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GLASS MELTER OFF-GAS SYSTEM PLUGGAGES: CAUSE, SIGNIFICANCE, AND REMEDIATION (U)

by

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GLASS MELTER OFF-GAS SYSTEM PLUGGAGES: CAUSE, SIGNIFICANCE, AND REMEDIATION (U)

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ABSTRACT

Liquid high-level nuclear waste will be immobilized at the Savannah River Site (SRS) by vitrification in borosilicate glass. The glass will be produced in the Defense Waste Processing Facility (DWPF) where the glass will be poured into stainless steel canisters for eventual disposal in a geologic repository. Experimental glass melters used to develop the vitrification process for immobilization of the waste have experienced problems with pluggage of the off-gas line with solid deposits.

Off-gas deposits from the DWPF 1/2 Scale Glass Melter (SGM) and the 1/10th scale Integrated DWPF Melter System (IDMS) were determined to be mixtures of alkali rich chlorides, sulfates, borates, and fluorides with entrained Fe_2O_3, spinel, and frit particles. The distribution and location of the alkali deposits throughout the off-gas system indicate that the deposits form by vapor-phase transport and condensation. Condensation of the alkali-rich phases cement the entrained particulates causing off-gas system pluggages.

The identification of vapor phase transport as the operational mechanism causing off-gas system pluggage indicates that deposition can be effectively eliminated by increasing the off-gas velocity. Scale glass melter operating experience indicates that a velocity of >50 fps is necessary in order to transport the volatile species to the quencher to prevent having condensation occur in the off-gas line. Hotter off-gas line temperatures would retain the alkali compounds as vapors so that they would remain volatile until they reach the quencher. However, hotter off-gas temperatures can only be achieved by using less air/steam flow at the off-gas entrance, e.g. at the off-gas film cooler (OGFC). This would result in lower off-gas velocities. Maintaining a high velocity is, therefore, considered to be a more important criterion for controlling off-gas pluggage than temperature control.

The DWPF melter off-gas velocity design basis is 47 fps and the velocities at 350-450°C are calculated to be between 43-46 fps. It is, therefore, recommended that DWPF design velocities be increased to >50 fps by increasing the air/steam flow entering the off-gas film cooler (OGFC) entrance, by reducing the pipe diameter, or by upgrading the process chiller capacity. Since the process chiller design upgrade for DWPF is based on keeping the off-gas system cool, it is recommended that the chiller design upgrade be re-evaluated based on a velocity criterion rather than on temperature control.

The cementitious alkali borates, halides, and sulfates comprising the off-gas line deposits were determined to be water soluble. If pluggages occur beyond the off-gas line entrance then the deposits can be effectively removed with water and/or steam. It is recommended that the off-gas pipe, e.g. the jumper, be water or steam cleaned.In addition, engineering designs should be investigated to take advantage of the water soluble nature of the off-gas deposits for in-situ cleaning. This would minimize the need for mechanical disassembly and cleaning of the highly radioactive (^{137}Cs and ^{106}Ru) off-gas line could be minimized in DWPF.

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Table of Contents

Table of Contents (Cont'd)

LIST OF FIGURES

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LIST OF TABLES

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GLASS MELTER OFF-GAS SYSTEM PLUGGAGES: CAUSE, SIGNIFICANCE, AND REMEDIATION (U)

INTRODUCTION

High-level liquid nuclear waste (HLLW) will be immobilized at the Savannah River Site (SRS) by vitrification in borosilicate glass in the Defense Waste Processing Facility (DWPF). Glass will be poured into stainless steel canisters and eventually disposed of in a geologic repository. Since the late 1970s, experimental glass melters have been used to develop the vitrification process for immobilization of the waste. Several of these melters have experienced problems with pluggage of the off-gas line with solid deposits.

The Scale Glass Melter (SGM) exhibited problems with off-gas line pluggages between the off-gas film cooler (OGFC) and the quencher (Figure 1). The SGM pluggages were at greater distances from the off-gas line entrance than pluggages in previous research melters, e. g. the Small Cylindrical Melter (SCM) and the Large Slurry Fed Melter (LSFM). The SGM pluggages were not anticipated because the OGFC had been designed and tested[1-9] during the last several LSFM campaigns. The OGFC in the LSFM was reported to alleviate the off-gas line pluggage problems by

- cooling the off-gas entrance to between 350-450°C with high velocity air and/or steam to stop adhesion of partially melted particulates from the melter plenum
- removing splattered glass and entrained particulates at the off-gas line entrance by use of a mechanical reaming device (the off-gas film cooler brush)

Use of the OGFC in SGM successfully minimized deposits at the off-gas system entrance but did not eliminate deposits farther along the off-gas system.[10-11] The design used in the 1/2 scale SGM is prototypic for the 1/10th scale Integrated DWPF Melter System (IDMS) and the full scale Defense Waste Processing Facility (DWPF) melter.

The current IDMS and DWPF off-gas system design and operation are based on a hypothesis that pluggages near the off-gas line entrance are caused by adhesion of partially melted entrained particulates.[1-9] The particulate adhesion was determined to be aggravated by insufficient cooling of the off-gas pipe.[4] This hypothesis has resulted in the use of air and/or steam to cool the off-gas and the off-gas line and reduce deposition at the off-gas line entrance during operation of the LSFM and the SGM.[1-12] After pluggages occurred in the 7th campaign of the SGM (SGM-7), elimination of the off-gas line pluggages became DWPF Technology Issue #8.2.[10]

Preliminary analyses[13] indicated that low melting sulfate, chloride, and fluoride phases formed the cementitious portion of the off-gas system pluggages. Therefore, the 9th SGM campaign was run in a temperature-control mode for nine months and no deposits were observed to form in the off-gas line.[12,14] The lack of deposition was attributed to temperature control at 350 ± 25°C. Although off-gas velocities were not reported during SGM operation in the temperature control mode,[12] subsequent calculations[15] indicated that the off-gas velocity had to be 70 ± 5 fps in order to maintain the off-gas below 350 ± 25°C.

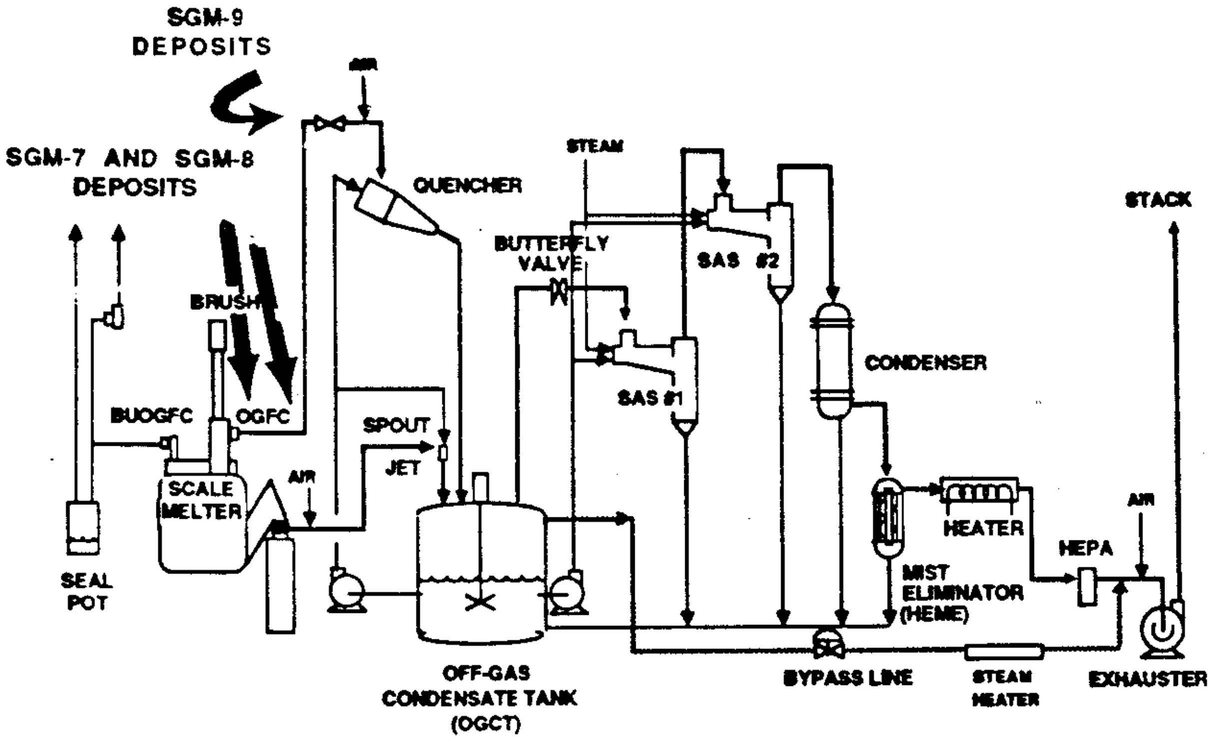

Figure 1.

The Scale Glass Melter Off-gas System.

Since initial startup the IDMS was run in the recommended temperature control mode at an off-gas velocity of only 40 fps. Off-gas deposits formed in the horizontal pipe near the OGFC during Campaigns #1 and #2 confirming that temperature control alone did not alleviate off-gas system pluggages.

OFF-GAS PLUGGAGE STUDY OBJECTIVES

The objectives of this study were to determine the phase identification and chemistry of off-gas deposits taken from the 1/2 scale SGM, the 1/100th scale 774-A minimelter, and 1/10th scale IDMS. Once the phase chemistry and phase distribution as a function of distance along the off-gas line was determined, the mechanism responsible for deposit buildup was identified. When the depositional mechanism was understood, then the operating conditions necessary to eliminate deposition could be defined. Understanding the depositional mechanism and the phase chemistry further provided a suggested remediation for deposit removal should it occur.

The off-gas system deposits analyzed in this study include the following:

- SGM-7 off-gas pipe deposits (June 1987)
- SGM-7 Cyclone Separator deposits (June 1987)
- Glass Technology's minimelter off-gas pipe deposits (July 1987)
- SGM-8 off-gas pipe deposits (September 1987)
- SGM-9 off-gas pipe deposits (January 1988)
- SGM-9 Quench Nozzle, HEME Nozzle, and Condensate Tank deposits (April 1988)
- IDMS Campaign #1, Run #2 off-gas pipe deposits (March 1989)
- IDMS Campaign #2, Run #3 off-gas pipe deposits (January 1990)

DESCRIPTION OF DEPOSITS AND DEPOSITION CONDITIONS

Off-gas Line Deposits During LSFM Campaigns

Between 1981 and 1984, off-gas line deposits from the Large Slurry Fed Melter (LSFM) Campaigns were analyzed. Deposits from LSFM-2 were found to contain crystalline NaCl, Fe_2O_3, and entrained glass.[16] The deposits were found to be enriched in Na, SO_4, Cl, F, Li, Si, B, and Cr. Deposits analyzed from the off-gas line entrance during LSFM-6, were found to be molten waste glass enriched in spinels, Na_2SO_4, and Cs_2SO_4.[3] Two mechanisms were postulated for the deposit accumulation. Initially, the entrainment of feed particles, at or near their melt temperature, which contact the hot metal pipe and adhere.[3,4] Subsequently the continual heating of the off-gas line to high temperature results in melting or softening of these particles producing a partial bond to the metal, which is extremely difficult to remove.[4] These hypotheses suggested the need for cooler off-gas line temperatures with higher velocity flows in order to alleviate particle contact and deposit buildup. This led to the design of the first OGFC which was tested during LSFM-7.

