOT-SCALE MELTER	I	Validation of laboratory scale glass formulation at larger scale
3. Melter maximum rate	Scale-up information (i.e., melter surface area vs. throughput anticipated)	C. PILOT-SCALE MELTER	I	VSL data indicated throughput greater than 1 MT/d at 1 m^2, VITPP achieved steady state throughput was 1.0 MT/d at 1 m^2; limited by feed and off-gas systems
4. Materials of construction (i.e., electrodes, refractory, etc.)	Determine corrosion rate of various components under anticipated service (i.e., temperature chemistry)	A. LABORATORY-SCALE - CRUCIBLE	New	As a result of the incident, further testing of electrode and refractory materials will be needed to design the next generation melter or VITPP upgrade

(a) I = Phase I data need
II = Phase II data need
Waste Retrieval Demo. = Waste Retrieval Demonstration data need
New = New data need identified in this report

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TABLE 2-1
DATA NEEDS SUMMARY

System: Glass Product Handling and Packaging

Data Need	Reason for Data	Data Acquisition Options	Original VITPP Phase	Remarks
1. Gem machine maximum production rate during steady state operations	Design of future product-forming equipment	M-Area - Gem Production Data Only	I	Viscosity and temperature of glass is critical to correlate with other facilities
2. Monolith devitrification analysis	Design of future product-forming equipment	Sample existing monoliths and analyze for devitrification	I	Critical to alternative analysis on other product forms (monolith)

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TABLE 2-1
DATA NEEDS SUMMARY

System: Waste Retrieval and Feed Preparation

Data Need	Reason for Data	Data Acquisition Options	Original VITPP Phase	Remarks
1. Rheology of waste slurries:	Provide design basis for waste retrieval and feed preparation equipment	Waste retrieval demonstration including silo waste retrieval and an in-plant test rig combined with laboratory testing	Waste Retrieval Demo.	Utilizing proven surrogate, demonstrate waste retrieval techniques, thickener tank performance, and feed preparation loop
a. Flow properties, viscosity as a function of concentration, temperature and shear pH and water content				Laboratory work shall be performed with actual silo residues to design surrogate for waste retrieval demonstration.
b. Particle size distribution				
c. Settling curves				
d. Impact on slurry rheology w/varying concentrations of bentonite				
e. Dewatering characteristics				
f. Suspended solids				
g. Density of slurries				
2. Hydraulics of waste slurries	Provide design basis for waste retrieval and feed preparation equipment	Waste retrieval demonstration including silo waste retrieval and an in-plant test rig combined with laboratory testing	Waste Retrieval Demo.	
a. Pressure drops in various piping configuration				
b. Flow rates				

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TABLE 2-1
DATA NEEDS SUMMARY

Data Need	Reason for Data	Data Acquisition Options	Original VITPP Phase	Remarks
3. Analysis of K-65 residue including: a. Compound analysis b. Radiolytic characteristics c. Radon emanation d. Particle size e. Elemental analysis	Validation that surrogates simulate actual residues	A. LABORATORY TESTING	New	Utilizing actual silo residues

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TABLE 2-1
DATA NEEDS SUMMARY

System: Melter

Data Need	Reason for Data	Data Acquisition Options	Original VITPP Phase	Remarks
1. Radioactive glass properties	Demonstrate correlation w/previous glass chemistry for: a. Target temperature b. Waste loading c. Viscosity d. Conductivity e. Liquidus temperature	A. LABORATORY SCALE - CRUCIBLE	II	Verify (in laboratory) that simulants simulate
	g. Redox state as it relates to lead and sulfur h. Lead and sulfate control	B. MINI-MELTER	II	Initial redox curves will be generated during laboratory testing

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TABLE 2-1
DATA NEEDS SUMMARY

System: Off-Gas

Data Need	Reason for Data	Data Acquisition Options	Original VITPP Phase	Remarks
1. Gas composition of off-gas from the target glass formulation, as a function of temperature: a. Radon concentration b. NO_x concentration c. SO_x concentration d. Solids carryover e. Miscellaneous	Design control/abatement equipment and acid gas removal equipment. Validate the material balance.	B. MINI-MELTER	II	Utilizing actual silo residues.

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TABLE 1
DATA NEED SUMMARY

System: Glass Product Handling and Packaging

Data Need	Reason for Data	Data Acquisition Options	Original VITPP Phase	Remarks
1. Determine final waste form, (e.g., gems, cullet, monoliths, frit, etc.)	Design of next generation of Vitrification Plant	a. Perform study of demonstrated technologies b. Perform surrogate testing analysis	II	Produce radioactive waste form to determine dose rate and radon emanation

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TABLE 2-1
DATA NEEDS SUMMARY

System: Health Physics

Data Need	Reason for Data	Data Acquisition Options	Original VITPP Phase	Remarks
1. Determine dose rate of slurry and glass and contact dose on facility components	Data will be used to validate dose modeling	C. MINI-MELTER	II	Utilize archive material or silo material to be retrieved

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3.0 IDENTIFYING OUTSTANDING AND ADDITIONAL DATA REQUIREMENTS

A system-by-system review of the original data matrix (Appendix A) was performed and the data were screened. In light of lessons learned from pilot plant operations to date, the screening process re-evaluated the need and the importance of the data. Data no longer relevant or required for design of the final remediation facility were deleted from the matrix. In addition, the level of detail listed in the original matrix (see Appendix A) was no longer appropriate, since the information requested was originally developed based on the actual configuration of the pilot plant. The type of information indicated in the original data needs matrix (Appendix A) is still required but the specific sample points do not apply. Table 2-1, the new simplified matrix, reflects the screening effort and lists the generic data required for development of the upgraded pilot plant and/or final remediation facility. A detailed scope of work will be developed for each of the four options (identified in Section 2.0) requesting specific data needs prior to initiating any testing.

During VITPP operations, many process enhancements were made, eliminating some of the operating constraints for the feed and off-gas systems. However, these modifications, combined with process lessons learned, resulted in the pilot plant no longer being prototypical of the proposed upgrade or final remediation facility. Consequently, data needs identified for the feed and off-gas systems were not fulfilled. Slurry handling and thickener performance data originally anticipated to come from pilot plant testing were moved from the Phase I Campaign 4 test to the waste retrieval demonstration. As a result of the melter incident, materials of construction testing were identified as a new data need critical to the design of a final melter.

In summary, the most important data needs required for the future design of the final remediation facility are compositional data from the optimized low-temperature glass formulations (Series A and B), rather than physical data. Physical data consisting of rheology

and hydraulic data of the slurry will be developed in the laboratory and simulated conditions will be confirmed in the waste retrieval demonstration. It is important to demonstrate that the behavior of the actual silo material closely resembles the surrogate material, therefore enabling various tests to be performed -- tests with differing variables to optimize the slurry-handling aspects of the facility and design parameters (throughput, redox state, etc.) for the full-scale melter. The compositional data that are required will support the design of the off-gas treatment system. Compositional data is defined as information regarding the composition of the off-gas and other streams that may be contaminated. This information will be used to develop the material balance and process flow diagrams for the final remediation facility.

Operation of the pilot plant during the testing phase has developed operating personnel with unique melter operating experience. Phase I operational testing of the VITPP has been the proving ground for the equipment, process, and methodology of the vitrification process. Although the VITPP was not necessarily prototypical of the final remediation facility envisioned for treatment of silo material, significant operational experience was gained during the testing of the feed preparation and slurry handling; vitrification of surrogate Silos 1, 2, and 3 materials, proving the ability to vitrify materials with sulfates, phosphates, high lead and barium; and off-gas control and treatment.

Throughout the planning and implementation of Silos Project activities (especially design, construction, and operation of the VITPP), lessons have been learned from a variety of internal and external sources. One of the primary sources of the lessons learned has been the evaluation of the VITPP operations. Other internal sources of lessons learned include design reviews, start-up, system operability, construction acceptance testing, formal and informal input from personnel involved in the project, and from the investigation of the melter incident. Applying this operations experience to the future vitrification program is critical because this material and process are unique.

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4.0 EVALUATION OF OPTIONS FOR OBTAINING DATA

This section provides a detailed analysis of the specific options for meeting the data needs, based on the minimum scale of testing required. Table 4-1 identifies the four facility scales, as well as specific options for performing the tests necessary to meet the data needs.

When developing the options available for meeting the data needs, the team's primary consideration was given to scale and the potential dynamics of the equipment used (i.e., crucible melts may not adequately demonstrate the actual dynamics of a melter, if required). These options are discussed in terms of the minimum scale for which the data would be representative.

The detailed alternatives have been evaluated for their ability to meet the technical requirements. In each case, the information was gathered informally and, therefore, is only considered preliminary at this time. Prior to initiating testing, additional formal discussions with potential vendors will be needed to confirm the assumptions and conclusions made in this section.

In general, where glass composition is the concern, it appears to be appropriate to perform crucible melts. On the other hand, composition and rate information to support the off-gas design is considered to be much more representative when samples are taken from a melter where a feed can be sustained and steady state operations achieved.

A major consideration with regard to the data required and the need for the data reflects the level of risk that is acceptable in the design of the final remediation facility. Much of the risk is considered to be in the scale-up; however, it is equally important that the equipment and process work efficiently. The potential success of scale-up is difficult to evaluate; and therefore, it is assumed that minimal scale-up results in decreased risk. Completion of the VITPP Campaign 4 (on or off site) may reduce some of this risk since the scale-up from the pilot scale to the final remediation facility is considered reasonable. The process verification

would be addressed concurrently with completion of the Campaign 4 requirements in a prototypical pilot-scale facility.

As shown in Table 4-1, there are:

- Five options for laboratory scale
- Five options to meet mini-melter requirements
- Nine options to meet pilot-scale melter requirements
- Two options to meet full-scale melter requirements.

The rest of this section provides an evaluation of each option.

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TABLE 4-1
DATA ACQUISITION OPTIONS

Facility Requirements	Options
A. Laboratory scale - crucible (may include other laboratory-related activities in addition to vitrification testing)	1. Catholic University VSL 2. Clemson University 3. FEMP - laboratory 4. PNNL 5. SRS
B. Mini-melter ~10 kg - 100 kg/day	1. Clemson University 200 pounds (lbs)/day 2. Catholic University 10-100 kg/day 3. West Valley pilot melter 4. PNNL 5. Commercial facilities
C. Pilot-scale melter ~1 MT/day	1. VITPP at current capacity - single chamber 2. VITPP at current capacity - three chambers 3. Catholic University ~1 MT/d 4. TVS ~3 MT/d 5. PNNL - HTM .2 m^2 6. Commercial facilities - Vectra, others 7. SRS - Stir Melter 8. MAWS Facility 9. M-Area (for gem production data only)
D. Full-scale melter ~6 MT/day	1. Module #1 (~6 MT/d) of planned remedial facility 2. VITPP with upgraded capacity

Catholic University (VSL) = The Catholic University Vitreous State Laboratory
MAWS = Minimum Additive Waste Stabilization (facility)
PNNL = Pacific Northwest National Laboratory
PNNL HTM = PNNL High-Temperature Melter
SRS = Savannah River Site
TVS = Transportable Vitrification System

4.1 Option A - Laboratory Scale

The laboratory-scale scope of work includes testing corrosion rates of construction material; comparing surrogate material to actual waste; and studying low-temperature glass formula development, redox, and rheology of the K-65 residues. Table 4-2 summarizes the evaluation of sources, and details of the evaluation are provided, followed by the scope of work explanation.

The evaluation of options for the laboratory-scale work focused on the capabilities of potential vendors, since cost and schedule were essentially the same. The cost estimate, approximately $280,000, is considered rough order of magnitude or budgetary only. The components of the estimated cost are shown in Appendix B. The estimated schedule is five to six months for all vendors. The schedule could be shortened if the corrosion/erosion testing is separated from the laboratory testing package and is issued to a separate vendor. Both crucible melts and the corrosion/erosion tests, performed in accordance with American Society of Testing and Materials (ASTM) procedures, would utilize laboratory furnaces and each vendor has a limited number.

4.1.1 Evaluation of Laboratory-Scale Sources

All of the vendors evaluated appeared capable of performing the work. However, there are some differences in the manner in which the work would be accomplished between the vendors. These differences are described in the following paragraphs.

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Table 4-2
Evaluation of Laboratory-Scale Sources

Location	Cost	Schedule	Risk
1. FDF Laboratory	$300K	6 months	L
2. Catholic University	$300K	6 months	L
3. Clemson University	$300K	6 months	L
4. PNNL	$300K	6 months	L
5. SRS	$300K	6 months	L

Option A-1, FDF Laboratory. The FDF laboratory has most of the equipment for the laboratory-scale studies. Redox determination and some of the rheology samples would be sent to other laboratory(s), such as Corning and/or Florida International University, to perform the balance of the testing. Glass making is relatively new to FDF; and therefore, the staff may not be as experienced and qualified as staff at other laboratories. However, FDF could perform the corrosion/erosion tests in accordance with standard ASTM procedures.