During LSFM-7, steam was found to be more efficient at maintaining wall temperatures because design limitations allowed more steam to be supplied to the OGFC than air. The only deposits observed at the entrance to the OGFC after LSFM-7 and LSFM-8 were comparable to the waste glass composition being melted but somewhat enriched in frit components.[4,5] Deposition was attributed to splatter and entrainment mechanisms.[4,5]

During LSFM-9, minimum deposition was observed in the OGFC which was attributed to the high transport velocities (averaged 60 fps) used during this campaign.[8] The film cooler brush assembly installed during LSFM-7 was successfully tested and found to mechanically remove deposits formed by particulate entrainment and glass splattering.[8] Some deposits were found farther along the off-gas line, in the elbows and flange areas beyond the OGFC.[7] The deposits were determined to be not only sludge rich, but enriched in cesium and chloride. Because enrichment of cesium and chloride was found on the off-gas filter, off-gas condensation was ruled out as an operable mechanism in favor of semi-volatile transport.[7] The success of the OGFC was attributed to the boundary layer of air and/or steam that cooled the interior walls of the off-gas line entrance so that contact and adherence of particles was retarded.[9]

Off-gas Line Deposits During SGM Campaigns

The Scale Glass Melter (SGM) had been operating for approximately sixteen months (Campaigns 6 and 7) when melter pressure control problems prompted the first inspection of the off-gas line. The problems were found to be caused by solid deposits which had severely restricted the cross-sectional area of the off-gas line.[10] The deposition was partially attributed to poorer off-gas decomposition during the SGM-7 campaign when the effects of deliberately lowering the vapor space temperatures (as low as 590°C) were investigated.[10] The section of the off-gas line between the film cooler and the quencher (Figures 1 and 2) had heavy accumulations of solid deposits which were black, white, and yellow in color. Samples were taken at various locations in the the line for analysis and the entire off-gas line was scraped clean. About 1.8 pounds of deposits were removed.[10]

SGM-8 was a dry feed campaign which was run to investigate pouring at the DWPF reference rate.[11] Pressure drops in the off-gas line (header) were also experienced during Campaign 8.[11,12] Inspection of the off-gas line after SGM-8 revealed similar deposits had accumulated on the walls of the line between the film cooler and the quencher (Figures 1 and 2). Samples were taken for analysis and the entire off-gas line was cleaned. About 0.6 pounds of deposits were removed.[11]

Since preliminary analysis[13] had indicated that the cementitious portion of the off-gas deposits was composed of water soluble salts which had low melt temperatures, e.g. (Na,K)Cl, $(Na,K)_2SO_4$, and $(Na,K)BF_4$, the film cooler was operated in a temperature-control mode during SGM-9 which maintained the temperature of the off-gas exiting the film cooler at 350° ± 25°C (below the melting point of the lowest salt phase identified, KBF_4).[12,14] Air flow rates were held constant while the steam flow was allowed to vary. After one half of the campaign, the off-gas header was inspected and showed no appreciable deposit buildup. This was attributed to the lower off-gas temperatures achieved by adding steam in the film cooler. During the second half of the campaign, a DWPF prototypic off-gas header was installed. At the end of the campaign, the line was again inspected and no deposits had accumulated. However, the 90° bend between the off-gas header and the isolation valve was inspected for the first time during SGM operation (Figures 1 and 3). Deposits covered nearly 50% of the cross-sectional area. This region of the off-gas line had never been inspected before and the formation of the deposits was attributed to accumulation over the lifetime of the melter. Samples of this material were taken for analysis.

Minimelter Off-gas Line Deposits

Off-gas deposits had been observed in the Glass Technology Group's 1/100th scale minimelter in 774-A and samples of this material were taken for analysis and comparison.

IDMS Off-gas Line Deposits

The Integrated DWPF Melter System was run in the temperature control mode suggested by Rabon[12] but at off-gas velocities of 40 fps. Off-gas deposits formed after the first and second campaigns. The first campaign consisted of startup with 165 black startup frit, and Runs #1 and #2 with slurry-fed 165 frit and sludge-only waste. Deposits labeled IDMS #1-2 were taken from the horizontal portion of the off-gas system near the entrance to the film cooler. Both loose deposits and deposits scraped from the off-gas pipe were sampled for analysis. After the third run of the second IDMS campaign, deposition was observed in the film cooler and in the horizontal portion of the off-gas system between the OGFC and the inspection port (Figure 4). Black deposits were located closer to the off-gas system entrance while white salt like deposits were located further along the off-gas line (Figure 4). Three samples of the black deposits were taken for analysis.

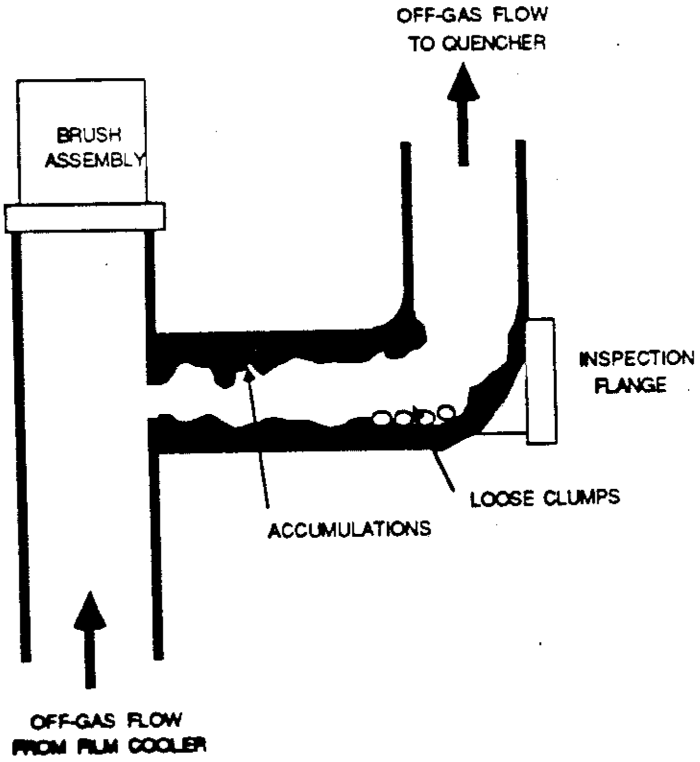

Figure 2.

Location of SGM-7 and SGM-8 Off-Gas Deposits.

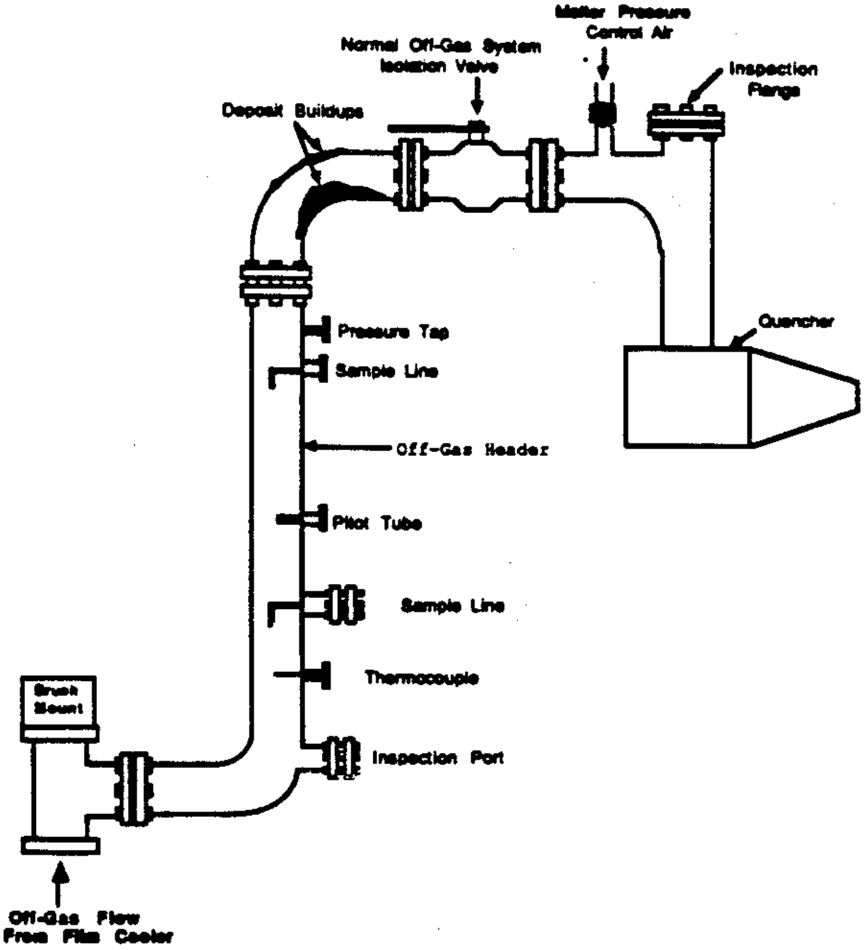

Figure 3.
Location of SGM-9 Off-Gas Deposits.