Option A-2, The Catholic University Vitreous State Laboratory (VSL). The Catholic University VSL has the capability to perform most of the required tests. Some of the rheology samples would be sent to a separate laboratory(s). The risk of satisfactorily accomplishing this work is low since The Catholic University has significant experience in performing this type of work.

Option A-3, Clemson University. Clemson University has the capability to perform most of the required tests. Some of the rheology samples would be sent to a separate laboratory(s). The risk of satisfactorily accomplishing this work is low since Clemson University has significant experience in performing this type of work.

Option A-4, PNNL. PNNL has the capability to perform most of the required tests. Some of the radioactive rheology samples will need to be sent to a separate laboratory(s). The risk of satisfactorily accomplishing this work is low since PNNL has significant experience in performing this type of work.

Option A-5, SRS. SRS has the capability to perform most of the required tests. Some of the rheology samples will need to be sent to a separate laboratory(s). The risk of satisfactorily accomplishing this work is low since SRS has significant experience in performing this type of work.

4.1.2 Option A - Laboratory-Scale Scope of Work

The laboratory-scale scope of work includes the five following tests.

Corrosion Rates of Construction Material. Sufficient corrosion and erosion data of the refractory and electrode material is not available, especially at temperatures in the range of 1,100 - 1,400°C. However, as evident by wear shown in the FEMP's pilot plant melter and the refractory and electrode materials' ability to reduce lead compounds in the waste to lead metal, the use of molybdenum and molybdenum disilicide in a melter's bath is not recommended for processing K-65 residues. In accordance with standard ASTM testing procedures, tests will be performed to compare the relative corrosion (erosion) rates of possible material of construction. The knowledge of the absolute corrosion (erosion) rates is less important than the relative rates because these materials can be compared to those materials used in the VITPP. Corrosion data for the electrode material should be obtained in a test within a joule-heated melter where the electrode materials can be exposed to both electrical current and the hot glass. Corrosion rate is a function of the current density (amperes per square centimeter [A/cm^2]). For example, higher corrosion rates are expected with higher current density. Alternative testing

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methods could be developed in accordance with ASTM procedures that may provide appropriate information at a similar level of confidence.

Table 4-3 gives an overview of materials that should be considered for testing. The existing materials used in the VITPP should be included in the test and used as a basis for comparison of corrosion (erosion). Common stainless steel also should be tested, because some ancillary equipment made of stainless steel (e.g., gem-making machine, canister, etc.) may contact the molten glass at lower temperatures.

Comparing Surrogate to Actual Waste. The K-65 residues have been analyzed for elemental composition and variability. However, little is known about the chemical composition or bonding of the elements as determined by the elemental analysis. Therefore, the K-65 residues should be analyzed to gain a better understanding of the chemical composition of the waste. Some questions to answer include:

- Does the sulfur in the waste bond mostly to barium as barium sulfate or bonded to other metals?
- Is the lead mostly as lead oxide, or as some other compound?

The K-65 surrogate recipes should be modified, if necessary, to provide a better representation of the chemical composition of the K-65 waste. Crucible melts should be performed to determine if the surrogate recipes accurately simulate the K-65 residues. This can be accomplished by making the same glass, via crucible melts, from actual K-65 residues and from the surrogates. The behavior of the actual K-65 material and the surrogates can be compared for the following characteristics, at a minimum:

- Elemental composition in the slurry and glass
- Glass viscosity, as a function of temperature

Table 4-3
Corrosion/Erosion Testing Matrix

Material	K 3®	Mono-frax E®	Mono-frax® H/Mono-frax S-5®	ER 2161®	ER 1681®	ER SOL®	SERV-30®	SnO_2	Inconel ®				Other	Platinum	Stainless steel
									690	601	625	617			
Refractory material	*	*	*	*			*								
Melter plenum material					*	*			*	*			* HR 160 MA758		
Electrode material		R						*	*						
Materials for ancillary equipment									*P	*	*	*		*	R

Legend: P = preferred material
R = only as reference material
ER = from SEPR (France)
SERV-30 = North American Refractory Company (NARCO)

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- Glass conductivity, as a function of temperature
- Glass liquidus temperature
- Sulfate destruction, as a function of temperature

If the surrogates do not behave like the actual wastes, adjustments must be made, as necessary, and the crucible melts performed again. This can be included in the low-temperature glass formula development study.

Low-Temperature Glass Formula Development. Most of the emphasis has been on producing glass at 1,250°C; only a few crucible melts have been performed to develop low-temperature glasses (1,150°C). Consequently, the glass chemistry has not been optimized for the best combination (compromise) of waste loading versus processing parameters (e.g., conductivity, viscosity, and liquidus temperature) and sensitivity to change in the waste composition for the lower temperature. A low-temperature glass, known as S12L-01, has been developed and can be used as a starting point since the TCLP leachability measured <0.5 parts per million (ppm) lead for this glass. It is estimated that approximately 20 crucible melts, with accompanying analytical work, are necessary to determine effectively the best glass chemistry formula. Appendix B provides this estimate.

Redox Study. A reductant, urea, is used to aid in processing and destroying the sulfates in the K-65 wastes. However, it is not known what amount of reduction causes the precipitation of lead sulfide and/or lead metal in the molten glass. Therefore, a series of crucible melts should be performed to produce redox curves for Silos 1 and 2 glasses; this will allow determination of the redox potential (measured as a ratio of Fe^{+2}/Fe^{+3} in the glass) that lead sulfide and metallic lead precipitate from Silos 1 and 2 glasses. The curve then will be validated in mini-melter runs (described later in this section).

Rheology of the K-65 Residues. The surrogates used thus far were based on the analyzed elemental composition of the silo residues and are pertinent only to vitrification or making glass in the melter. They were not developed specifically to accurately simulate the rheology of the silo residues. More information needs to be known about the rheology of the silo residues to select and size waste retrieval and feed preparation equipment for a vitrification or solidification process. Therefore, the following parameters should be empirically determined in the laboratory for K-65 slurries using actual K-65 residues:

- Flow properties, viscosity as a function of concentration, temperature shear, pH, and water content
- Particle size distribution
- Settling curves
- Impact on slurry rheology with varying concentrations of bentonite
- Dewatering characteristics
- Suspended solids on total dissolved solids
- Density of slurries.

After the determination of the above parameters, a nonradioactive and nonhazardous surrogate shall be formulated that can be used for the testing of pilot and full-scale equipment.

4.2 Option B - Mini-Melter

This section provides a rationale for mini-melter tests, explains the scope of work, and evaluates the potential sources to perform the mini-melter tests described in the scope of work.

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4.2.1 Overview of Option B - Mini-Melter

At a minimum, testing of radioactive material should be performed initially at a mini-melter scale that is typical of the configuration and materials of construction to be used in the full-scale design.

The use of molybdenum or molybdenum disilicide in the melt bath is not recommended, as evidenced by the corrosion shown in the VITPP melter. For example, the Envitco melter at Clemson University uses molybdenum electrodes that are in direct contact with the melt bath. One could replace electrodes with Inconel metal electrodes. However, as a result of end-firing, the Inconel electrode could overheat at the tips, causing them to deform or melt. This restricts the operating temperature. Clemson also offers a Stir Melter mini-melter for use. Unfortunately, its recommended operating temperature is 1,050°C, which is too low to effectively destroy sulfates; additionally, its unique design does not provide scale-up data that would be applicable for more conventional melter designs.

Some mini-melters available for use are simply too large to use for the limited amount of materials available to use for the mini-melter tests. The K-65 materials would be consumed before the mini-melter operation stabilized.

The Catholic University has a 10-kg single, low-temperature melter and a 10-kg, high-temperature, three-chamber melter which are of the right size and configuration for use in the mini-melter studies.

It is recommended that the mini-melter testing be performed after materials of construction testing. An option to the known facilities (sources) listed is to subcontract

the construction and operation of a new mini-melter. The new design could be constructed of the most promising materials of construction, as determined by testing and incorporation of lessons learned to date. This type of testing would impact the schedule, but would provide prototypical data at a mini-melter scale to be utilized in the final design.

4.2.2 Evaluation of Mini-Melter Sources

Five facilities were evaluated as potential sources to perform mini-melter testing. The evaluation follows and is summarized in Table 4-4.

Table 4-4
Evaluation of Mini-Melter Sources

Location	Cost	Schedule	Risk	Remarks
1. Clemson	$1.0M	12 Months	M	Equipment needs to be relocated from SRS.
2. Catholic University	$1.2M	14 Months	L	10 kg single chamber
3. West Valley	$N/A	N/A Months	H	Too large
4. PNNL	$0.9M	12 Months	H	Not yet built
5. Commercial Facility	$N/A	N/A Months	H	None identified

Note: Costs here include estimated costs of testing (see Appendix B), as well as any associated construction costs (based on professional judgment).

Shading indicates most viable option.

Option B-1, Clemson University. Clemson University has two mini-melters; one is supplied by Envitco and the other is supplied by Stir Melter. Each will be discussed separately:

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- The Envitco melter has a bath capacity of 60 pounds (lbs.) and is designed to operate at a nominal 20 pounds per hour (lbs/hr) or 480 pounds per day (lbs/d). The electrodes are molybdenum. Based on this information, the melter is not applicable for FEMP use because: (1) the electrodes are molybdenum which should not be used in direct contact with high-lead glass compositions; (2) the capacity of the melter is too large to get extended run times with the amount of K-65 residues available; and (3) the residence time in the melter is too short (three hours) to allow for the destruction of sulfates.

- The small Stir Melter at Clemson has a bath capacity of 5 kg (10 lbs) of glass and is designed to operate at a nominal 2 kg/hr, or 50 kg/d (5 lbs/hr or 120 lbs/d). Because of its unique design, the Stir Melter has the potential problem of the undissolved/ unreacted sulfates being discharged with the glass. The impeller in the melter creates a vortex, and therefore may draw the sulfates down into the glass melt. Sulfate deposits in the glass will increase greatly the possibilities of the glass failing TCLP tests. The melter and its impeller are of Inconel metal construction. Therefore, the generally accepted maximum operating temperature is approximately 1,050°C to limit fatigue failure of the impeller due to torque on the Inconel shaft. An operating temperature of 1,050°C is considered too low to effectively destroy and handle sulfates. Thus, the melter is not applicable for FEMP use because: (1) the sulfates can be drawn into the glass without being destroyed; (2) the operating temperature is too low; and (3) the melter is of unique design and has operating conditions that are not applicable for scale-up of a melter of conventional design. The risk of this option is high because of the noncompatibility cited above. Clemson has the technical expertise to perform the work, but currently does not have the equipment. Clemson could construct a mini-melter facility or move a mini-melter from the DOE Savannah River Site (SRS). An SRS mini-melter (20 kg/d) that would

be compatible with the waste and its process requirements is available to be moved to Clemson.

Option B-2, The Catholic University VSL. The Catholic University has three mini-melters. They are:

- A 10 - 20 kg/d high-temperature, three-chamber melter with molybdenum electrodes in side chambers (similar construction to the VITPP melter)
- A 10 kg/d mini-melter with Inconel plate electrodes
- A 100 kg/d melter with Inconel plate electrodes.

The two 10 kg/d melters are of the appropriate size to accommodate the limited amount of K-65 materials available. The 100 kg/d melter is too large. There is some concern that the current location of the three-chamber melter is acceptable for the higher radioactive levels of the K-65 wastes. The melter would have to be moved to a new location in the laboratory at The Catholic University that is set up for radioactive contamination control. It is estimated that it would cost approximately $500,000 to relocate this melter and require an estimated 7-month addition to the schedule. All the melters are of an Inconel plate-type electrode design or simulate this phenomena and are conducive to uniform heating in the bath. This helps provide data that are applicable to scale-up for the type of melter designs envisioned for the full-scale facility. The 10-kg Inconel melter best fits the FEMP needs of the three melters considered and possibly of the mini-melter options proposed, due to the uniform heating as a result of the plate-type design, resulting in higher allowable operation temperatures (1150°C). However, The Catholic University has not agreed to allow the use of the 10-kg/d single-chamber for running the K-65 tests. Therefore, immediate availability of this melter to the FEMP is uncertain. The risk with this option would be low, once permission is

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granted for use of the 10-kg/d single-chamber melter. The Catholic University is experienced in this type of work.

Option B-3, West Valley Pilot Melter. West Valley's Scaled Vitrification System (SVS-II) is not likely to be cost or schedule effective. The mini-melter at West Valley is currently a non-radioactive testing system. Isolation and radon controls would be required to test K-65 materials.

The West Valley test melter is about 12 inches (in.) wide by 24 in. long (0.2 square meters [m^2]) surface area. This melter was used for testing redox control, melting rates, etc., for their production plant and its start-up. The melter currently is not operating but could be used for non-radioactive testing fairly quickly. This system could be upgraded conceptually to Phase II operation, but it would require licensing and the construction of administrative and physical barriers. This melter size is too large for FEMP's mini-melter run needs and too small for the FEMP's pilot-scale run needs.