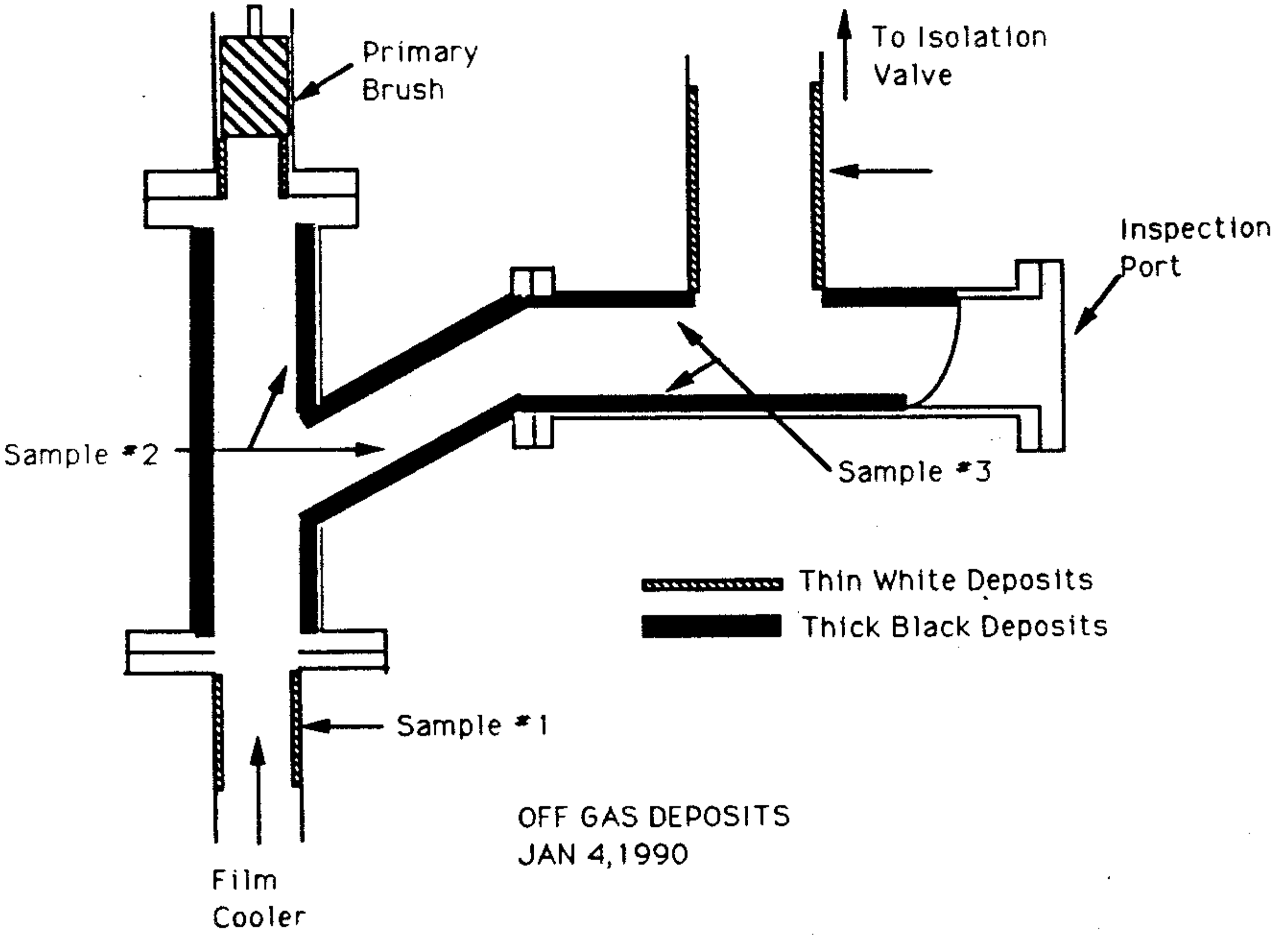

Figure 4.

Location of IDMS Off-gas Deposits after Campaign #2.

SGM Cyclone Separator Deposits

The pour spout vacuum line plugged with fibrous "hair-like" material on several occasions during SGM-3.[10] These pluggages made the pouring system inoperable and became a concern for DWPF design. To prevent pluggages, the Engineering Department proposed that a funnel baffle be installed in the bellows assembly and added to the DWPF design. Prior to SGM-6, a cyclone separator was installed on the vacuum line (Figure 5) to collect and remove these fibers.

After SGM-6 the cyclone separator was inspected and only minor salt deposits were present.[10] After SGM-7, however, a buildup of fibers in the cyclone separator caused the pour system to be inoperable. This occurred during periods of low glass pour rates.[10] During inspection of the cyclone separator, a large clump of fibers and salt deposits fell from the separator and were retained for analysis. Use of the cyclone separator became the contingency design for DWPF.[10,17] Inspections of the Cyclone Separator after SGM-9 and SGM-10 indicated that small amounts of salts had collected but no glass fibers were present. The absence of fibers in the separator indicated that the funnel baffle in the bellows was performing properly.[17]

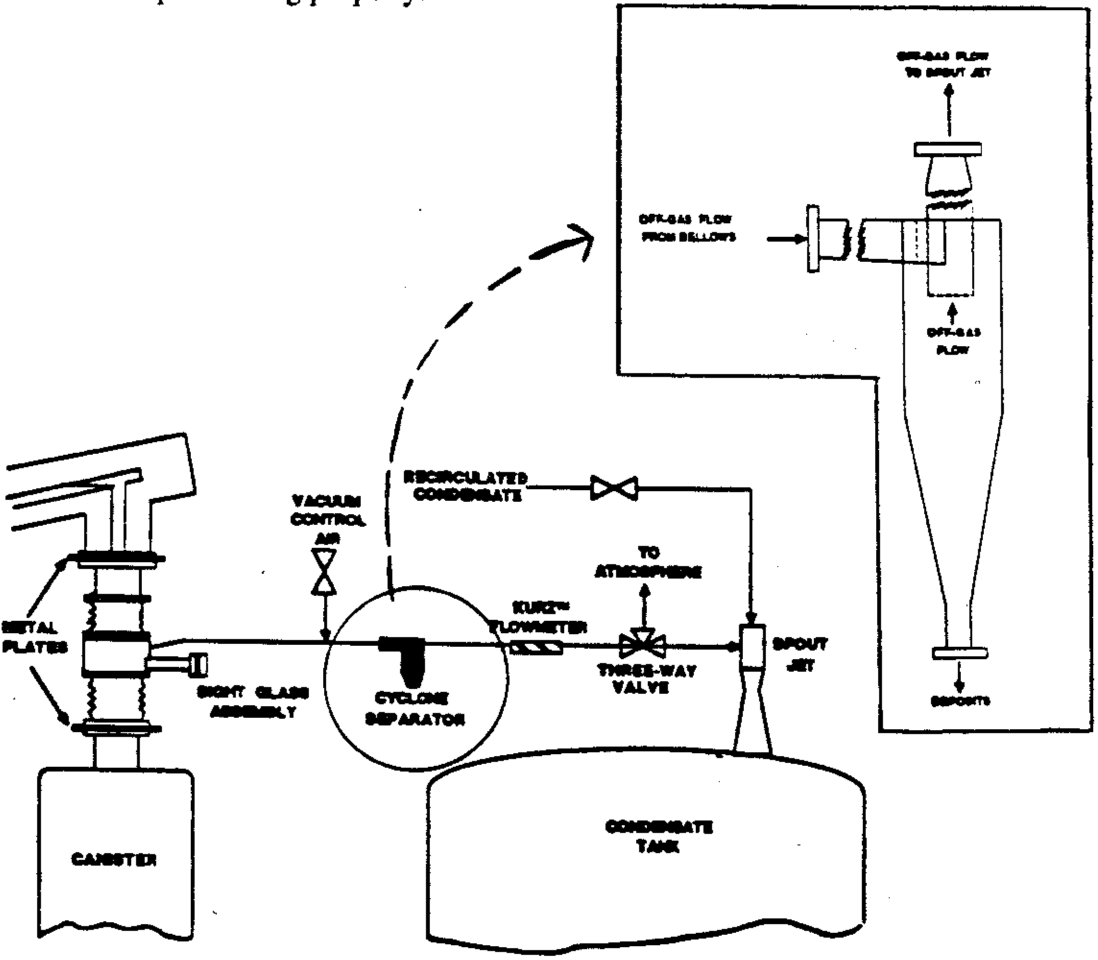

Figure 5.

Vacuum Line Cyclone Separator.

SGM Quencher Nozzle, HEME Spray Nozzle, and Condensate Tank Deposits

The SGM quencher uses recirculated condensate to cool the hot off-gas (Figure 1).[14] The recirculated condensate is sprayed into the off-gas through a DWPF prototypical nozzle, which was installed prior to SGM-7.[10] During SGM-9 bellows air inleakage tests a pluggage in the line was detected. Visual inspection indicated that both white and black colored deposits had formed. Black and white colored deposits were also found on the spray quencher nozzle. These deposits were scraped off for analysis (Figure 6).[18]

Deposits were also found in the spray nozzle (Figure 1) used in the High Efficiency Mist Eliminator (HEME) and scraped off for analysis.[18] Since the HEME spray nozzle also uses recirculated condensate, samples of the liquid and solids portion of the condensate tank (Figure 1) were requested and analyzed.

EXPERIMENTAL

Off-gas pluggage samples taken from various locations along the off-gas line after SGM-7 (Figure 7) were received in June 1987. A sample of the "hair" from the Cyclone separator was received at the same time (Figure 8). The wt% solids was determined for the two samples by heating in Al_2O_3 crucibles to 900°C for 21 hours. Each off-gas pipe sample and the Cyclone separator sample were analyzed for crystalline phase content by x-ray diffraction (XRD). Since the waste glass is amorphous and cannot be detected by XRD analysis, X-ray fluorescence (XRF) analysis was employed to determine if the heavier elemental components of the waste glass were present. Each sample was also analyzed by Scanning Electron Microscopy (SEM) coupled with the qualitative elemental analytic capability of Energy Dispersive Analysis by X-ray (EDAX).

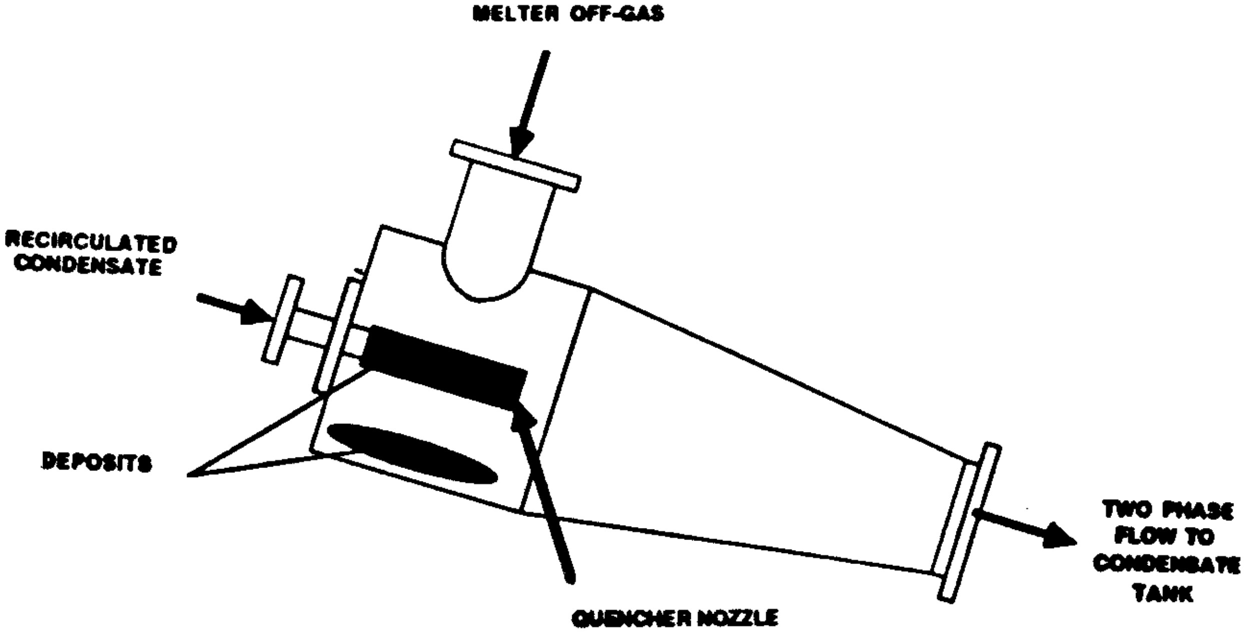

Figure 6.