The risk of this option is high because West Valley's melters are not immediately available for radioactive runs and because the pilot melter is too large for the mini-melter run.

Option B-4, PNNL. PNNL proposes installing a research-scale melter (6-in. diameter melter with a nominal processing rate of 1 kg/hr) in a radioactive facility. This melter would be of the appropriate size and design; however, it is not immediately available. Cost and schedule uncertainties include the off-gas system, facility modifications, and permits required at the PNNL location. The assembly and installation of the mini-melter is estimated at $200,000. Actual melter operation is estimated to take one month. However, it is estimated to take approximately one year to complete, starting in June 1997.

The risk of this option is high because: (1) the facility does not exist; and (2) the facility needs to be designed, built, and tested before mini-melter runs can be performed.

Option B-5, Commercial Facilities. No commercial vendors (other than those mentioned already) that have mini-melters that can be operated with radioactive wastes are known by the committee.

4.2.3 Option B - Mini-Melter Scope of Work

The primary purpose of mini-melter testing is to determine radioactive glass properties and off-gas composition utilizing actual silo residues. The mini-melter shall be operated with surrogates before running the actual K-65 residues. An initial information need is to gain a better understanding of the redox impact on the glass chemistry of the surrogates. The impact of different stoichiometric amounts of urea compared to the sulfur and nitrate content in the radioactive waste should be tested in the mini-melter. The urea concentration shall be increased until the lead in the glass precipitates as lead sulfide or lead metal. The redox of the glass will be determined at these concentrations of urea, and a lead and sulfate mass balance will be performed through sampling and analysis of the glass in the melter. A "snapshot" of all processing parameters for this condition shall be measured, or samples of appropriate streams taken for analysis. These streams include the feed, off-gas, and glass.

It is estimated that three mini-melter runs will be needed. To conserve the limited amount of K-65 residue, the mini-melter shall be started with surrogates that have been validated by analyzing the actual K-65 wastes. After the mini-melter has reached steady state with the surrogate and after the operators are confident operating the mini-melter, the first mini-melter run (with actual K-65 residues) shall be performed, using

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the optimal recipe as determined by the crucible melts. The remaining two runs shall be run with the actual residues that have been spiked with a high concentration of the components of concern (e.g., lead, barium, and sulfate). The Operable Unit 4 Feasibility Study report gives the expected high ranges for these components.

The off-gas shall be analyzed for the following (at a minimum) during the melter runs:

- Radon concentration
- NO_x concentration
- SO_x concentration
- Solids carryover -- This can be determined by: (1) analyzing the off-gas equipment liquid streams for residuals; (2) cleaning the major piping (especially the exhaust pipe between the melter and quencher) for solids buildup; and (3) examining the off-gas filters after each run.

Also, the melter (especially the melter bottom) should be investigated for lead or lead sulfide.

The radioactive aspects of the process shall be determined during steady-state operation. For example, the dose rate of slurry and glass shall be determined in the laboratory and the contact dose equipment faces measured. Radon in-growth as a function of time for the glass product is important in determination of the complexity of the product-handling design. This information is valuable for calculating expected full-scale facility dose rates.

The general estimated cost for the mini-melter runs assumes three radioactive runs with some preliminary run-time with surrogates. The details of the estimated cost are provided in Appendix B and should be considered rough order of magnitude or budgetary only.

The estimated schedule varies by facility option and is estimated to take approximately four months to prepare the equipment and facility for the runs, and to actually perform the work. The final draft report would be issued two months thereafter to FDF, for a total schedule of six months.

4.3 Option C - Pilot-Scale Melter

This section provides the rationale of the pilot-scale melter tests, discusses and evaluates nine sources with potential to perform the tests identified in the scope of work.

4.3.1 Overview

Testing for the plant's scale-up should be conducted in a test system that is typical of the system to be used in the plant design. The design reflected by the melters discussed in the following text is fairly diverse. Clemson's Envitco design and Vectra design employ designs that will perform differently from the PNNL and The Catholic University (VSL) designs. The Envitco design uses end-firing between the electrodes. This causes a high-temperature zone between the tips and can be used to achieve higher specific throughput with a smaller melter. This can also cause reboil foaming or the release of oxygen from multivalent cations, such as iron. This could frustrate pilot testing until suitable reductants are found that can pre-reduce the waste. Although the Vectra design is conceptually quite similar to the PNNL system, the Vectra design approach requires higher power input; thus, a similar reboil problem is also likely for the Vectra design but potentially not as severe as with the Envitco design. The PNNL approach also could be challenged by reboil but to a lesser extent than the Envitco and Vectra designs. The design least likely to result in reboil is the plate electrode design of VSL; however, reboil would still be possible with the VSL design.

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Some vendors propose the replacement of molybdenum electrodes so the melter can be used with the lead K-65 slurries/glasses. However, these melters are prone to form hot spots at the electrodes, as indicated by the reboil potential discussion. This implies that the Inconel replacement could get hotter than the desired bath melt temperature of 1,150°C and would result in melting the electrodes. Therefore, such an option is not recommended. This leaves the VSL 1 metric ton per day (MT/d), or the PNNL 2 MT/d as technically feasible options.

4.3.2 Evaluation of Pilot-Scale Sources

Nine sources were evaluated for their capability to perform the pilot-scale tests with surrogate materials as described in the scope of work. The evaluation results are provided in Table 4-5.

Table 4-5
Evaluation of Pilot-Scale Sources

Location	Cost	Schedule	Risk	Remarks
1. VITPP Single Chamber	$5.0M	7 + Months	H	Unsuitable materials of construction
2. VITPP Three Chambers	$5.0M	7 + Months	H	Unsuitable materials of construction
3. Catholic University	$0.7M	2-4 Months	L	Single chamber - Inconel
4. TVS - Oak Ridge	$N/A	N/A Months	H	Cannot handle lead
5. PNNL	$0.8M	4 Months	M	Design concerns
6. Vectra, Others	$1.0M	4-6 Months	H	Molybdenum electrodes
7. SRS	$1.0M	6 Months	H	Sulfates
8. MAWS Facility	$N/A	N/A Months	H	Not available
9. M-Area	$N/A	N/A Months	N/A	Gem production only

Shading indicates most viable option(s).

Option C-1, VITPP at Current Capacity, using a Single-Chamber Melter. One option is to use the existing VITPP melter with some modifications. Because of the undesirable molybdenum electrode material, the electrodes should be exchanged with Inconel 690 rod electrodes or plates to avoid reduction of lead compositions to metallic lead or lead sulfide. The E-bricks should be removed before start-up. Additionally, due to extreme refractory corrosion of material in the melter plenum and subpavers, the VITPP melter will need to be rebricked before going back in service. Furthermore, a complete replacement of ceramic (insulation) material, thermocouples, feed pipe, heater, electrodes, etc., will also be needed.

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Cost: The cost for replacing this material is approximately $512,000. However, operations and readiness preparation could add as much as $4,500,000.

Schedule: Seven months or more

Risk: High. Replacing ceramic material in a used melter has never been done before.

Option C-2, VITPP at Current Capacity, using Three Chambers. Another option is to re-brick the VITPP melter and place the melter back in service as designed. As in Option C-1, this will include a complete exchange of electrode material, all refractory, and other ceramic (insulation) material. Ancillary equipment (i.e., thermocouples, feed pipe, heater, electrodes, etc.) will also need to be replaced.

Cost: The total repair cost for this melter work was estimated at $512,000. Again, operations and readiness preparation could add as much as $4,500,000.

Schedule: Seven months or more

Risk: High, because of the unsuitable three-chamber design (with molybdenum electrodes) for vitrifying Silo 1 and Silo 2 waste.

Option C-3, The Catholic University ~1 MT/d Capacity. The Catholic University 1 MT/d "HAZMAT" melter is located at the VSL facilities in Washington, D.C. The melter is of rectangular construction and has four (two pair) Inconel 690 plate electrodes with a maximum operating temperature of 1,150°C. The electrodes are mounted on two opposing walls. The bottom two opposing electrodes fire together, as do the top two opposing electrodes. This arrangement allows more uniform current distribution and resultant heat distribution in the molten glass bath, than all the other melter options except the Fernald VITPP melter. This electrode configuration also limits reboil in the melter bath. The melter's bath surface is 1.14 square meters (m^2). The glass discharge is taken from the bottom of the melter and then flows over a weir. Discharge

can be air-lift assisted/controlled. The melter has multiple air bubblers to increase agitation.

Cost: $675,000

Schedule: The VSL melter is currently in warm standby and can be put into operation within approximately 30 days. The estimated duration of the melter operation is 14 days. The VSL has commitments for other customers, so specific timing and availability are not known. With adequate lead time, The Catholic University should be able to accommodate FDF's schedule ± 30 days.

Risk: Low. The melter is of the right size, configuration, and materials of construction to perform the K-65 low-temperature surrogate production runs. Information gained from this melter would be applicable for scale-up to a full-scale facility. The Catholic University is experienced in this type of work. They have used this melter for performing High-Level Waste (HLW), Hanford Low-Activity Waste (LAW), asbestos, medical non-radioactive, and hazardous materials studies.

Option C-4, TVS with ~3 MT/d Capacity. The Transportable Vitrification System (TVS) in Oak Ridge, Tennessee, is a large-scale, fully integrated vitrification system designed for treatment of low-level mixed wastes in the form of sludges, soils incinerator ash, and other waste streams. The unit is designed to be transportable and easily decontaminated. The melter is divided into three chambers. The largest is the main, central processing chamber which has an octagonal shape. This chamber contains the primary electrodes (molybdenum). Glass is drawn through a refractory lined "throat" into the glass drain chamber. Glass drain is controlled by moving a submerged spindle in the drain valve at the bottom of the glass drain chamber. The melter was designed for ~ 3 MT/d glass throughput.

Cost: Not available

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Schedule: Because of a glass leak, the melter drained through a joint between the ceramic refractory blocks in November 1996. Melter will be rebuilt. New start-up date unknown.

Risk: High. This melter is designed for consumable molybdenum electrodes. Because of the construction principle, the molybdenum electrodes cannot be revised to Inconel electrodes.

Option C-5, PNNL - HTM 2 m^2. This melter has a capacity of ~ 2 ton/day; 1,150°C+ glass melting, off-gas system, and sampling are available. The melter is currently configured with four 7-in. diameter, top-entering Inconel 690 electrodes, immersed 14 inches into the melt, and suitable for 1,150°C testing. Higher temperatures (1,250-1,500°C) could be achieved by using consumable, top-fed molybdenum or graphite top entering electrodes. Side conductive E-blocks also may be available for high-temperature operation, if desired. The PNNL off-gas system and sampling is available to the FEMP. The HTM melter (also known as the pilot-scale high-temperature melter) is in warm standby and can be operational within ~30 days. The HTM was constructed for support of the Hanford HLW testing for higher waste loading and reduced disposal volume. Since Hanford's privatization initiative was awarded in fiscal year (FY)96, the mission for this melter has been significantly reduced.

Cost: $655,000, including one week of mini-melter pre-testing, or $755,000 with melter draining and examination of corrosion plus restart.

Schedule: ~ 60 days

Risk: Low to moderate. The melter was designed primarily for high-temperature operation. However, the testing completed to date has only been with Inconel 690 electrodes. Over 300 testing hours using simulated HLW slurries have been completed in this arrangement. Although the electrode is totally immersed in the glass, control cooling in the sleeve at and above the glass levels removes heat that can maintain a cooler electrode. Additionally, due to the electrode configuration and low temperature operations, a sulfate layer is probable that could result in an electrical short.

Option C-6, Commercial Facilities: Vectra. This transportable melter is in cold shut down at Richland, Washington. The Vectra melter is currently housed in a three-trailer arrangement using two support trailers and the contiguous vitrification/off-gas trailer. A computer-control trailer is also part of the system. For a 1,350 - 1,500°C glass-melting temperature, it uses consumable, fed molybdenum or graphite top-entering electrodes. Inconel 690 rods could be used instead of the molybdenum or graphite to simplify testing at 1,150°C without electrode feeding and sheathing for oxidation resistance. The melter has only a bottom drain for melt discharge.

The system was designed for processing combustible dry active waste, ion exchange resins, and steam generator sludge, so the unit has a thermal oxidizer. Shielding was part of the design, so wastes up to one to five rem presumably could be vitrified. Its primary non radioactive testing was completed while melting Hanford LAW (~10 tons).

Cost:	$550,000 to $1,000,000 includes mobilization and setup at an unspecified location on the east coast (Fernald, Portsmouth, in Columbia, SC.)
Schedule:	~ 90 to 100 days until testing begins
Risk:	High. Apparently this melter was designed for consumable electrodes, such as molybdenum, etc. Because of the sloped arrangement of the electrodes, the resistivity between the electrodes is different. Thus, the power release (glass temperature) in the molten glass is different. The closer the electrodes, the higher the power release..

Option C-7, SRS Stir Melter. The SRS Stir Melter is designed to operate at a nominal 2.5 MT/d. Because of its unique design and because the impeller in the melter creates a vortex that can draw the sulfates down into the glass melt, the Stir Melter has the potential problem of undissolved/unreacted sulfates discharging with the glass. Sulfate deposits in the glass will increase greatly the possibility of the glass failing TCLP. The melter and its impeller are of Inconel metal construction. Therefore, the generally

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accepted maximum operating temperature is approximately 1,050°C to limit fatigue failure of the impeller. An operating temperature of 1,050°C is considered too low to effectively destroy and handle sulfates. Based on this information, the melter is not applicable for FEMP use because: (1) the sulfates can be drawn into the glass without being destroyed; (2) the operating temperature is too low; and (3) the melter is of unique design and has operating conditions that are not applicable for scale-up to a melter of conventional design.

Cost:	Set-up is $ 700,000. Operating cost per month of running is $150,000 and includes feed, and crew; non-routine data collection is extra.
Schedule:	Set up in three months.
Risk:	High. In addition to the reasons listed above, the low-temperature operations could result in a sulfate layer that would cause an electrical short due to the electrode configuration.

Option C-8, MAWS Facility. The Minimum Additive Waste Stabilization (MAWS) facility is located at the FEMP and has a pilot-sized melter with Inconel plate electrodes capable of processing 300 to 1,000 kg/d. The melter is too large for mini-melter runs, but would be useful for pilot-sized data for scale-up to a full-scale facility. Unfortunately, some parts of the off-gas and feed systems have been dismantled, and the facility is located in Plant 9, which is scheduled for demolition. Also, the melter is scheduled for relocation to Reactive Metals, Incorporated (RMI) in northern Ohio. The melter has processed mildly contaminated wastes from the FEMP's Waste Pit 5. Therefore, the glass processed (at least the first few batches) may have to be treated and disposed as radioactive or mixed waste. The schedule to start-up and run the required tests are estimated to take six months to one year. The risk of this option is medium because parts of the supporting system are dismantled and the building housing the facility is scheduled for decommissioning. In addition, operation of the facility would require similar administrative procedures as anticipated for the restart of the VITPP. This

translates into additional cost and schedule considerations for the process of the ORR and preparation of operating procedures, training, etc. These costs are anticipated for the pilot plant to be as high as $4,500,000. The major obstacles with the start-up of this facility are related to support systems, such as the off-gas feed system. DOE-FEMP and FDF remediation priorities would need to change in order to reactivate the MAWS equipment in Plant 9.

Option C-9, M-Area (for Gem Production Data only). The SRS M-Area gem-making machines are similar to the VITPP gem-making machine. These machines are connected to a 5-MT/d melter. Making glass gems is not glass chemistry dependent. The most important parameter in making gems is the viscosity of the glass. As long as the viscosity of the glass is similar over a given temperature range, empirical data could be acquired from the M-Area gem machine production.

4.3.3 Option C - Pilot-Scale Scope of Work

The pilot-scale melter tests would be utilized to determine the specific melting rate for Silos 1 and 2 material, and for Silos 1 and 2 material plus bentonite used in sizing and design of the plant melter (6 MT/day). The feasibility of vitrifying surrogate Silo 1 and Silo 2 material must be demonstrated at lower temperatures (1,150°C) at about 1 MT glass throughput per day. For the purpose of this pilot-plant testing, assume the specific melting rate is a function of waste glass compositions, degree of agitation, and operating temperature.

The melter maximum glass throughput has to be evaluated under different conditions. Lower glass viscosity possibly can have an impact at the glass throughput (hydrodynamical influence) and at the gem-making machine.

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Validation of the known "rule of thumb" ~ 40kg glass throughput / (m^2/h) must be performed. Definition of the impact on the specific glass throughput with variable solid/liquid relationships in the feed under steady state conditions needs to be developed.

The impact on vitrifying higher quantities of bentonite with Silo 1 and Silo 2 material should be tested in separate melter runs. In steady state operation, a throughput of 1 MT/d has to be demonstrated.

4.4 Option D - Full-Scale Melter Testing with ~6 MT/Day Capacity

The primary scope of full-scale melter testing is confirmation of the scale-up and the opportunity to gain operating experience. This, in itself, dictates that the melter and facility should be prototypical. Operation would be with surrogate material, with eventual changeover to actual K-65 materials. The information gained in these operations would be used to refine the melter specification such that the lessons learned can be incorporated into the ultimate final melter design. Two sources were evaluated.

- **Option D-1, Module #1 (~6 MT/day) of Full-Scale Facility.** Full-scale testing could be performed as part of the final remediation program. The current concept is to use multiple trains of melters in the full-scale facility. Feed preparation and final off-gas treatment would be common (along with utilities, etc.), while each individual melter would be supported by its own feed system and initial off-gas treatment. This path would assume that sufficient information is developed during the smaller-scale testing to support the design of the full-scale melter. The modular approach would require the design of the entire facility, considering staged construction, including all the trains of melters. The construction would be limited to only those items that would be required to support a single-melter train running surrogate materials. The

balance of the plant would be constructed after the successful completion of the melter testing. This approach would allow minimum investment in the final full-scale facility prior to final confirmation of the melter design. Appropriate planning would allow completion of the remediation facility in a short period of time while concurrent melter procurement activities take place. Full-scale testing would allow the incorporation of lessons learned into the subsequent trains without consideration of the scale-up issue.

- **Option D-2, VITPP with Upgraded Capacity.** Upgrading the VITPP and repair or replacing of the existing melter could be accomplished to continue the data development process, but there are some limitations. The current melter design may not be appropriate for the K-65 material, as evidenced by the recent melter incident. It appears that potentially a single-chamber, low-temperature melter may be desirable for the full-scale facility. Repair of the existing melter would, therefore, provide little confirmation of the ultimate design. Replacement of the melter in the VITPP would have some additional restrictions that would not be required if constraints of the existing design were removed. Many of these restrictions, such as physical space, off-gas configuration, and final remediation facility location of ancillary equipment, could result in a melter that may not be optimum. It appears that current consideration of data needs and the full- or large-scale facility primarily reflect the concerns of scale-up. Scale-up will remain a concern until a full-scale melter has been developed and tested. The number of intermediate steps taken between pilot and final scale are difficult to estimate and, while they may reduce the risk, they elevate costs.

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5.0 RECOMMENDED PATH FORWARD

This section provides eight specific recommendations for the path forward for the vitrification program at the FEMP. These recommendations are based on the analysis provided in this report and on the conclusion stated in Section 5.1.

5.1 Basis of Recommendations

The VITPP's unique three-chamber design was envisioned to address the following two specific design considerations as it applies to waste glass chemistry:

- The high-sulfate concentration in the Silo 3 residues requires high temperatures to destroy sulfates, thus improving leachability performance in the final waste.
- High lead, which requires an oxidizing environment to avoid lead reducing out and forming a metallic phase, would eventually cause electrical shorting of the melter.

Theoretically, the unique design was well suited for the glass chemistry that resulted from combining Silos 1, 2, and 3 material.

The primary design consideration was to utilize molybdenum electrodes for the high-temperature service, in an isolated electrode chamber (E-chamber) containing benign glass. In operations, the separation of the E-chamber from the corrosive, high-lead waste is impractical. When the waste leaks into the E-chamber, the electrodes experience accelerated corrosion and cause lead to form a metallic phase in the reducing environment required to protect the molybdenum. Therefore, the unique design for the glass chemistry provides equally unique problems that potentially can be catastrophic in their impact on the melter materials of construction.

A more practical approach is to focus on glass chemistry in order to allow usage of commercially available, proven techniques and materials of construction. This approach can be accomplished best by eliminating the high-sulfate Silo 3 waste from the vitrification program, and a waste loading target of 60 percent, and ensuring an oxidizing environment with close redox monitoring, as well as a means of dealing with metallic lead when it occurs.

5.2 Recommendations

The Data Analysis and Path Forward Team has developed a path forward for the vitrification program at the FEMP that is based on the recommendation that the VITPP melter does not restart (Recommendation 1). Once this premise is accepted, the next five recommendations (Recommendation 2 through 6) can be performed concurrently, but may have varying durations. These recommendations (Recommendation 2 through 6) collectively meet the data needs required for design, construction, and operations of Module 1 of the Remedial Facility (Recommendation 7) and subsequent module(s) (Recommendation 8). This path forward is provided in Figure 5-1.

RECOMMENDATION 1 - VITRIFICATION PILOT PLANT SAFE SHUTDOWN

Based on the fact that the three-chamber design, with molybdenum electrodes and multiple bottom penetrations, is unsuitable for vitrifying Silos 1 and 2 waste, it is recommended that the VITPP melter not be restarted, but be placed in a safe shutdown condition. This recommendation is further supported by the cost and schedule required for design, construction, and operational readiness involved in a VITPP restart.

In the event the VITPP melter is not restarted, the balance of data not yet supplied by VITPP operations (needed to support design of the remedial facility), will need to be gathered at

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alternative facilities (as discussed in Section 4.0 of this report). The primary vitrification program data need (as defined in Section 3.0 of this report), along with recommendations for acquisition of this data, are as follows:

RECOMMENDATION 2 - PERFORM WASTE RETRIEVAL DEMONSTRATION

Waste retrieval and feed system data should be supplied in the VITPP as part of the planned comprehensive waste retrieval demonstration following detailed rheology testing in an off-site laboratory. The waste retrieval demonstration is included in the Silos Project approved baseline.

RECOMMENDATION 3 - PERFORM MATERIALS OF CONSTRUCTION TESTING WITH K-65 RESIDUES

As a result of the melter incident, further materials of construction testing is needed for electrodes, refractory materials, and ancillary equipment. This testing typically would be performed at a qualified laboratory, using actual residues, in accordance with standard ASTM tests.

RECOMMENDATION 4 - PERFORM LABORATORY TESTING OF K-65 RESIDUES TO VALIDATE SURROGATES

The K-65 residues should be analyzed to gain a better understanding of the chemical composition of the waste. The analysis would be used to validate or modify the recipe for silo surrogate materials.

Additional physical data consisting of the rheology and hydraulic data of the slurry will be developed in the laboratory, with simulated conditions confirmed in the waste retrieval

demonstration. It is important to prove that the behavior of the actual material closely resembles the surrogate material, therefore enabling various tests to be performed, using differing variables to optimize the slurry-handling aspects of the facility.

RECOMMENDATION 5 - PERFORM PILOT PLANT SCALE-UP WITH SURROGATE AT A 1 MT/DAY OFF-SITE FACILITY

The primary Phase I Campaign 4 data gap left by the VITPP incident is the lack of empirical scale-up data (throughput versus melt pool surface area) as it applies to a low-temperature formulation. After evaluating data needs, the team determined that data is needed at a 1 MT/d or greater (surrogate glass) scale. Various facilities are available, including the 1 MT/d, Inconel electrode, melter at The Catholic University (VSL) or possibly the 2 MT/d, high- temperature melter at PNNL. Both VSL and PNNL could perform the low-temperature testing with a surrogate that has been validated against analysis of actual silo material. Both options could provide needed Phase I data at a lower cost and in a shorter time frame.

RECOMMENDATION 6 - PERFORM MINI-MELTER TESTING WITH K-65 RESIDUES

The primary data that were to be supplied by Phase II (radioactive VITPP tests) were the radioactive glass properties at a 1 MT/d scale, off-gas composition, and health physics data. After careful evaluation of data needs for full-scale remedial design, the team recommends that radioactive testing with actual silo residues can be performed in mini-melter tests and correlated against pilot-scale runs with surrogates that have been validated against K-65 elemental and compound analysis. Radioactive mini-melter testing would demonstrate low-temperature vitrification parameters, as well as provide off-gas composition and dose measurements that could be utilized to validate dose modeling. In summary, all Phase II data needs could be met at the mini-melter scale, excluding radioactive operation at the larger scale (1 MT/d), and prototypical off-gas and feed system testing.

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RECOMMENDATION 7 - MODULE 1, REMEDIAL FACILITY DESIGN, CONSTRUCT AND OPERATE

To reduce the risk of proceeding to full-scale design with limited pilot-scale testing, a sequential approach should be adopted. It is recommended that the first of several melter modules be used as a verification step, prior to hot operations, as confirmation of the melter design. The risk of scale-up is reduced in the fact that the plan will include extended comprehensive testing at full scale with surrogate material prior to operations with radioactive materials or procurement of the remaining full-scale modules. Operation of the first module will provide significant experience and serve as "proof of process testing," with the opportunity to incorporate "lessons learned" into the production modules.

RECOMMENDATION 8 - ADDITIONAL REMEDIAL MODULES DESIGN, CONSTRUCT, AND OPERATE

Following successful operation of the first module, larger-scale prototypical modules could be designed, constructed, and operated in parallel to the initial module, to fully stabilize the silo contents.