Location of Off-Gas Quencher Deposits.

Each off-gas sample was sent for chemical analysis in July 1987. Poor replicate analyses and precision, based on the findings of the XRD and SEM-EDAX analysis, required that the solid samples be resubmitted for analysis in October 1987 and in June 1988. The solid samples were chemically analyzed by the following techniques:

- Dissolution by Na_2O_2 with an HCl uptake
  - ICP for Al, Ca, Fe, Mg, Mn, Li, Si, Cr, B, Sr, Ti, P, Ba, Pb, Mo, Zn, Cu

- Dissolution by HCl/HF bomb
  - ICP for Na, Ni, Zr
  - AA for Na, K, Cs

- Dissolution by Na_2O_2 with a H_2O uptake
  - IC for SO_4, NO_3, NO_2
  - ISE for Cl and F

A hard solid piece of the SGM-7 pluggage material was put in deionized water in an ultrasonic bath at room temperature for ~ 5 minutes. The cementitious portion of the sample was water soluble and left a residue of dispersed black solid particulates. The water was decanted and sent for the following analyses:

- ICP (Al, Ca, Fe, Mg, Mn, Li, Si, Cr, B, Sr, Ti, P, Ba, Pb, Mo, Zn, Cu, Na, Ni, Zr)
- AA (Cs, K, Na)
- IC (SO_4, NO_3, NO_2)
- ISE (Cl, F)

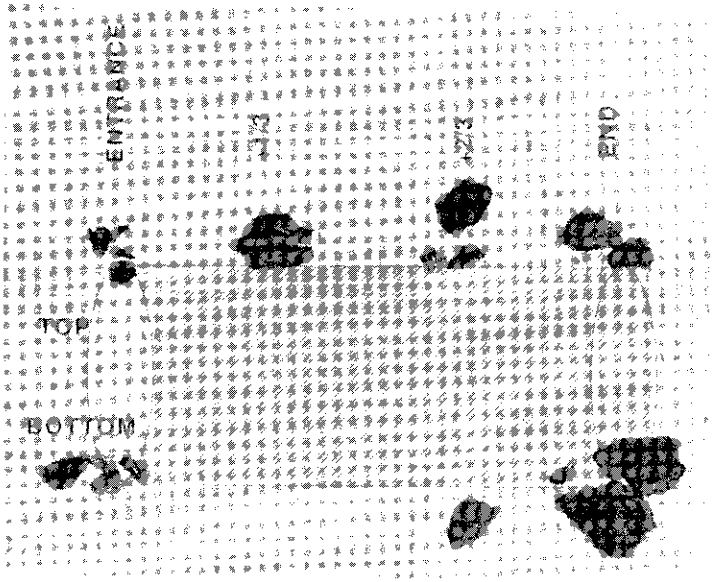

Figure 7.

Location of Off-Gas Deposits Sampled at Various Locations Along the Off-Gas Line after SGM-7.

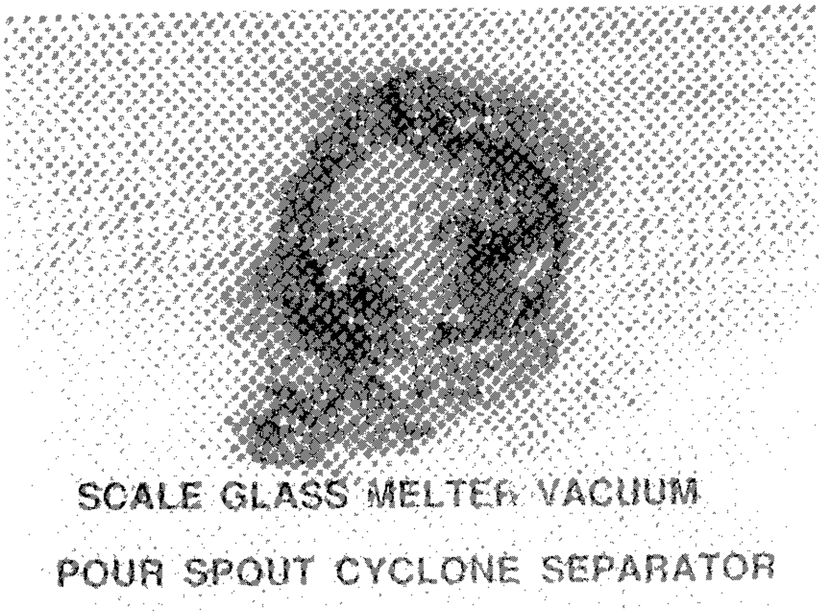

Figure 8.

Sample of "Hair-Like" Deposits Which Fell From the Cyclone Separator.

A similar experiment was run on the Cyclone separator "hair" deposits. The solid residues were sent for XRD phase identification.

Off-gas deposits from the Glass Technology Group's minimelter were received in July 1987 and samples from SGM-8 were received in September 1987. Deposits from the SGM-9 off-gas line above the off-gas header (Figure 1 and 3) were received in January 1988. Samples from IDMS Campaign #1 Run #2 were received in March 1989 and samples from Campaign #2 Run #3 were received in January 1990 (Figure 4). XRD analysis and chemical characterization by dissolution as outlined above were performed on these samples for comparison with the SGM-7 off-gas pluggage materials.

Deposits from the SGM-9 quencher nozzle and HEME spray nozzle were received in April 1988. Samples of the condensate tank contents were requested at that time for chemical comparison. The quencher nozzle and spray nozzle deposits were sent for XRD and SEM-EDAX as outlined above for other solid deposits. There was insufficient sample for dissolution and whole element chemistry. The condensate was composed of a liquid and a solid portion. The liquid was filtered off and sent for ICP and AA analyses comparable to those used for the ultrasonic rinse given above. The solid, which was caught on the filter paper, was air dried and sent for XRD and SEM to determine phase identification, morphology, and quantitative chemistry. There was insufficient solid condensate material to determine the chemistry by dissolution and chemical analysis.

Different ICP instruments were used for analyses between December 1986 to October 1988 and November 1988 to present. The detection limits for each of these instruments is listed in Table 1. The major elements (K, Na, Ca, Mg, Si, Al, and Fe) are accurate to within ±10% at concentrations >1 ppm. Within a factor of two of the detection limits listed (Table 1), the precision of any element is only ±100%.

QUALITY ASSURANCE

All the Scale Melter activities and analyses were performed in accordance with the Savannah River Quality Assurance Plan (DWP-82-111-2) and the Defense Waste Processing Section Quality Assurance Plan (DPST-QA-83-4-2). The QA plan assures that research, development, and demonstration are performed in a safe and controlled manner, so that the resulting technical data are valid and retrievable. All the data for this study are recorded in WSRC-NB-90-391 (DWPT-QA-89-1041).

Table 1.

Detection Limits of Inductively Coupled Plasma Spectroscopy (ICP) and Atomic Adsorption (AA)

Element	December 1986-October 1988 ppm (Ref. 19)	November 1988-present ppm (Ref. 20)	Technique
Al	0.09	0.025	ICP
Ca	0.03	0.0001	ICP
Cu	0.02	0.003	ICP
Fe	0.01	0.004	ICP
K	0.005	0.005	AA
Mg	0.003	0.0001	ICP
Na	0.06	0.025	ICP
P	0.04	0.03	ICP
Pb	0.07	0.05	ICP
Ru	0.02	0.025	ICP
Si	0.03	0.02	ICP
Ti	0.002	0.003	ICP
Zr	0.002	0.004	ICP

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RESULTS AND DISCUSSION

Weight Loss Measurements

Samples of the nine SGM-7 off-gas line deposits were heated to 900°C for 21 hours in open Al_2O_3 crucibles. No weight loss was observed indicating that the material was not composed of low vapor pressure organics nor hydrous crystalline phases.

Chemical Analysis of the SGM-7 Off-gas Line Deposits

The chemical analyses of the nine SGM-7 off-gas deposits taken at various locations along the horizontal section of the line (Figures 1 and 7) revealed that they were enriched in Na, K, and Cs compared to the relative amounts in the waste glasses melted in SGM from startup (Table 2). The deposits were also highly enriched in anions, primarily SO_4 and Cl but small amounts of F were present as well (Table 2). Enrichment in Cr, Fe, and B was also observed.

The deposits from the off-gas pipe entrance contained more Fe and B than the deposits further along the line indicating that entrained glass and iron-rich spinels were probably present as observed at the off-gas system entrance during the LSFM campaigns. The x-ray fluorescence (XRF) analyses indicated the presence of the heavier minor constituents of the glass, e.g. Ti, Zr, and Sr. This confirmed the presence of entrained glass.

Deposits further from the off-gas pipe entrance contained more Cr, Cl, and SO_4. Sodium and F were distributed evenly in the off-gas pipe deposits regardless of location. The initial chemistry, therefore, suggested that salt deposits, entrained spinel, and glass were the major species present.

Phase Analysis of the SGM-7 Off-gas Line Deposits

The x-ray diffraction analysis of the nine SGM-7 off-gas line deposits revealed very complex crystalline phase chemistry. The samples contained from six to twelve individual crystalline phases (Table 3). Many of the phases are solid solutions of halide salts such as (Na,K,Cs)Cl. Pure NaCl [Joint Committee on Powder Diffraction Standards (JCPDS) # 5-628), pure KCl (JCPDS #4-587), and mixtures such as $(Na_{0.4}K_{0.6})Cl$ (JCPDS # 26-920) can all be present giving distinctly different but closely overlapping x-ray spectra. A qualitative analysis of the phase assemblages in each sample is given in Table 3.

The crystalline portion of the SGM-7 deposits are primarily mixtures of (Na,K,Cs)Cl, $(Na,K,Cs)_2SO_4$, spinel of the $NiFe_2O_4$ type, Fe_2O_3 (believed to be an unreacted sludge component), SiO_2 and $Na_2B_4O_7$. Traces of KBF_4 and $(Na,K)CrO_4$ were also detected. The major constituent of each deposit is NaCl and either Na_2SO_4 or mixed $(Na,K,Cs)_2SO_4$ (Table 3). The X-ray analysis indicated that stainless steel (which has an x-ray spectra similar to Inconel 690) was also present in most of the samples. The source of the steel is likely from adhesion of some of the Inconel 690 pipe to the deposits when then are removed.