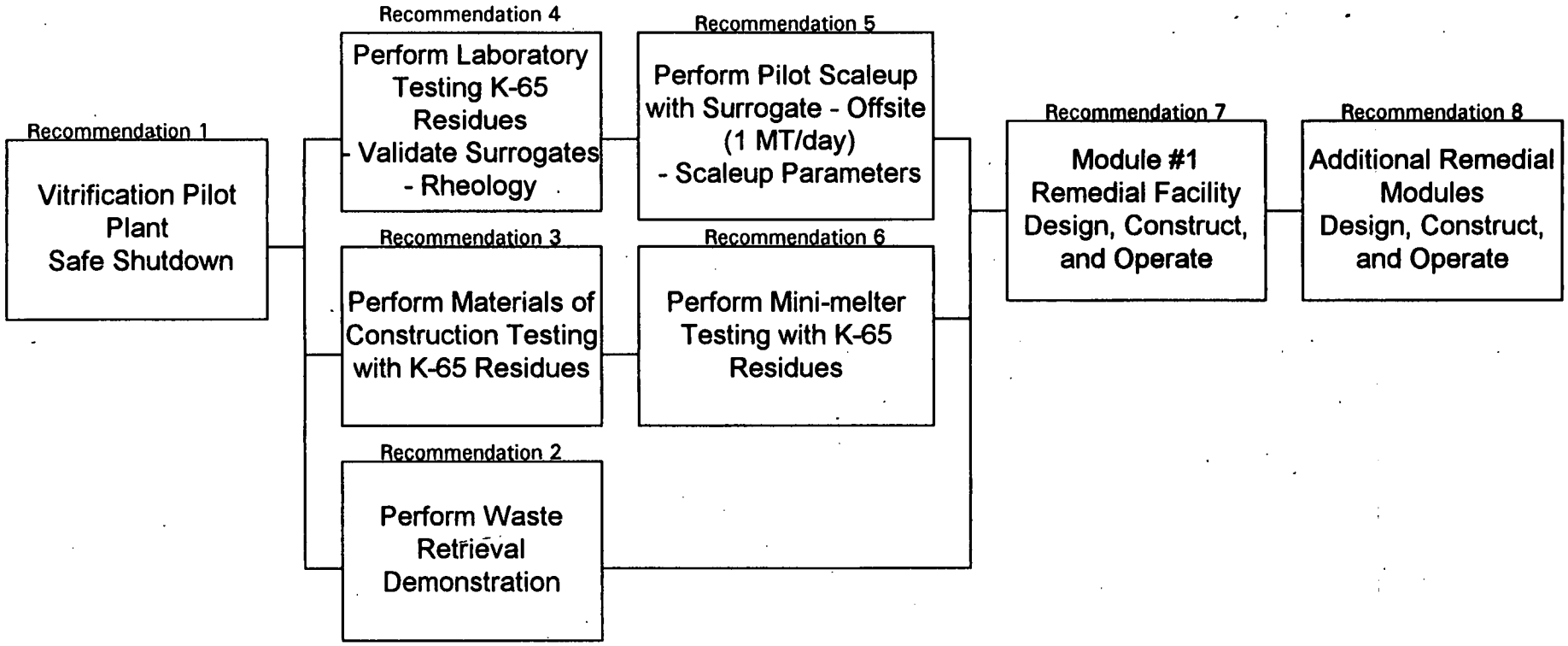
5.2 Path Forward

These recommendations (refer to Figure 5-1) provide the technical basis for the FEMP vitrification program path forward. Additionally, the team will work closely with the ongoing Silos Project Independent Review Team (IRT), which was formed in November 1996. The IRT's charter is to recommend the path forward for the overall remediation of the silo residues.

The December 26, 1996, melter incident (evaluated in depth by the three teams), and these recommendations will be considered by the DOE, FDF, regulators, and the public when making the overall path forward decision for remediating Operable Unit 4 wastes during spring of 1997.

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Figure 5-1
Data Analysis and Path Forward Team Recommendations



Notes:

1) The above activities have varying durations but can be performed in parallel as indicated.

2) Silo 3 residues are not considered in the recommendation.

3) The recommendation does not include Pilot Plant restart.

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APPENDIX A

ORIGINAL VITRIFICATION PILOT PLANT DATA MATRIX

Waste Removal (Silo Waste Retrieval)

Data ID #	Data Required	Silos Project Need (Reason for Data)	Need Level	VITPP Phase	P&ID No.	Existing Instrument #	Sample Point	Line or Equipment Number	Expected Value (per Exist. PFD)	Remarks
11-1	Water flow to residue removal	Validate Pilot Plant material balance	H	II	274	FI-022 FQR-022		RC-3"-A2-7117-ET	41.1 GPM	
11-2	Retrieved waste slurry composition, density, suspended solids content, size & density of largest particles	Material balance & rheology of plant feed slurry	H	II	269		V130, V131 (S-02)*	SL-3"-A-7000-ET	SG = 1.151	These data also required for design of Dewatering & Feed Preparation System
11-3	Retrieved waste slurry flow rate	Validate Pilot Plant material balance, rheology of plant feed slurry, and hydraulic characteristics	H	II	269	FI-020		SL-3"-A-7000-ET	50 GPM	These data also required for design of Dewatering & Feed Preparation System

*Sample point ID from Project Specific Plan for Vitrification Pilot Plant Phase I Process Sampling; typical for all tables.

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APPENDIX A ORIGINAL PILOT PLANT DATA MATRIX

Dewatering & Feed Preparation

Data ID #	Data Required	Silos Project Need (Reason for Data)	Need Level	VITPP Phase	P&ID No.	Existing Instrument #	Sample Point	Line or Equipment Number	Expected Value (per Exist. PFD)	Remarks
12-1	Flocculant addition rate	Thickener performance	H	I & II	269	FI-402		FL-1/2"-A-7400-ET	1.2 GPM	
12-2	Thickener bottoms slurry density, % suspended solids, size & density of largest particles	Thickener performance	H	I & II	269		V138, V139 (S-03)*	SL-1 1/2"-A-7003-ET	SG = 1.487	
12-3	Thickener overflow dissolved & suspended solids & particle size	Thickener performance, determine downstream treatment of overflow and material balance	H	I & II	269		V136, V137 (S-16)*	TO-6"-A-7100-ET		
12-4	Recycle water flow to Quench Tower	Thickener performance	H	I & II	273	FIC-070		RC-1 1/2"-A-7103-ET	40.1 GPM	Equal to water flow to thickener
12-5	Thickener overflow flow rate	Thickener performance	H	I & II	269					Obtain by material balance made from other instruments
12-6	Thickener bottoms flow rate	Thickener performance	H	I & II	270	FI-290		SL-1 1/2"-A-7003-ET	15.5 GPM	
12-7	Residue slurry pump discharge pressure	Rheology of thickener bottoms	H	I & II	269	PI-040, PI-041		SL-1 1/2"-A-7003-ET, SL-1 1/2"-A-7004-ET		
12-8	Melter feed composition slurry density, % solids size and density of largest particle	Material balance, glass quality correlations, and Rheology	H	I & II	271		V236, V237 (S-05)*	SL-1 1/2"-A-7010-ET, SL-1 1/2"-A-7009-ET	SG = 1.6	
12-9	Slurry Tank Pump discharge pressure	Hydraulics of melter feed loop	H	I & II	271	PI-343, PI-341		SL-1 1/2"-A-7010-ET, SL-1 1/2"-A-7009-ET		
12-10	Total Slurry Tank pump discharge rate/melter flow rate	Hydraulics of melter feed loop	H	I & II	271	FI-170		SL-1 1/2"-A-7010-ET	40 GPM	Local instrumentation acceptable

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Melter

Data ID #	Data Required	Silos Project Need (Reason for Data)	Need Level	VITPP Phase	P&ID No.	Existing Instrument #	Sample Point	Line or Equipment Number	Expected Value (per Exist. PFD)	Remarks
13-1	Electrode movement	Electrode corrosion rate	L	I&II	272		(S-09)*	5-FU-17		Record the time, date, insertion distance, and rotation angle for each electrode movement
13-2	Electrode power	Melter energy consumption	L	I&II		JIC-171B		5-FU-17	500 kW	
13-3	Glass Production Rate	Melter effectiveness	H	I&II			Gem Hopper		1 tonne/day	Record weight of each hopper and time since last hopper removal. Comment on abnormal events such as melter idling to determine residence time
13-4	Glass properties	Determine required residence time, temp., etc.	H	I&II	272		Gem Hopper (S-10)*			Use results of laboratory testing for TCLP, etc. This data needs to be correlated with residence time and glass temp data
13-5	Glass temperature	Melter effectiveness	H	I&II		TIC-171A		5-FU-17	1150-1350 C	

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APPENDIX A ORIGINAL PILOT PLANT DATA MATRIX

Melter Offgas

Data ID #	Data Required	Silos Project Need (Reason for Data)	Need Level	VITPP Phase	P&ID No.	Existing Instrument #	Sample Point	Line or Equipment Number	Expected Value (per Exist. PFD)	Remarks
14-1	Compressed air rate to melter	Quantify Melter inleakage to validate dynamic simulation	M	I & II		FI-001 to FI-008 plus 5 viewport rotameters		Duratek package	by Duratek	Verify that 5 viewport air purge line rotameters exist
14-2	Compressed air rate to Film Cooler	Quantify Melter inleakage to validate dynamic simulation	M	I & II		FI-020		Duratek package	50 SCFM	
14-3	Quench Tower gas cooling	Evaluate Quench Tower performance	L	I & II	273	TIR-071, TIR-072, TI-220, TIR-073		OG-4"-T-5605-I/P not defined RC-1 1/2"-A-7103-ET, RC-2"-A-7111-ET	600 F in. 97 F out	
14-4	Quench Tower overhead gas flow rate	Quantify Melter inleakage to validate dynamic simulation	M	I & II	273	Not yet installed (see Remarks)		Quench Tower overhead	130.3 SCFM	See VITPP SOW for A/E Services Phase IIB Modifications, Sect. 4.7.1, continuous FI/FIR to be installed
14-5	Film Cooler outlet temp.	Proper design of quench scrub & MOG system	M	I & II	272	Duratek T/C #20		OG-4"-T-5605-I/P	600 F	
14-6	Film Cooler pressure drop	Film Cooler design	M	I & II	272	PDI-172		OG-4"-T-5605-I/P	by Duratek	
14-7	Melter operating pressure	Develop Melter pressure control logic	M	I & II	272	PDI-171		OG-4"-T-5633	-4" WC	
14-8	Quench Tower overhead gas composition (Nox, Sox, CO2)	Design acid gas removal/abatement capabilities, and verify material balance	H	I & II	273	Not yet installed (see Remarks)		Quench Tower overhead	<1.9, 0.05, & <12.8 lb/hr	See VITPP SOW for A/E Services Phase IIB Modifications, Sect. 4.3, continuous AIR to be installed
14-9	Composition & pH of spent quench liquid	Decontamination efficiency of Quench Tower, and verify material balance	M	I & II	273		V279, V280 (S-06)*	RC-2"-A-7112-ET		

Melter Offgas (Continued)

Data ID #	Data Required	Silos Project Need (Reason for Data)	Need Level	VITPP Phase	P&ID No.	Existing Instrument #	Sample Point	Line or Equipment Number	Expected Value (per Exist. PFD)	Remarks
14-10	Composition & pH of Quench Tower recycle water supply, suspended solids content and size distribution dissolved solids content and composition, Phi; plus radon concentration, PhII	Decontamination efficiency of Quench Tower, verify material balance, track radon, and evaluate Waste Water Filter performance	H	I&II	274		V350, V351 (S-14)*	RC-2"-A-7103-ET		
14-11	Composition & pH of scrubber recirculating liquor	Quantify acid gas removal by scrubber	M	I&II	273		V283, V284 (S-07)*	WW-1"-A-7106-ET/PG-5		
14-12	Temperature of scrubber liquor	Quantify scrubber gas cooling	L	I&II	273	TI-222		5-SB-07/22		
14-13	Scrubber liquor supply pressure	Calculate total scrubber liquor flow	L	I&II	273	PI-220				
14-14	Packed column liquor supply rate	Evaluate packed column performance	L	I&II	273	FI-TBD			17 GPM	
14-15	Gas temp. to Desiccant Tower	Evaluate Desiccant Tower performance	L	I&II	273	TIR-420		OG-4"-A-5602	88 F	
14-16	Gas temp. to Desiccant Tower	Evaluate Desiccant Tower performance	L	I&II	273	TIR-210		OG-4"-A-5609	88 F	
14-17	Moisture content of Desiccant Tower overhead	Evaluate Desiccant Tower performance	L	I&II	273	TIR-210		OG-4"-A-5609	15% RH	
14-18	Radon content of gas to Carbon Bed	Quantify radon removal by Carbon Bed	H	II	315	MR-210	(S-18)*	OG-4"-A-5609		
14-19	Radon content of gas from Carbon Bed	Quantify radon removal by Carbon Bed	H	II	315	AIR-191	(S-19)*	OG-4"-A-5622		

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APPENDIX A ORIGINAL PILOT PLANT DATA MATRIX

Melter Offgas (Continued)