The SEM and EDAX analyses confirmed that the white and yellow deposits were rich in the salt components S, Cl, K, and Na while the black deposits were enriched in the glass and spinel components, Fe, Ni, Mn, and Ti.

Table 2. Analyses of Deposits from SGM-7, -8, -9, the Cyclone Seperator, the GTG Minimelter, and IDMS.*

ELEMENT (WT%)	SGM-7 ENTRANCE TOP	SGM-7 ENTRANCE BOTTOM	SGM-7 +1/3 TOP	SGM-7 BOTTOM VARIOUS	SGM-7 +2/3 TOP	SGM-7 +2/3 BOTTOM	SGM-7 END SCRAPINGS	SGM-7 FLANGE SCRAPINGS	SGM-7 VARIOUS SCRAPINGS	SGM-7 CYCLONE SEPARATOR "HAIR"
Al (ICP)	3.29	3.32	1.81	1.70	1.79	1.60	1.65	1.67	1.62	1.69
Ca (ICP)	1.70	3.49	1.84	1.79	1.82	1.64	1.54	1.55	0.56	1.79
Fe (ICP)	18.90	18.06	14.00	13.80	14.50	12.79	12.90	12.40	3.54	13.50
Mg (ICP)	0.21	0.19	0.11	0.13	0.10	0.09	0.09	0.12	0.21	0.11
Mn (ICP)	0.99	1.46	3.11	2.90	3.02	2.66	2.74	2.46	0.70	1.94
Na (AA)	12.18	13.18	13.52	13.46	13.93	12.58	13.40	14.39	13.94	15.13
Li (ICP)	0.69	1.19	0.54	0.78	0.65	0.55	0.60	0.73	0.88	0.67
Ni (ICP)	1.55	0.91	1.34	0.98	1.19	1.25	1.13	1.10	1.13	0.49
Si (ICP)	2.56	4.21	2.49	3.47	2.56	2.65	2.34	4.33	7.18	2.94
Cr (ICP)	0.28	0.61	0.31	0.49	0.41	0.41	0.50	0.46	0.91	0.47
B (ICP)	7.00	5.73	1.80	3.56	3.34	1.80	3.44	1.57	3.99	3.65
Sr (ICP)	0.12	0.12	0.06	0.04	0.05	0.04	0.04	0.03	0.00	0.44
Zr (ICP)	0.12	0.02	0.04	0.06	0.04	0.05	0.04	0.09	0.08	0.06
Ti (ICP)	0.03	0.03	0.21	0.27	0.22	0.21	0.03	0.25	0.13	0.24
K (AA)	6.93	7.72	6.77	4.71	5.88	7.90	7.11	6.21	7.37	6.14
Cs (AA)	0.98	2.01	0.73	1.61	1.16	1.95	1.61	2.15	1.31	3.86
Cl (ISE)	4.29	3.49	4.08	4.64	4.24	4.76	6.72	3.10	6.43	13.43
F (ISE)	0.12	0.13	0.09	0.42	0.11	0.15	0.14	0.10	0.15	5.89
SO4 (IC)	9.07	17.01	18.58	14.23	15.20	14.70	14.62	19.96	13.56	5.28
NO3/NO2 (IC)	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
MOLAR BALANCE (M)										
(Na,K)2SO4	14.93	28.00	30.59	23.43	25.02	24.20	24.07	32.86	22.32	8.69
(Na,K)Cl	8.05	6.54	7.66	8.69	7.95	8.92	12.60	5.82	12.06	25.18
(Na,K)BF4	0.18	0.20	0.14	0.65	0.18	0.23	0.22	0.16	0.24	9.13
(K,Na)2B4O7	35.10	28.71	8.98	17.59	16.70	8.94	17.19	7.82	19.94	14.13
Na2CrO4	0.88	1.89	0.97	1.51	1.26	1.27	1.57	1.44	2.82	1.45
OXIDES	50.39	56.28	46.49	46.36	47.24	46.94	41.33	49.63	36.20	45.75
SUM	109.53	121.62	94.83	98.23	98.35	90.50	96.97	97.72	93.58	104.33
NORMALIZED MOLE %										
SALTS	21.15	28.57	40.48	33.36	33.70	36.85	38.04	39.74	37.00	41.22
BORATES	32.04	23.61	9.47	17.91	16.98	9.88	17.73	8.00	21.31	13.55
CHROMATES	0.81	1.55	1.03	1.54	1.29	1.40	1.62	1.48	3.01	1.39
OXIDES	46.00	46.27	49.02	47.20	48.03	51.87	42.62	50.78	38.68	43.85
SUM	100.00	100.00	100.00	100.01	100.00	100.00	100.01	100.00	100.00	100.01

* For details see Appendix I

Table 2. Analyses of Deposits from SGM-7, -8, -9, the Cyclone Seperator, the GTG Minimelter, and IDMS.*

ELEMENT (WT%)	SGM-8 PLUGGAGE	SGM-9 PLUGGAGE	GTG MINI-MELTER	IDMS 1-2 PIPE "STUCK" Apr-89	IDMS 1-2 PIPE "LOOSE" Apr-89	IDMS 2-3 OGFC DEPOSIT 1 Jan-90	IDMS 2-3 OGFC DEPOSIT 2 Jan-90	IDMS 2-3 DEPOSIT 3 Jan-90
Al (ICP)	1.22	1.43	2.00	1.83	2.95	0.70	2.57	2.75
Ca (ICP)	0.85	1.07	1.72	3.70	4.15	0.24	1.58	1.74
Fe (ICP)	6.57	9.79	10.38	11.06	17.63	3.04	12.83	14.58
Mg (ICP)	0.23	0.11	0.17	0.14	0.20	0.02	0.12	0.13
Mn (ICP)	1.37	1.57	3.70	2.29	3.35	0.66	2.82	3.07
Na (AA)	13.50	12.56	8.75	13.31	8.75	NO DATA	10.83	10.64
Li (ICP)	1.18	1.18	1.19	0.83	1.01	1.09	0.68	0.75
Ni (ICP)	0.37	0.89	0.74	0.98	1.57	0.23	1.08	3.19
Si (ICP)	9.52	6.94	4.18	5.17	7.89	0.67	2.92	3.21
Cr (ICP)	0.80	0.35	1.22	1.42	0.73	1.18	1.09	2.06
B (ICP)	4.64	1.10	2.38	2.37	2.70	1.50	1.62	1.39
Sr (ICP)	0.01	0.27	0.04	0.01	0.01	0.01	0.03	0.03
Zr (ICP)	0.25	0.07	0.09	0.08	0.13	NO DATA	0.04	0.04
Ti (ICP)	0.11	0.14	0.17	0.04	0.07	0.05	0.22	0.23
K (AA)	3.51	5.92	1.37	1.67	0.74	NO DATA	5.93	6.51
Cs (AA)	0.44	1.00					1.10	1.40
Cl (ISE)	11.85	5.84	10.53	5.84	2.06	NO DATA	13.42	11.57
F (ISE)	0.13	0.26	12.77	2.18	1.29	NO DATA	1.11	3.87
SO_4 (IC)	7.35	27.14	0.89	7.57	1.99	NO DATA	8.07	11.50
NO_3/NO_2 (IC)	BDL	BDL	BDL	BDL	BDL	NO DATA	BDL	BDL
MOLAR BALANCE (M)								
$(Na,K)_2SO_4$	12.10	44.68	1.47	12.47	3.27	NO DATA	13.29	18.93
$(Na,K)Cl$	22.22	10.95	19.74	10.95	3.86	NO DATA	25.16	21.69
$(Na,K)BF_4$	0.20	0.00	19.80	3.39	2.00	NO DATA	1.72	6.00
$(K,Na)_2B_4O_7$	23.23	5.34	2.81	10.33	12.64	NO DATA	7.35	4.21
Na_2CrO_4	2.48	1.07	3.79	4.42	2.27	NO DATA	3.39	6.42
OXIDES	38.39	37.31	48.25	48.71	69.08	NO DATA	39.69	46.04
SUM	98.61	99.76	95.86	90.26	93.12		90.60	103.29
NORMALIZED MOLE %								
SALTS	35.00	56.17	42.78	29.70	9.81	NO DATA	44.33	45.14
BORATES	23.56	5.36	2.93	11.44	13.57	NO DATA	8.11	4.08
CHROMATES	2.51	1.08	3.95	4.90	2.44	NO DATA	3.75	6.21
OXIDES	38.93	37.40	50.33	53.96	74.18	NO DATA	43.81	44.57
SUM	100.00	100.01	99.99	100.00	100.00		100.00	100.00

* For details see Appendix I

Table 3. Phase Identification of Crystalline Species in Deposits from SGM-7, -8, -9, the Cyclone Seperator, the GTG Minimelter, and IDMS.

CRYSTALLINE SPECIES	SGM-7 ENTRANCE TOP	SGM-7 ENTRANCE BOTTOM	SGM-7 +1/3 TOP	SGM-7 BOTTOM VARIOUS	SGM-7 +2/3 TOP	SGM-7 +2/3 BOTTOM	SGM-7 END SCRAPINGS	SGM-7 FLANGE SCRAPINGS	SGM-7 VARIOUS SCRAPINGS	SGM-7 CYCLONE SEPARATOR "HAIR"	SGM-7 ULTRA-SONIC SOLIDS
NaCl (JCPDS 5-628)	MAJOR**	MAJOR	MAJOR	MAJOR	MAJOR	MAJOR	MAJOR	MAJOR	MAJOR	MAJOR	Trace
KCl (JCPDS 4-587)		Present	Trace	Present	Trace	Trace	Present	Present	Present		Present
K0.6Na0.4Cl (JCPDS 26-920)	Trace			Present							
Na2SO4 (JCPDS 37-1465)	MAJOR	Present	MAJOR	MAJOR	Present	MAJOR	Present	Present	Present	MAJOR	Present
K2SO4 (JCPDS 25-681)		Trace	Present	Present	Trace	Present	Trace	Present			
(Na,K)2SO4 (JCPDS 33-664)		MAJOR	MAJOR	Present	Present	Present	Present	Present	MAJOR		
Spinel (JCPDS 10-325)	MAJOR	Present	MAJOR	Present	Present	Present	Present	Present	Trace		MAJOR
Fe2O3 (JCPDS 13-534)	Present	Present	Present	Present	Present	Present	Present	Present		Trace	MAJOR
SiO2 (JCPDS 33-1161/11-695)	Present	Present	Present	Present	Present	Present	Present	Present		Present	Present
(K0.25Na0.75)2CrO4 (JCPDS 26-1467)		Present							Present		
KBF4 (JCPDS 16-378)		Present					Present			MAJOR	Present
Na2B4O7 (JCPDS 22-1376)		Trace	Present	Present	Trace			Trace			
Stainless Steel (JCPDS 35-1375)		Trace	Present	Present	Present	Present	Present	Present	Present		Present
Sulfur (JCPDS 23-562)											

** MAJOR 25-100%
Present 5-25%
Trace <5%

Table 3. Phase Identification of Crystalline Species in Deposits from SGM-7, -8, -9, the Cyclone Seperator, the GTG Minimelter, and IDMS.

CRYSTALLINE SPECIES	SGM-8 PLUGGAGE	SGM-9 PLUGGAGE	GTG MINI-MELTER	IDMS 1-2 PIPE "STUCK" Apr-89	IDMS 1-2 PIPE "LOOSE" Apr-89	IDMS 2-3 OGFC DEPOSIT 1 Jan-90	IDMS 2-3 OGFC DEPOSIT 2 Jan-90	IDMS 2-3 DEPOSIT 3 Jan-90
NaCl (JCPDS 5-628)	MAJOR**	MAJOR	MAJOR	Present	MAJOR	MAJOR	MAJOR	MAJOR
KCl (JCPDS 4-587)		Present	Present	Present	Present	MAJOR	Present	Present
K0.6Na0.4Cl (JCPDS 26-920)		Present						
Na2SO4 (JCPDS 37-1465)	Present	MAJOR	Trace	Present	MAJOR			
K2SO4 (JCPDS 25-681)				Present	Present			
(Na,K)2SO4 (JCPDS 33-664)	MAJOR	MAJOR				Present	Present	Present
Spinel (JCPDS 10-325)	Present	Present	Present	MAJOR	Present	Present	MAJOR	MAJOR
Fe2O3 (JCPDS 13-534)	Present	Present	Present	Present	Present		Present	Present
SiO2 (JCPDS 33-1161/11-695)		Present		Present	Present			
(K0.25Na0.75)2CrO4 (JCPDS 26-1467)								
KBF4 (JCPDS 16-378)	Present	Present			Trace			
Na2B4O7 (JCPDS 22-1376)	Present		Trace	Present	Present			
Stainless Steel (JCPDS 35-1375)	MAJOR	MAJOR		Trace	MAJOR			
Sulfur (JCPDS 23-562)		Present		Trace	Present			

** MAJOR 25-100%
Present 5-25%
Trace <5%

A large SGM-7 off-gas line deposit which appeared to have white and yellow salt deposits layered in between the dark oxide layers was analyzed by SEM/EDAX (Figure 9). The EDAX analysis of the salt and oxide deposits were similar to the other analyses of SGM-7 deposits. The micrographs did, however indicate that deposition of the salts and oxides (mostly spinel and glass) may be cyclic. The salt deposits appear to bind the oxide layers to each other.

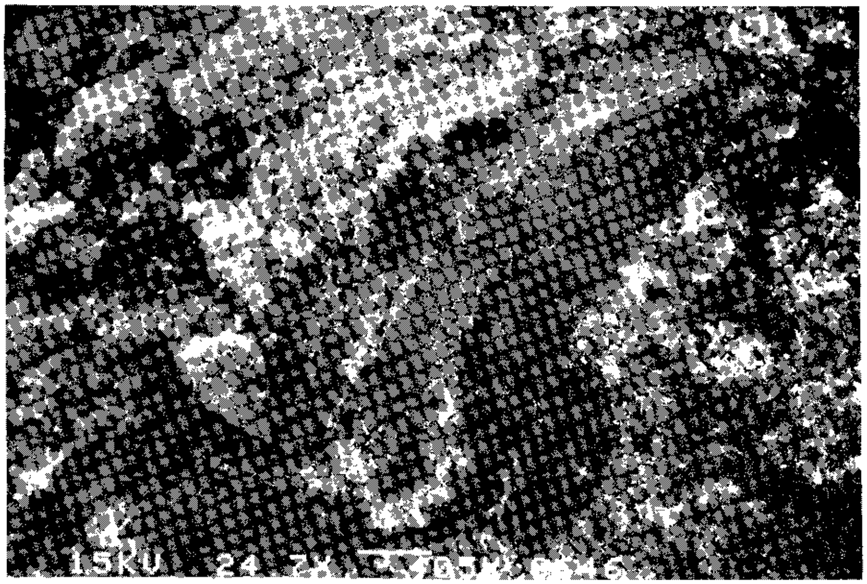

Figure 9.

"Layered" or "Cyclic" Nature of the Salt versus Oxides Components of the SGM-7 Off-Gas Deposits.
Note that the Salt Deposits Appear to Bind the Oxide Deposits Together.

The major amorphous species in the SGM-7 off-gas deposits is entrained glass. Based on the known Li/B ratio of the glasses run in SGM, boron is enriched in great excess over Li. This indicates that boron enrichment is coming from a secondary source and not merely from entrained glass. Since x-ray diffraction analysis detected $Na_2B_4O_7$ in almost all the samples, the excess boron is likely to be present as partially amorphous or poorly crystallized $Na_2B_4O_7$ in all the samples.

Mass Balance Calculations for SGM-7 Off-gas Line Deposits

The x-ray diffraction and chemical analyses of the SGM-7 off-gas deposits indicate that the amorphous and crystalline phases are the following:

- Sodium/potassium/cesium salts
  $(Na,K,Cs)_2SO_4$
  $(Na,K,Cs)Cl$
  $(Na,K,Cs)BF_4$

- Sodium/potassium borate
  $(Na,K)_2B_4O_7$

- Sodium/potassium chromates
  $(Na,K)_2CrO_4$

- Oxides
  $NiFe_2O_4$ (spinel)
  Fe_2O_3
  SiO_2
  Glass

A molar mass balance based on the five sodium/potassium rich phases and the residual oxides was calculated (Table 2). Although cesium will be present primarily as halides and sulfates, the small amount of cesium present in most samples (0.73-2.01 wt%) was, for simplicity, calculated as an oxide forming glass component. This adds only a small error to the overall mass balance which is based primarily on the speciation of alkali salts and alkali borates to oxides.

All of the SO_4 was assumed to form $(Na_{0.5}K_{0.5})SO_4$. All of the Cl was assumed to form $(Na_{0.5}K_{0.5})Cl$, all of the F was assumed to form $(Na_{0.5}K_{0.5})BF_4$. All of the Cr was assumed to form Na_2CrO_4. The boron remaining over that assigned to $(Na,K)BF_4$ was assigned to $(Na_{0.5}K_{0.5})B_4O_7$. This probably overestimates the amount of $(Na_{0.5}K_{0.5})B_4O_7$ somewhat since boron is not calculated as a component of the entrained glass. By assuming that the lithium is not preferentially incorporated into the off-gas alkali salts, the magnitude of the overestimation can be calculated from the known lithium concentration. Since the boron/lithium ratio of SGM-7 glass 200 is known to be ~2.2 and the lithium concentrations in the off-gas deposits range from 0.54 to 1.19 wt%, the boron concentrations which could be assignable to entrained glass are 1.2 to 2.6 wt%. This converts to a potential overestimation of the $(Na_{0.5}K_{0.5})B_4O_7$ of 6 to 13 mole%. If the boron is preferentially assigned to entrained glass rather than to the $(Na_{0.5}K_{0.5})B_4O_7$ during the calculation, then there is insufficient boron to predict the formation of $(Na_{0.5}K_{0.5})B_4O_7$ which is observed in the x-ray diffraction patterns. The B was, therefore, assigned to the $(Na_{0.5}K_{0.5})B_4O_7$ phase rather than to entrained glass and the remaining elements were calculated generically as oxides.

Appendix I gives the calculational equations used and Appendix II gives the values calculated for the SGM-7 samples. The mass balance results from Appendix II are summarized in Table 2. The mass balance sums calculate to be between 91-122 mole %. The mass balance summaries (Table 2) are in qualitative agreement with the results of the x-ray diffraction analyses in Table 3.

Analysis of SGM-8 and SGM-9 Off-gas Line Deposits

The SGM-8 deposits were found in the same section of the off-gas system as the SGM-7 deposits (Figures 1 and 2). This section of the pipe had been cleaned after SGM-7 and dry feed (165 black frit) had been melted during Campaign 8.[11] The chemistry of the deposits were similar to those of SGM-7 but the Cl concentrations were significantly higher (Table 2). The SO_4 concentrations were somewhat lower than those measured for the SGM-7 deposits but the fluoride concentrations were the same as the concentrations observed in the SGM-7 deposits.

The major phases identified by x-ray diffraction analysis were (Na,K)Cl, $(Na,K)_2SO_4$ and stainless steel (Table 3) supporting the results of the chemical analyses (Table 2). Other phases present were $(Na_{0.5}K_{0.5})B_4O_7$, spinel, and Fe_2O_3. Only minor traces (~ 0.2 mole%) of KBF_4 were present as noted in the SGM-7 deposit analysis. The molar mass balance given in Appendix II and then summarized in Table 2 predicts that the salts compose ~ 35 mole% of the deposit, while about 23% is $(Na_{0.5}K_{0.5})B_4O_7$ and ~40 percent is oxides. The oxide sum appears to include some contribution from the stainless steel which probably adhered to the deposits during sampling.

The SGM-9 off-gas deposits were found further along the off-gas line (Figures 1 and 2) between the off-gas header and the isolation valve. These deposits had Cl and F concentrations equivalent to those found in SGM-7 but were extremely enriched in SO4 (Table 2). The major phases identified by x-ray diffraction were $((Na,K)_2SO_4$ (Na,K)Cl, and stainless steel (Table 3) supporting the results of the chemical analysis. Entrained Fe_2O_3, spinel, and sulfur were also identified by x-ray diffraction. Entrained spheres of Si rich glass frit (Figure 10) were observed in scanning electron microscopy and identified by use of the chemical EDAX spectra associated with scanning electron microscopy analysis.

Phase Distribution for SGM-7/SGM-9 Off-gas Line Deposits

The relative percentage of the $(Na,K)_2SO_4$ and the (Na,K)Cl salts found in the SGM-7 and SGM-9 off-gas deposits (based on the normalized mole% in Table 2) were superimposed on a schematic of their relative positions along the off-gas pipe (Figure 11). A similar schematic was made for the borate phases, primarily $(Na,K)_2B_4O_7$ with a minor (0.14-0.65 mole%) contribution from $(Na,K)BF_4$, the chromate phase, and the oxide phases (Figure 11). Deposits from SGM-8 were omitted since these occurred in the same location as the deposits in SGM-7 but formed from a dry feed run.

From the schematics shown in Figure 11 it is readily apparent that the major salt deposits form further from the off-gas entrance while the major entrained oxides (glass, spinel, Fe_2O_3, SiO_2) are relatively constant regardless of position along the off-gas pipe. The chromate phase appears to be concentrated higher in the off-gas system line as well but the distribution is somewhat erratic. This may in part be due to contributions of Cr in the deposits from adhering Inconel 690 rather than due to vapor phase transport of Cr from the melt.