Data ID #	Data Required	Silos Project Need (Reason for Data)	Need Level	VITPP Phase	P&ID No.	Existing Instrument #	Sample Point	Line or Equipment Number	Expected Value (per Exist. PFD)	Remarks
14-20	Combined MOG and process vessel vent non-condensables flow rate	Evaluate radon removal & desiccant performance	H	I&II	315	FI-250		OG-4"-A-5626	238.7 SCFM	
14-21	Scrubber overhead gas composition	Measure No_x scrubbing performance	H	I&II	273	Not yet installed (see Remarks)		OG-4"-A-5608	<1.9 lb/hr NO_x	See VITPP SOW for A/E Services Phase IIB Modifications, Sect. 4.3, continuous AIR to be installed
14-22	Desiccant Tower operating pressure	Quantify volumetric gas flow through Desiccant Tower & Carbon Bed	M	II	273	PI-TBD		5-DH-21	-9" WC	
14-23	Carbon Bed pressure drop	Quantify volumetric gas flow through Desiccant Tower & Carbon Bed	M	II	315	PDR-190		OG-4"-A-5609 & OG-4"-A-5622		
14-24	Radon concentration at Exhaust Stack	Quantify total radon emissions	M	II	315	AIR-250		5-XS-20		
14-25	Gamma concentration at Exhaust Stack	Quantify total gamma emissions	M	II	315	RIOR-252		5-XS-20		
14-26	Exhaust Stack discharge rate	Quantify total radionuclide emissions	M.	II	315	FI-251		5-XS-20	238.7 - 588.7 SCFM	
14-27	Gas temperature to HEPA filters	Material balance & pressure profile through system	M	I&II	315	Not yet installed (see Remarks)		OG-4"-A-5622	88 F	See VITPP SOW for A/E Services Phase II B Modifications, Sect. 4.7.3, continuous TI to be installed
14-28	Gas temperature to Exhaust Fan	Material balance & pressure profile through system	M	I&II	315	Not yet installed (see Remarks)		OG-4"-5625	88 F	See VITPP SOW for A/E Services Phase II B Modifications, Sect. 4.7.3, continuous TI to be installed
14-29	No_x loading on Carbon Bed	Quantify No_x interaction with activated carbon	H	II	315		Not yet installed (see Remarks)	5-RN-19A 5-RN-19B		See VITPP SOW for A/E Services Phase II B Modifications, Sect. 4.1.3, carbon sampler to be installed

Melter Offgas (Continued)

Data ID #	Data Required	Silos Project Need (Reason for Data)	Need Level	VITPP Phase	P&ID No.	Existing Instrument #	Sample Point	Line or Equipment Number	Expected Value (per Exist. PFD)	Remarks
14-30	Pressure drop across film cooler outlet line	Provide indication of solids deposition/fouling inside line	H	I&II	272 273	PDI-173		OG-4"-T-5605-I/P		See DCN-037
14-31	Temperature of Sparge Tank liquid	Evaluate radon stripping efficiency	H	II	805	TI-390		5-AE-39	140 F	
14-32	Air flow rate to Sparge Tank	Evaluate radon stripping efficiency	H	II	805	FI-390		1A-1"-W-3021	50 SCFM	
14-33	Radon content of Sparge Tank liquid effluent	Evaluate radon stripping efficiency	H	II	805		V813, V814 (see Remarks)	WW-1 15-AE-391A-1:-W-3021/2"-A-7033-ET	Normally not present	Sample point ID and PSP TBD
14-34	Radon emission from Stack	Demonstrate compliance to Rn-222 negotiated site limit	H	II	315	AIR-250		5-XS-20		Continuous sampler
14-35	Emissions from Stack; Pb, Ba, CR for Ph I; radionuclides for Ph II	Demonstrate compliance with 40 CRF 61 and DOE Orders	H	I&II	315		PG-3 Isokinetic sampler (S-12)*	5-XS-20		
14-36	1 time Stack performance test of emissions of radionuclides, visible particulates, heavy metals TOC, NO_x, SO_x, Hcl, and fluorides	Demonstrate compliance with OAC 3745-17-07 and -17-11 for particulate emissions	H	II	315		PG-3 Isokinetic sampler (S012)*	5-XS-20		

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APPENDIX A ORIGINAL PILOT PLANT DATA MATRIX

Process Vessel Vent

Data ID #	Data Required	Silos Project Need (Reason for Data)	Need Level	VITPP Phase	P&ID No.	Existing Instrument #	Sample Point	Line or Equipment Number	Expected Value (per Exist. PFD)	Remarks
15-1	Operating pressure of Thickener, Slurry Tanks, Recycle Water Tank, Building Sump Tank and Sparge Tank	Monitor pressure profile through process vessel vent system, confirm negative gauge pressure in each tank, monitor effects of tank filling and pump out	M	I&II	269 270 274 805	Not yet installed (see Remarks)		VE-1 1/2"-A-5601 VE-1 1/2"-A-5602 VE-1 1/2"-A-5603 VE-1 1/2"-A-5600 VE-1 1/2"-A-5604 OG-2"-A-5630	-0.5" WC	See VITPP SOW for A/E Services Phase IIB Modifications, Sect. 4.7.4, to be installed

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Wastewater Treatment

Data ID #	Data Required	Silos Project Need (Reason for Data)	Need Level	VITPP Phase	P&ID No.	Existing Instrument #	Sample Point	Line or Equipment Number	Expected Value (per Exist. PFD)	Remarks
20.1	**This Data ID # has been deleted; Data ID # 14-10 is the same information**									
20.2	Waste Water Filter effluent, suspended solids content and size distribution	Evaluate Filter Performance	H	I & II	274		V312 V313 (S-01)*	WW-1'-A-2408-ET		
20.3	Pressure drop across Waste Water Filter	Evaluate Filter Performance	H	I & II	274	PDI-030, PDI-031		5-FL-03A & B		Record at the beginning and end of each campaign
20.4	Flow rate through Waste Water Filter	Evaluate Filter Performance	H	I & II	274	FIC-030		WW-1'-A-2408-ET	9 GPM	
20.5	Cumulative flow through Waste Water Filter between back flushes	Evaluate Filter Performance	H	I & II	274	FQR-030		WW-1'-A-2408-ET		
20.6	Pilot Plant Building Sump effluent	Pilot Plant building waste water characterization, Ph I; plus radionuclides, Ph II	H	I & II	274		V336 V337 (S-17)*	WW-1 ½'-A-7109-ET	Intermit	

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APPENDIX A ORIGINAL PILOT PLANT DATA MATRIX

Health Physics System

Data ID #	Data Required	Silos Project Need (Reason for Data)	Need Level	VITPP Phase	P&ID No.	Existing Instrument #	Sample Point	Line or Equipment Number	Expected Value (per Exist. PFD)	Remarks
62-1	Thickener Tank Shield Wall gamma dose rate survey	Modeling Correlation		II						Periodic HP survey
62-2	Furnace Room Shield Wall gamma dose rate survey	Modeling Correlation		II						Periodic HP survey
62-3	Carbon bed shield wall gamma dose rate survey	Modeling Correlation		II						Periodic HP survey
62-4	Slurry Tank Shield Wall gamma dose rate survey	Modeling Correlation		II						Periodic HP survey
62-5	Major Component Survey: Thickener Tank	Modeling Correlation		II						Periodic HP survey
62-6	Major Component Survey: Recycle Water Tank	Modeling Correlation		II						Periodic HP survey
62-7	Major Component Survey: Carbon Bed(s)	Modeling Correlation		II						Periodic HP survey
62-8	Major Component Survey: Off-gas HEPA filter housing	Modeling Correlation		II						Periodic HP survey
62-9	Major Component Survey: Building Sump Tank	Modeling Correlation		II						Periodic HP survey
62-10	Major Component Survey: Silo Material Transfer Piping	Modeling Correlation		II						Periodic HP survey

Health Physics System (Continued)

Data ID #	Data Required	Silos Project Need (Reason for Data)	Need Level	VITPP Phase	P&ID No.	Existing Instrument #	Sample Point	Line or Equipment Number	Expected Value (per Exist. PFD)	Remarks
62-11	Major Component Survey: Gem Hopper (over time)	Modeling Correlation		II						Periodic HP survey
62-12	Major Component Survey: Shipping Container	Modeling Correlation		II						Periodic HP survey
62-13	Major Component Survey: Quench Tower	Modeling Correlation		II						Periodic HP survey
62-14	Major Component Survey: Desiccant Tower	Modeling Correlation		II						Periodic HP survey
62-15	Major Component Survey: Slurry Tank	Modeling Correlation		II						Periodic HP survey
62-16	Silo Material Transfer Line:	Modeling Correlation		II						Periodic HP survey
62-17	Silo 3 Surge Bin	Modeling Correlation		II						Periodic HP survey
62-18	Silo 3 filter/receiver	Modeling Correlation		II						Periodic HP survey
62-19	Thickener area radon concentration	Radon release from Thickener		II						Periodic HP survey

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APPENDIX A ORIGINAL PILOT PLANT DATA MATRIX

Glass Product Packaging

Data ID #	Data Required	Silos Project Need (Reason for Data)	Need Level	VITPP Phase	P&ID No.	Existing Instrument #	Sample Point	Line or Equipment Number	Expected Value (per Exist. PFD)	Remarks
25-1	Gem temp as a function of time after discharge into hopper	Required staging time before gems can be loaded into concrete boxes	H	I			Hopper or drum surface			Record surface temperature of inner container using attached TE until temperature is below 100 F
25-2	Radiation field from glass as a function of time after discharge from melter	Design of shielding for material handling device and optimization of waste container	H	II			Hopper or drum surface			One reading per shift using a hand-held Geiger counter for 30 days
25-3	Monolith temp as a function of time after pouring	Required staging time before drums can be loaded into concrete boxes	H	I			Drum surface			Record surface temperature of monolith using portable TE until temperature is below 100 F
25-4	Radiation field at the surface of the concrete waste container	Verify the optimization of the waste container design to minimize shielding and weight of container, maximize payload, and minimize disposal costs	H	II			Waste container surface		50-80 mrem/hr	Use hand-held device. Monitor entire surface, record average and any high or low spikes. Monitor 60 days after creation of glass.

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Glass Product Handling

Data ID #	Data Required	Silos Project Need (Reason for Data)	Need Level	VITPP Phase	P&ID No.	Existing Instrument #	Sample Point	Line or Equipment Number	Expected Value (per Exist. PFD)	Remarks
24-1	Gob cutter wheel and roller temp as a function of time	Reliability/operability of the gem maker	H	I			Surface Temp	5-CU-24C 5-RO-24D	500 - 600 C	Use optical pyrometer or similar device
24-2	Outside surface temp of Gem Conveyer housing	Feasibility of locating equip near the conveyor	M	I			Surface Temp	5-CV-24E	200 F	Use of hand-held device is acceptable. Record after 24 hours of continuous operation
24-3	Size, shape, and weight of gems as a function of time	Reliability of gem maker	H	I			Product Conveyor			Observations record in Operator's log
24-4	Operating hours before failure for all equip in this system	To estimate mean time before failure for all equipment	M	I & II						

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APPENDIX B

COST ESTIMATES

Estimate for Mini-Melter Testing

Item	Amount	Each, $	Total, $
Post run with surrogate and redox testing	1	65,000	65,000
Cost per minimelter run			
Crucible melts to support run	3	15,000	45,000
Minimelter run	3	65,000	195,000
Analysis for run	3	12,000	36,000
Program Management	3	16,000	48,000
Subtotal			324,000
Science	25%	na	81,000
Reports	1	16000	16,000
Waste Disposal	15%	na	32,400
Overhead	50%	na	162,000
Total			680,400

Estimate for Laboratory Testing -- Crucible Melts

Item	Amount	Each, $	Total, $
Low Temperature Crucible Melts			
Residue Chemical Analysis	Lot	15,000	15,000
Crucible Melts -- surrogate	15	200	3,000
Crucible Melts -- radioactive	5	300	1,500
viscosity and conductance -- rad	5	750	3,750
VISCOSITY and conductance -- surrogate	15	200	3,000
ICP	20	200	4,000
Gamma spec	5	300	1,500
Liquidus	20	75	1,500
Analysis	10	250	2,500
Subtotal			35,750
Science	25%	na	8,938
Project Management	10%	na	3,575
Overhead	50%	na	17,875
Report			3,863
Total			70,000
Corrosion/Erosion Study			
Purchase and prepare sample cylinders	20	500	10,000
Perform Corrosion/Erosion Tests	20	1000	20,000
Subtotal			30,000
Science	25%	na	7,000
Project Management	10%	na	3,000
Overhead	50%	na	15,000
Report			5,000
Total			60,000

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Item	Amount	Each, $	Total, $
Redox Study			
Crucible Melts -- surrogate	10	200	2,000
Redox -- Fe2/Fe3	10	250	2,500
ICP	10	200	2,000
Analysis	10	250	2,500
Subtotal			9,000
Science	25%	na	2,250
Project Management	10%	na	900
Overhead	50%	na	4,500
Report			4,350
Total			30,000
Rheology			
Flow properties vs conc., temp., and shear	lot	20,000	20,000
pH vs. slurry temp. and water content	lot	1,000	1,000
Particle size distribution	lot	5,000	5,000
Settling curves	lot	5,000	5,000
Thickening vs. bentonite concentration	lot	15,000	15,000
Dewatering characteristics	lot	1,000	1,000
Suspended solids	lot	200	200
Density of slurries	lot	200	200
Subtotal			47,400
Science	25%	na	11,850
Project Management	10%	na	4,740
Overhead	50%	na	23,700
Report			32,310
Total			120,000
Grand Total			280,000

FLUOR DANIEL FERNALD
OP & I
ESTIMATING SERVICES

29 January 97

BASIS OF ESTIMATE

PROJECT DESCRIPTION: Restart Melter (Evaluation)
WBS NUMBER: 1.1.1.1.4.1.4.1
PROJECT LOCATION: VitPP-Fernald
PROJECT ENGINEER: Dennis Nixon
ESTIMATOR: G.D.Walters
ESTIMATE NUMBER: C4-97-01-02

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SUPPORTING DOCUMENTATION:

Verbal Scope	x	P & ID's		Work Plan	
Drawings	x	Equipment List	x	Site Walk	
Sketch	x	Specifications		Eng Mtg	x
Flow Diagrams		Site H & S Plan		Price Quotes	x

TYPE OF ESTIMATE:

Plan/Feasbl	x	Budg/Concpt		Title I Des	
Title II Des		Government		Independent	
Construction		Baseline			

BASIS OF ESTIMATE:

This conceptual rough order of magnitude (ROM) estimate is for the labor and material to repair the melter and resume Phase I Operations data analysis collections.