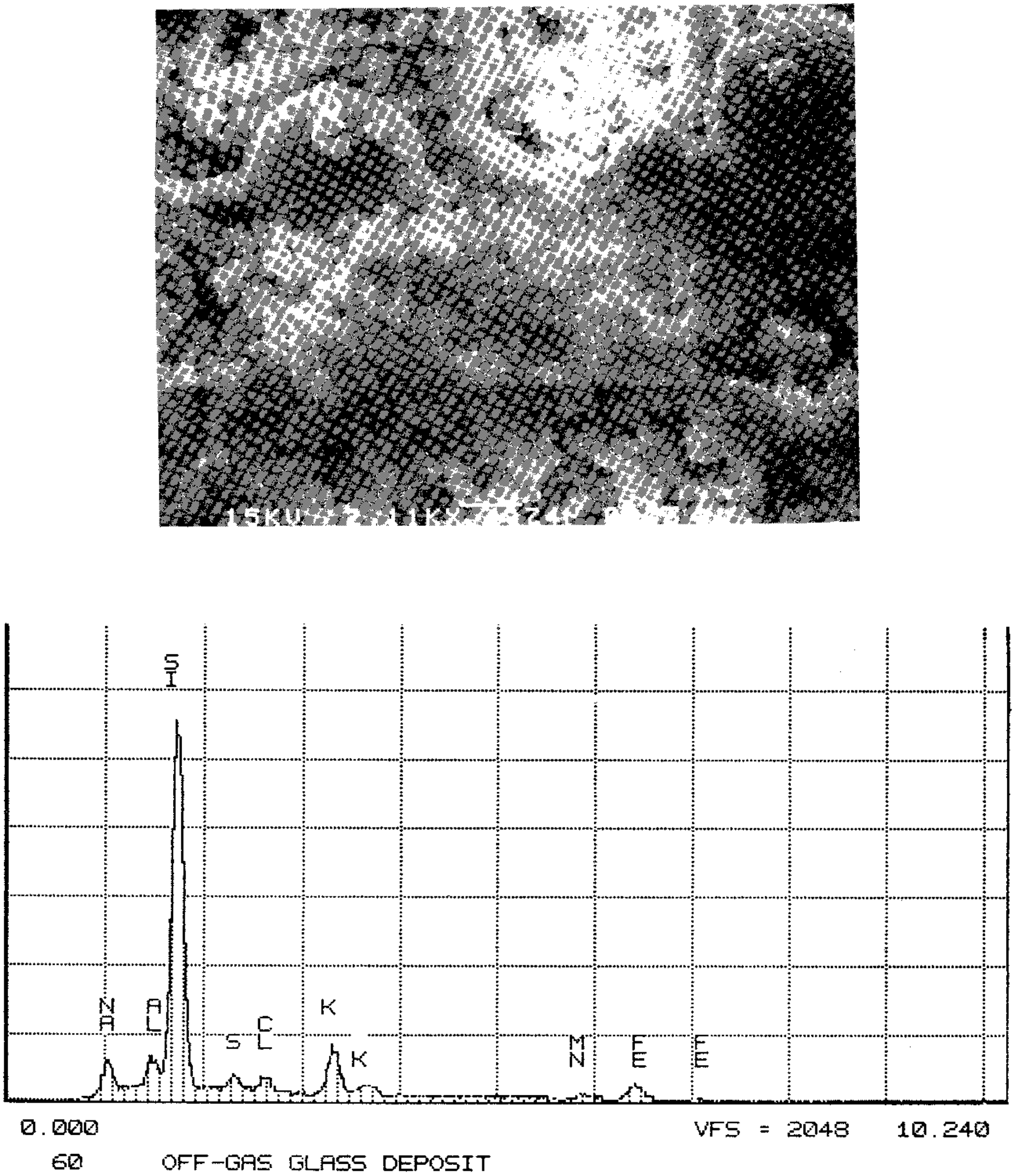

Figure 10.

Scanning Electron Micrograph of a Glass Frit Particle in the SGM-9 Off-Gas System Deposits. Note the Si Rich Chemical Spectra Associated with this Particle Which Enabled Its Identification.

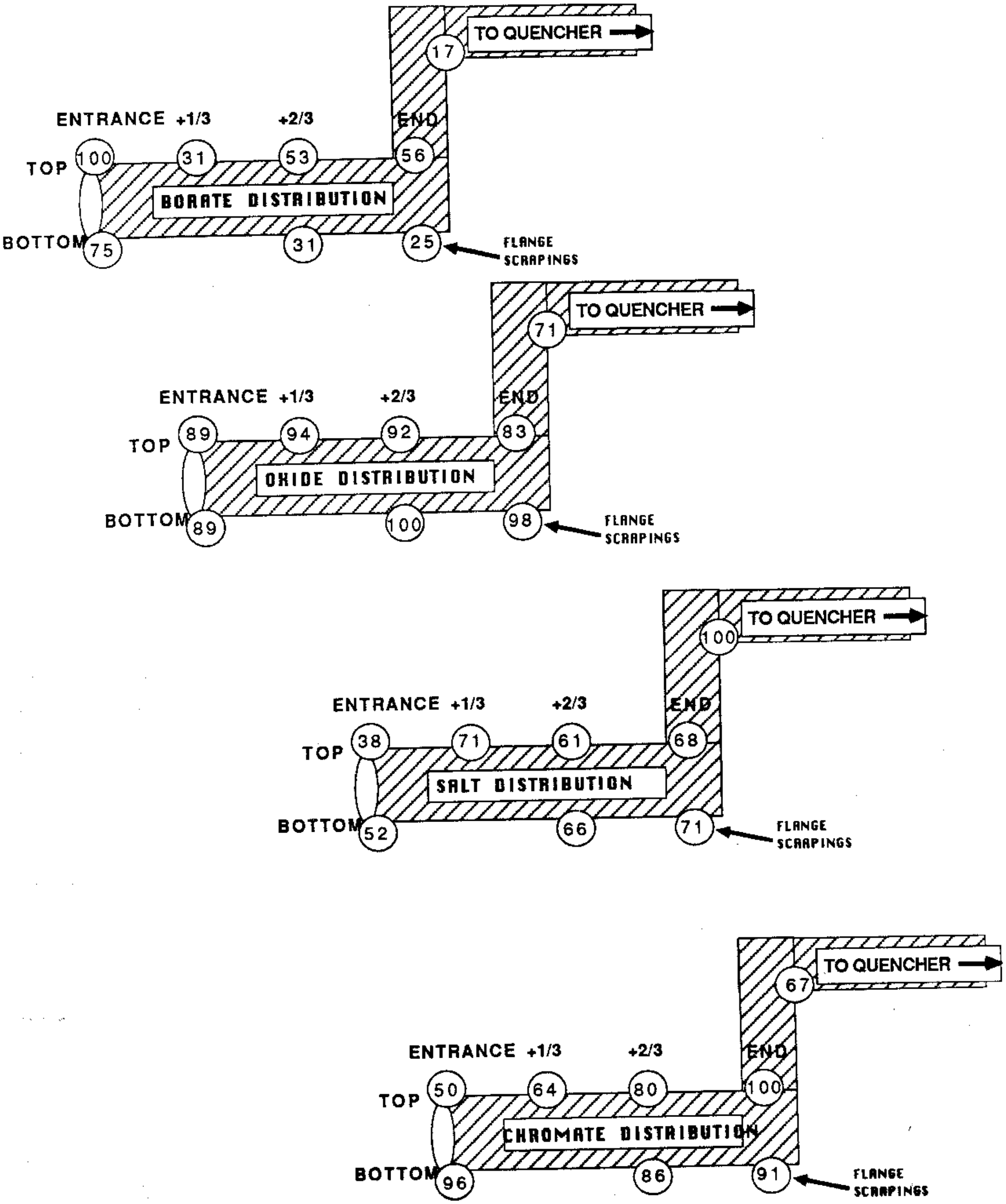

Figure 11.

Off-Gas System Schematic Distribution of the Relative % Salt (Halide Plus Sulfate), Oxide, Chromate, and Borate Phases from the SGM-7 and SGM-9 Off-Gas Pluggages.

The borate phases are concentrated at the off-gas system entrance and primarily along the top surfaces of the pipe. This is especially noticeable in the lack of borate phases in the portions of the off-gas line furthest from the entrance, e.g. in the vicinity of the SGM-9 deposits. Concomitantly, the chlorides and sulfates are at their maximum concentration along this portion of the line.

The phase distribution indicates that the alkali halides, chromates, and borates form by vapor phase transport and subsequent condensation. The borate phases, having a higher vapor pressure, condense lower in the off-gas line than the chlorides, fluorides, sulfates, and chromates. The oxide species are distributed evenly along the off-gas line and hence are deposited by entrainment and cemented in place by the condensing vapor species.

Identification of the "Low-Melting" Phases in the SGM-7 and SGM-8 Off-gas Deposits

Samples of SGM-7 and SGM-8 off-gas deposits were heat treated from 250°C to 800°C in 50°C intervals. Starting at 700°C, a yellow liquid phase separated from the remaining solid black deposits (Figure 12). The yellow deposits were completely liquid at 750°C.

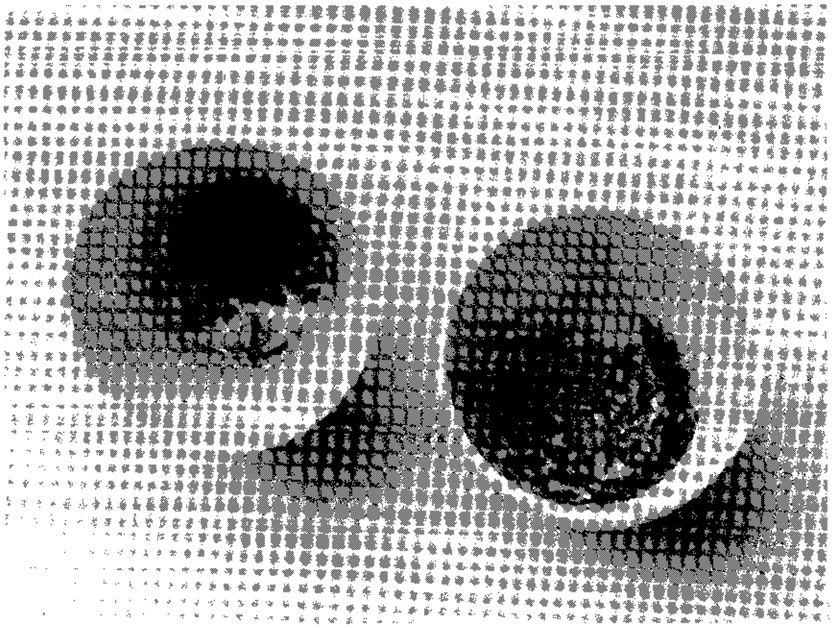

Figure 12.

SGM-7 Off-Gas Pluggage Melted at 750°C Showing the Formation of a Low-Melting Yellow Salt Deposit Which Covers the Unmelted Spinel and Frit Beneath.