Using the Spare Parts Inventory list provided to CRU4 by Duratek Inc., and material costs provided by various vendors, the repairs to the melter were developed using estimated quantities by the Project Engineer.

The scope of work includes the following:

1. Replace & Purchase Lid and Discharge Heaters
2. Re-brick and Re-insulate Melter Walls, Roof, Discharge Trough and orifice, Riser Block
3. Rework Bubbler Path
4. Replace Thermocouples
5. Replace Film Cooler
6. Prepare Bottom Drain Container
7. Install New Moly Electrodes
8. Abandon (3) Bottom Drains and Prepare (1) Bottom Drain
9. Replace Feed Tube

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In addition, the Restart of Operations, Construction Management and Engineering are included based on actual past history developed and provided by Project Controls from the 4A141, 4A151, 4A121 and 4ACA3 cost accounts.

FLUOR DANIEL FERNALD
OP & I
ESTIMATING SERVICES

29 January 97

ESTIMATE ASSUMPTIONS

PROJECT DESCRIPTION: Restart Melter (Evaluation)
WBS NUMBER: 1.1.1.1.4.1.4.1
PROJECT LOCATION: VitPP-Fernald
PROJECT ENGINEER: Dennis Nixon
ESTIMATOR: G.D.Walters
ESTIMATE NUMBER: C4-97-01-02

DRAFT

EXECUTION:
Based on the schedule of activities developed by Project Controls
The estimate does not include overtime and/or premium time for the repairs to the melter performed by a subcontractor.

WAGE RATES:
Based on the FY97 Craft Composite Rates

ENGINEERING:
Fluor Daniel Fernald and A/E based on actual past history from 4ACA3

PRODUCTIVITY:
Based on the Site Specific Productivity Factor of 1.30%; Refer to Attachment Appendix "A"
It is assumed that all work is performed in a non-controlled area and does not require any PPE's.

ESCALATION:
Not Applicable

UNIT RATES:
Based on Construction Subcontractor Langdon for the installation and bricking of the melter.

G & A (HO EXPENSE):
Not Applicable

RISK BUDGET:
A cost element, based on a risk analysis calculated for this estimate to cover the statistical probability of a 50% chance of an overrun/underrun to the project.
The target estimate is the sum of the base estimate and risk budget. The target estimate is the basis for establishing the Performance Baseline. The risk budget for this estimate is 24% of the base estimate.

CONTINGENCY:
An amount budgeted to cover costs that may result from incomplete design, unforeseen and unpredictable conditions or uncertainties. The amount of the contingency will depend on the status of design, procurement, construction and the complexity and uncertainties of the component parts of the project. Contingency is not to be used to avoid making an accurate assessment of expected costs.
The contingency is calculated as the delta between a 50% chance of overrun and a 5% chance of overrun, based on a risk analysis. The contingency for this estimate is 17% of the base estimate.

FLUOR DANIEL FERNALD
OP & I
ESTIMATING SERVICES

29 January 97

ESTIMATE INCLUSIONS AND EXCLUSIONS

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PROJECT DESCRIPTION:	Restart Melter (Evaluation)
WBS NUMBER:	1.1.1.1.4.1.4.1
PROJECT LOCATION:	VitPP-Fernald
PROJECT ENGINEER:	Dennis Nixon
ESTIMATOR:	G.D.Walters
ESTIMATE NUMBER:	C4-97-01-02

INCLUSIONS:

Labor and Material for the Repair of the Melter
Replacement and Reworking of systems as per the scope of work
Resume Phase I Operations developed and provided by Project Controls
Construction Management developed and provided by Project Controls
Engineering FDF and A/E developed and provided by Project Controls

EXCLUSIONS:

Beyond Phase I Operations
Health Physics

PROJECT: Restart Melter
ESTIMATE NO.: C4-97-01-02
CLIENT: DOE
WBS NO.: 1.1.1.1.4.1.4.1

FLUOR DANIEL FERNALD
ESTIMATING SERVICES

DATE: 29-Jan-97
ESTIMATOR: G.D.Walters
LOCATION: FERNALD
TASK NO.: 4A11A

ESTIMATE SUMMARY SHEET

ITEM DESCRIPTION	M/H	RATE	LABOR $	S/C $	MAT'L $	EQUIPMENT $	TOTAL $
RESTART MELTER	970	$20.31	$19,700	$0	$369,100	$0	$388,800
DIRECT FIELD COSTS TOTAL	970	$20.31	$19,700	$0	$369,100	$0	$388,800
SUPERVISION - CONTRACTOR	165	$20.01	$3,300				$3,300
SMALL TOOLS & CONSUMABLES						$1,200	$1,200
S/C. EQUIPMENT RENTAL						$3,400	$3,400
TEMPORARY FACILITIES	29	$20.62	$600		$600		$1,200
TEMPORARY UTILITY HOOK-UP	19	$21.15	$400		$200		$600
JOB CLEAN-UP	44	$20.62	$900		$300		$1,200
SAFETY	19	$21.15	$400		$200		$600
HEALTH PHYSICS S/C							$0
CERCLA - 40 HRs/FTE			$3,200				$3,200
SITE ACCESS/JOB SPECIFIC TRAINING - 15 HRs/FTE			$1,800				$1,800
PAYROLL BURDENS & BENEFITS			$17,300				$17,300
OVERHEAD & PROFIT				$84,500			$84,500
BOND				$5,100			$5,100
INDIRECT FIELD COSTS TOTAL	275		$27,900	$89,600	$1,300	$4,600	$123,400
DIRECT & INDIRECT FIELD COSTS TOTAL	1,245	$38.22	$47,600	$89,600	$370,400	$4,600	$512,200
WASTE DISPOSITION - FDF (NTS Burial Costs Are Not Part of the Baseline)				NTS COSTS			$0
WASTE MANAGEMENT COSTS TOTAL			$0		$0	$0	$0
PHASE I OPERATIONS			$1,720,000	$150,000	$325,000		$2,195,000
CONSTRUCTION MGMT - FDF			$745,000				$745,000
FDF FIELD SUPPORT COSTS TOTAL			2,465,000	150,000	325,000	0	$2,940,000
ENGINEERING/DESIGN/INSPECTION			FDF $200,000	A/E $600,000			$800,000
ENGINEERING COSTS TOTAL							$800,000
TAXES TAX	SUBCONTRACT		$22,200	FDF	$19,500		$41,700
SUB-TOTAL (BASE ESTIMATE)							$4,293,900
RISK BUDGET 24.0%							$1,030,500
TARGET ESTIMATE (FY'97 DOLLARS)							$5,324,400

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ESTIMATE PERFORMED BY ESTIMATING SERVICES

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e Table97PLACOMP Estimator: G.D.Walters

FLUOR DANIEL FERNALD
Estimate Summary Report

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ject TitleRestart Melter - Rebrick

	Activity	Description	TakeOff Quantity	Unit	Total MH	Labor Hrs	Rate	Material Price	Labor Total	Sub/Other Total	Material Total	Equipment Total	TOTAL
TER	Heaters/Assemblies	Electrode Advancement Pieces	180.00	inch	.072	13	$20.27	2.75	260		500		$760
	Heaters/Assemblies	Install Discharge Heaters	10.00	ea	13.000	130	$20.79	800.00	2700		8000		$10,700
	Heaters/Assemblies	Install Film Cooler 14-FC-01	1.00	ea	104.000	104	$20.27	113,000.00	2110		113000		$115,110
	Heaters/Assemblies	Install Lid Heater	1.00	ea	13.000	13	$20.79	800.00	270		800		$1,070
	Heaters/Assemblies	Install Moly Electrodes	10.00	ea	1.300	13	$20.27	1,150.00	260		11500		$11,760
	Heaters/Assemblies	Patch & Plug Drain Assembly	1.00	ea	52.000	52	$20.27	1,500.00	1050		1500		$2,550
	Heaters/Assemblies	ReWork Bubbler Path	1.00	ea	26.000	26	$20.27	500.00	530		500		$1,030
	Heaters/Assemblies	Replace and Install Feed Tube	1.00	ea	5.200	5	$20.27	625.00	110		630		$730
	Heaters/Assemblies	Rework Bottom Drain Assembly	1.00	ea	104.000	104	$20.27	5,500.00	2110		5500		$7,610
	Heaters/Assemblies	Thermocouple/Thermowell Assbly	4.00	ea	6.500	26	$20.27	5,700.00	530		22800		$23,330
	TOTAL	Heaters/Assemblies				486			$9,930		$164,720		$174,650
TER	Refractory Brick	1" Fiberboard 24"x48" 1 ea	8.00	sf	.520	4	$19.84	40.00	80		320		$400
	Refractory Brick	2" Fiberboard 24"x48" 2 ea	16.00	sf	.520	8	$19.84	80.00	170		1280		$1,450
	Refractory Brick	HMS Cement	200.00	lbs	.026	5	$20.11	1.45	100		290		$390
	Refractory Brick	Install/Set Refractory Brick	71.90	cuft	4.520	325	$20.11		6540				$6,540
	Refractory Brick	K3	25.60	cuft		0		3,300.00			84480		$84,480
	Refractory Brick	K3 Modified	9.00	cuft		0		5,100.00			45900		$45,900
	Refractory Brick	MONOFRAX E	5.90	cuft		0		8,500.00			50150		$50,150
	Refractory Brick	MONOFRAX H	31.40	cuft		0		640.00			20100		$20,100
	Refractory Brick	Monofrax K Cement	250.00	lbs	.021	5	$20.11	2.15	100		540		$640
	Refractory Brick	RemoveDamagedRefractory Brick	1.00	ls	130.000	130	$20.11		2610				$2,610
	Refractory Brick	Zirmul 361 Mortar	600.00	lbs	.009	5	$20.11	2.25	100		1350		$1,450
	TOTAL	Refractory Brick				483			$9,710		$204,400		$214,110
	GRAND TOTAL:					969			$19,640		$369,120		$388,760

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APPENDIX "A"

SITE SPECIFIC PRODUCTIVITY MATRIX

EFFICIENCY / MULTIPLIER ANALYSIS

PROJECT: Restart Melter
ESTIMATE NO.: C4-97-01-02
CLIENT: DOE
WBS NO.: 1.1.1.1.4.1.4.1

DATE: 28-Jan-97
ESTIMATOR: G.D.Walters
LOCATION: FERNALD
TASK NO.: 4A11A

	PERCENT OF INFLUENCE ON CHART MANHOURS											
	40%	50%	60%	70%	80%	90%	100%	105%	110%		WT'D VALUE	PROD. RESULT
CRAFT SKILL (NOTE 1)	POOR			FAIR			STD	V.GOOD	EXCELLENT	90.0%	12.0%	0.108
CRAFT AVAIL.(NOTE 1)		POOR		FAIR			STD			90.0%	8.0%	0.072
CLIMATE (NOTE 2)	SEVERE	ICE/SNOW			RAIN		+40 TO +85			50.0%	20.0%	0.1
PLANT ELEVATION		OVER 10,000FT			5,000' TO 10,000 FT		UNDER 5,000 FT			100.0%	5.0%	0.05
WORK SPACE		MULTI SHIFT		200 SF	250 SF	300 SF	350 SF			100.0%	10.0%	0.1
WORK WEEK							4-10s / 5-8s			100.0%	15.0%	0.15
50 HOUR WORK WEEK		MULTI SHIFT		OVER 7 WEEKS	3 TO 7 WEEKS	UP TO 3 WEEKS						
60 HOUR WORK WEEK			OVER 7 WEEKS	3 TO 7 WEEKS	UP TO 3 WEEKS							
SHIFTWORK 2ND SHIFT 3RD SHIFT			3RD SHIFT		2ND SHIFT		OR ONE SHIFT ONLY			100.0% 100.0%	3.0% 5.0%	0.03 0.05
PROJECT SIZE					400M MH AND UP	300M TO 400M MH	200M TO 300M MH	200M MH OR LESS		105.0%	4.0%	0.042
PLANT TYPE				REVAMP ONLY	REVAMP & NEW	NEW IN EXIST PLT	GRASS ROOTS			50.0%	8.0%	0.04
AREA/UNION INFLUENCE	STRONG		MILD		SOME		NONE			30.0%	10.0%	0.03
											100.0%	77.2%

NOTES.....
1. TURNOVER HAS BEEN CONSIDERED
2. FOR EXTERIOR WORK ONLY

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EFFICIENCY (AS A % OFF CHART MANHOURS) 77.2%

MULTIPLIER - FOR NONCONTAMINATED AREA	[illegible]00
MULTIPLIER - FOR CONTAMINATED AREA WITH USE OF LEVEL MOD."D" PPEs	[illegible]2.40
MULTIPLIER - FOR CONTAMINATED AREA WITH USE OF LEVEL "C" PPEs	[illegible]
MULTIPLIER - FOR CONTAMINATED AREA WITH USE OF LEVEL "B" PPEs	[illegible]3.05

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APPENDIX C

DATA ANALYSIS AND PATH FORWARD TEAM BIOGRAPHIES AND SIGNATURE PAGES

Nina Akgündüz: Nina Akgündüz is currently with the DOE Fernald office as team leader of Operable Unit 4. Prior to DOE, she worked in ceramics industry where her experience ranges from research and development, to manufacturing of ceramic materials and plant operations.
Ms. Akgündüz has been working on environmental restoration projects, specifically in vitrification treatment technology, for more than six years. She has a bachelor of science degree in ceramic engineering from Rutgers University and is currently finishing her masters degree in environmental management from the University of Findlay.

Chris Chapman: Mr. Chapman has been directly involved in joule-heated waste glass melters for the past 25 years. He has led the design, construction, and operational testing of nine integrated vitrification systems that included feed, glass handling, and off-gas systems -- first in its initial development at DOE's Pacific Northwest Laboratory between 1972 and 1979 then as an independent consultant to the countries of Germany, Japan, and Canada. In 1982, Mr. Chapman led the process and remote equipment design, and eventually the testing and qualification program at DOE's West Valley Demonstration Project between 1982 and 1987. Since 1987, he has directed his focus toward adapting the DOE-developed waste vitrification technology to other waste streams including mixed, TRU, commercial, low-level waste (LLW), industrial hazardous, and non-hazardous solid wastes. In 1995, Mr. Chapman formed his own corporation to actively pursue commercialization of the technology to transform a diversity of wastes into glass and glass ceramic products.

Robert Frost: Mr. Frost has more than 10 years experience in environmental engineering, in both the private and governmental sectors. Past experience includes supervision of compliance and enforcement activities under RCRA for the State of Florida, hazardous waste permitting with the U.S. Environmental Protection Agency (Region IV), and water resources engineering with various consulting firms. His training includes a masters degree in environmental engineering from the University of Florida, and a bachelor of science degree in geology from Virginia Polytechnic Institute and State University (also known as Virginia Tech). He is a licensed Professional Engineer (PE #41907) registered in the State of Florida. He currently holds a position with Fluor Daniel Fernald, where he supports the CERCLA remediation projects using vitrification to stabilize radioactive waste.

Rod Gimpel: Rod Gimpel is Fluor Daniel Fernald's Testing and Data Manager for the Silos Project. He has five years experience in vitrification glass chemistry development, melter design, and process scale-up. He is a chemical engineer from the University of Idaho, with 21 years experience in environmental programs and project management, process development and design, and environmental safety analysis at Hanford, Richland, Washington; Idaho National Engineering Laboratory,

Idaho Falls, Idaho; and the Fernald Environmental Management Project, Cincinnati, Ohio. Previously, he was coordinator and project engineer for Fernald's CERCLA Operable Unit 1 (Waste Pit Area) Remedial Action. He organized and started development programs, including the Minimum Additive Waste Stabilization (MAWS) vitrification development program. He made and directed the life-cycle cost estimates for Operable Unit 1 and the MAWS programs. These development programs incorporated vitrification, soil washing, grouting/solidification, and water treatment technologies to turn wastes into glass, cement, clean soil, and clean water. Since these processes have not been used for large remedial projects (i.e., 100 ton/day), these processes are going through laboratory, bench, and pilot-scale development to determine scale-up parameters. He is presently serving on a peer review team on the waste pit solidification effort at Weldon Spring near Saint Louis, Missouri. He has published many papers on waste treatment, process scale-up, and vitrification.

Doug Maynor: Doug Maynor joined the DOE Ohio Field Office (OH) as a technology support engineer in 1994. Prior to joining DOE in 1993, Doug worked in the chemical processing industry in engineering and operations positions. His work experience with large multi-national companies and a small entrepreneurial company included service as start-up manager of three "grass-roots" plants, one in Brazil. He received a bachelor of science degree in chemical engineering from Virginia Polytechnic Institute in 1966 and a masters degree in business administration from Western New England College in 1969.

Dennis Nixon: Dennis A. Nixon is currently performing the role of Pilot Plant Program Manager for Fluor Daniel Fernald. Mr. Nixon has been involved in the environmental field more than 13 years, most of which includes experience at the FEMP. He has a bachelor of science degree in construction management and an associate degree in civil engineering.

Don Paine: As Silos Project Manager, Dr. Paine is responsible for the programmatic and functional operation for the environmental clean-up and restoration of Operable Unit 4. He has more than 20 years of ES&H and project management experience, including emergency services management, in all aspects of the nuclear fuel cycle applicable to the FEMP site. This includes fuel manufacturing, safe shutdown, waste management, decontamination and decommissioning (D&D) of contaminated facilities, and remediation of hazardous and mixed waste sites at DOE and NRC licensed facilities. Dr. Paine has successfully managed ES&H programs at an NRC Category I, Licensed Special Nuclear Fuel Fabrication Facility. He has a bachelor of science degree in mathematics and biology, a master of science degree in health physics, and a Ph.D. in radiology and radiation biology.

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John Plodinec: Dr. Plodinec is an internationally recognized expert in waste management and glass science. He has led glass development at the Savannah River Site (SRS) almost from the beginning of the Defense Waste Processing Facility (DWPF) project. He has made important contributions which range from waste characterization (co-inventor of a sludge sampler currently in use at SRS) to glass durability modeling (initial developer of free energy of hydration model). He helped to design, and then operated, the first four glass melters at SRS. Many of the features on the current DWPF melter are a direct result of his work (use of cameras for viewing, plenum heaters to improve melter throughput, and prevention of direct current to extend melter life).

He also was DOE's technical lead on the team which persuaded the EPA to declare vitrification to be the Best Demonstrated Available Technology for high-level waste and for heavy metals. He was DOE EM's primary author for the Waste Acceptance Product Specifications, which govern all of the high-level waste glass products produced in the United States. In 1995, he served on the Hanford Low-level Waste Melter Technical Advisory Panel. In late 1995, he led a team including personnel form DOE's Tanks, Mixed Waste, and Landfills Stabilization Focus Areas which prepared a database of melters available to DOE for testing. He currently is the Technical Integration Manager for the Immobilization Functional Area for DOE's Tanks Focus Area. He has a bachelor of science in chemistry from Franklin and Marshall College, and a Ph.D. in physical chemistry from the University of Florida.

Harry Robertson: He has a bachelor of science degree in chemical engineering from the University of Cape Town and a masters degree in business administration from California State University at Long Beach. Mr. Robertson has over 32 years of experience in the management and engineering environment of a variety of government and industrial projects, including several projects for the DOE.

Currently, he is Department Manager of the Environmental Engineering Department for Fluor Daniel Fernald.

Other assignments during his 28 years at Fluor Daniel have included: area manager for the Vitrification Building, the major process building on the Hanford Waste Vitrification Plant. This project was to design a plant to immobilize high-level nuclear waste in glass for permanent long-term storage.

John Smets: He has a bachelor of science mechanical engineering/West Coast University Business Management Certificate/University of California. Mr. Smets has 30 years of design engineering experience and nuclear experience. At Hanford's Process Facility Modification Plant, he was responsible for facility design and coordination of the design effort. In 1988, he was assigned as the engineering manager for the Hanford

Waste Vitrification Plant, generally responsible for plant design development including technical criteria, safety analysis, nuclear engineering, and process engineering. The intent to duplicate the DWPF required a significant understanding of the Savannah River Facility and the work performed at West Valley. He continued building his vitrification background with several assignments, including studies at Hanford for alternative processing schemes for the low-level waste in the underground tanks. These tasks resulted in extensive work, gathering background on all of the vitrification facilities in the world, including visits to plants in Belgium and France. He supported the Hanford Process Engineering group in the Low-Level Melter "melt off" for potential melter selection for a Low-Level Waste Vitrification Plant. His most recent assignment is the FEMP, at a project he has supported since mid-1995. He performs technical reviews of the pilot plant and initiates preliminary engineering tasks to support the ultimate design of the full-scale vitrification facility. He performed the Pilot Plant Upgrade Study and most recently lead the group to develop the capital cost estimates for the treatment alternatives identified for the silo wastes.

Detlef Stritzke: Dr. Stritzke is a radio chemist and chemical engineer with 24 years of experience in the nuclear industry (high temperature gas cooled reactor [HTGR], vitrification of high-level waste, PUREX-process). For five years, he was research and development manager at the PAMELA Vitrification Facility in Mol, Belgium. He was involved in development and operation of radioactive waste vitrification systems. He was also involved in the Tank Waste Remediation System at Hanford for high- and low-level waste vitrification.

Robert Vogel: Mr. Vogel has more than 10 years DOE site experience, most of which has been focused on the Operable Unit 4 silos and the selection of a treatment method for this material. This primarily has involved the testing of vitrification technology of Operable Unit 4 materials. This effort began in 1989 with a scoping study on K-65 material, a more elaborate test on K-65 material in 1990 involving off-gas generation. This was followed by a treatability study in 1992, which included Silo 3 as well as the K-65 silos, and which determined the potential for a separate metal phase. He also supported the glass development study, which determined the optimum durability of the waste form and maximum waste loading. His other primary area of focus has been the measurement and treatment of radon. Previous experience has been in the area of environmental analysis (microscopy), energy conservation, and cholesterol adsorption. He has a masters degree in engineering with an area of concentration in hazardous and toxic substances.

Dave Yockman: Mr. Yockman has a bachelor of science degree in chemical engineering from Virginia Polytechnic Institute and State University, and a master of science degree in environmental engineering from the University of Maryland. He has worked for the DOE for more than five years, over

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four of which was spent at DOE Headquarters as a program manager developing environmental restoration policy, guidance, budget, and overseeing clean-up activities at the Fernald and Savannah River sites. Most recently, he has spent the past six months at the Fernald site as the DOE project engineer overseeing operations of the Fernald Vitrification Pilot Plant, and other remediation activities.

DATA ANALYSIS AND PATH FORWARD TEAM APPROVAL PAGE

Nina Akgündüz	2/24/97 Date
Chris Chapman	See attached fax Date
Robert Frost	2/20/97 Date
Rod Gimpel	2-21-97 Date
Doug Maynor	2/24/97 Date
Dennis A. Nixon	2/20/97 Date
John Plodinec	See attached fax Date
Harry Robertson	25 FEB 97 Date
John Smets	2/24/97 Date
Detlef Stritzke	See attached page Date
Robert Vogel	2/21/97 Date
Dave Yockman	2/24/97 Date

DATA ANALYSIS AND PATH FORWARD TEAM APPROVAL PAGE

Detlef Stritzke — 02/20/1997

Detlef Stritzke — Date

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DATA ANALYSIS AND PATH FORWARD TEAM APPROVAL PAGE

Nina Akgündüz	Date
Chris Chapman	Date
Robert Frost	Date
Rod Gimpel	Date
Doug Maynor	Date
Dennis A. Nixon	Date
John Plodinec	2/19/97 Date
Harry Robertson	Date
John Smets	Date
Detlef Stritzke	Date
Robert Vogel	Date
Dave Yockman	Date

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DATA ANALYSIS AND PATH FORWARD TEAM APPROVAL PAGE

Nina Akgündüz	Date
Chris Chapman	Date 2-22-97
Robert Frost	Date
Rod Gimpel	Date
Doug Maynor	Date
Dennis A. Nixon	Date
John Plodinec	Date
Harry Robertson	Date
John Smets	Date
Detlef Stritzke	Date
Robert Vogel	Date
Dave Yockman	Date