A search was made of binary, ternary, and quaternary salt systems in order to determine whether the salt phases identified by x-ray diffraction were predicted to be molten at 700-750°C. Of the systems containing boron, those containing $Na_2B_4O_7$ with excess boron or fluoride melt between 680-700°C (Table 4). Of the systems containing SO_4, the binary mixtures of Na_2SO_4 and K_2SO_4 did not melt until 845°C. However, mixed (Na,K)Cl melts at ~700°C as did mixed alkali sulfates and chlorides (Table 4). The low melting compounds in the SGM off-gas deposits are, therefore, determined to be $Na_2B_4O_7$ and mixed $(Na,K)_2SO_4$-(Na,K)Cl. It should be noted that Cs, Li, and Ca can substitute for Na and K in these solid solutions and alter the melt temperature.

Identification of the Water-Soluble Phases in the SGM Off-gas Deposits

A hard piece of the SGM-7 pluggage material was put in deionized water in an ultrasonic bath at room temperature for ~5 minutes. The cementitious portion of the material was water soluble and rapidly left a residue of dispersed black solid particles. The water was decanted and found to be highly enriched in SO_4, Na, K, Cl, Cs, Li, Ca, B and Cr. The SO_4 was the most abundant species. If this is assumed to be ~50% of the soluble portion of the sample, then the remaining soluble species are 15, 8, 3, 1, 1, 1, 1, and 1/2% respectively. Fluoride was <0.01% of the soluble species compared to the soluble SO_4 species.

The dispersed black solid particles were dried and sent for x-ray analysis. Spinel of the $NiFe_2O_4$ type, Fe_2O_3, and pure SiO_2 were the main components of the insoluble particles (Table 3). Some residual NaCl and Na_2SO_4 were also present.

Analysis of Other SGM deposits

Cyclone Separator Deposits

After SGM-7, the cyclone separator became inoperable due to a buildup of "hair" like fibers (Figure 7). These deposits were extremely enriched in Cl and F over SO_4 and higher in Cs than the SGM off-gas deposits (Table 2). X-ray identification indicated that the major species present were NaCl, Na_2SO_4 and KBF_4 (Table 3).

Some of the "hair" deposit was put in deionized water in an ultrasonic bath at room temperature for ~5 minutes. The cementitious portion of the material was water soluble as observed with the SGM-7 off-gas deposits. The water was decanted and found to be highly enriched in Na, Cl, K, SO_4, B, F, Cs, Li, and Cr. The Na and Cl were the most abundant species. If Na and Cl are assumed to be ~50% of the soluble portion of the sample then the remaining soluble species are present at 14, 11, 7, 4, 1.5, 1.5, and 1% respectively.

The residue was dried and sent for x-ray analysis. SiO_2 was the main component of the insoluble material.

Table 4.
Lowest Melting Temperatures for Mixed Molten Salt Systems

System	Lowest Melt Temperature (°C)	Figure # in Phase Diagrams for Ceramists
Containing Borate and/or Fluoride		
$K_2(BO_2)_2$-$Na_2(BO_2)_2$-Na_2CrO_4-K_2CrO_4	774	790
$Na_2B_4O_7$-B_2O_3*	**730**	**4283**
NaF-$Na_2B_4O_7$*	**680**	**1688**
K_2O-Na_2O-B_2O_3-CrO_3	664	2603
KSO_4-Li_2SO_4-KBO_2-$LiBO_2$	570	1162
KBF_4-KCl	450	1638
KBF_4-$NaBF_4$	370, 398	1537,5807
Containing Sulfate and/or Chloride and/or Fluoride		
Na_2SO_4-K_2SO_4	845, 823	845,2889
NaCl-KCl*	**700,645**	**1258-9, 4749**
KCl-K_2SO_4*	**690**	**3834**
Na_2CrO_4-NaF	650	1691
NaCl-NaF-Na_2SO_4	624	1782
Li_2SO_4-K_2SO_4-Cs_2SO_4	616	2906
Na_2SO_4-KCl	540	6063
Cs_2SO_4-Li_2SO_4-Na_2SO_4	532	2907

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*Systems indicated in bold are those which melt at ~700°C

Quencher Nozzle

Due to insufficient sample, the quencher nozzle deposits from SGM-9 could only be analyzed by x-ray diffraction and SEM. Black and white colored deposits were analyzed separately. The SEM analyses indicated that the black deposits were enriched in Si, Al, Fe, and Cl but significant amounts of Mn, Ti, Na, K, S, and Mg were also present. The x-ray diffraction analyses indicated that an Fe-Ti spinel (JCPDS 31-645), SiO_2 (JCPDS 11-691) and mixed (Na,K)Cl were present. This phase identification is consistent with the elemental species identified during SEM analysis. The SEM analyses indicated that the white deposits were enriched primarily in Cl with some Ca and Si present. The x-ray diffraction analyses was complicated by the overlapping spectra of several calcium silicate phases and $CaCO_3$. The x-ray identification of the white deposits could not be definitively determined but is either $CaCO_3$ (JCPDS 5-586), $CaSiO_3$ (JCPDS 29-572), or $Ca_{10}Si_6O_{21}Cl_2$ (JCPDS 35-239). Based on the SEM-EDAX qualitative chemical analysis, the latter is the most probable.

HEME Spray Nozzle

Due to insufficient sample, the HEME spray nozzle deposits from SGM-9 could only be analyzed by SEM. The micrographs indicated that the deposit was composed of glass fibers (Figure 13 top). The qualitative SEM-EDAX analysis indicated that the deposits are Al, Si rich (Figure 13 bottom) glass and are probably fibers of the borosilicate glass which composes the HEME filters.[21] Boron is too light an element to be detected by EDAX analysis so the absence of B in the EDAX spectra is not inconsistent with the identification of these fibers as HEME borosilicate glass.

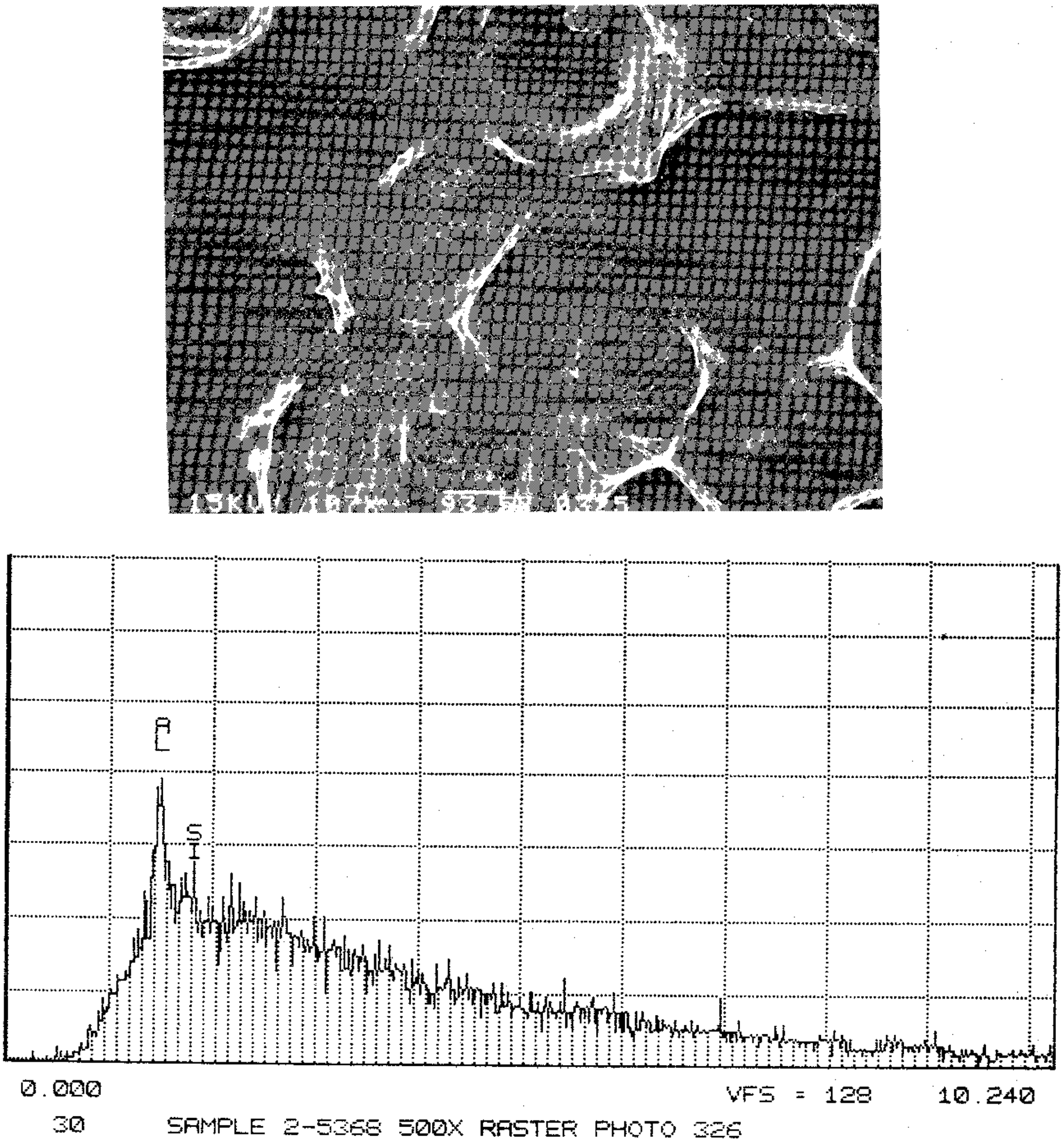

Figure 13.

Scanning electron micrograph of HEME filter borosilicate glass fibers which formed the HEME spray nozzle deposits (TOP). The Energy Dispersive Analysis by X-ray (EDAX) spectrum showing the main chemical constituents of these deposits (BOTTOM). Note that EDAX analysis cannot detect light elements like boron and lithium.

Condensate Tank Deposits

The sample taken from the SGM condensate tank was composed of a liquid and solid fraction. The liquid fraction was filtered and sent for chemical analysis. The liquid fraction contained 478 ppm Na, 35 ppm B, 7 ppm Ca, 5.87 ppm K, 2.5 ppm Li, 1.7 ppm Mg, 3 ppm P, and 4 ppm Si. All the other elements were present at < 1 ppm. Cs was detected at 0.014 ppm.

The solid portion of the condensate tank deposits were air dried and sent for SEM and x-ray analysis. The x-ray analysis indicated that spinel and SiO_2 were the major components of the condensate tank solids. This was confirmed by the SEM-EDAX analysis which demonstrated that the deposits were rich in Si and the spinel forming elements Al, Fe, Mn, Ni. Spherical glass frit particles were observed in the SEM (Figure 14) similar to those observed in the SGM-9 deposits. Therefore, entrained spinel and frit are being carried from the melter plenum all the way to the condensate tank.

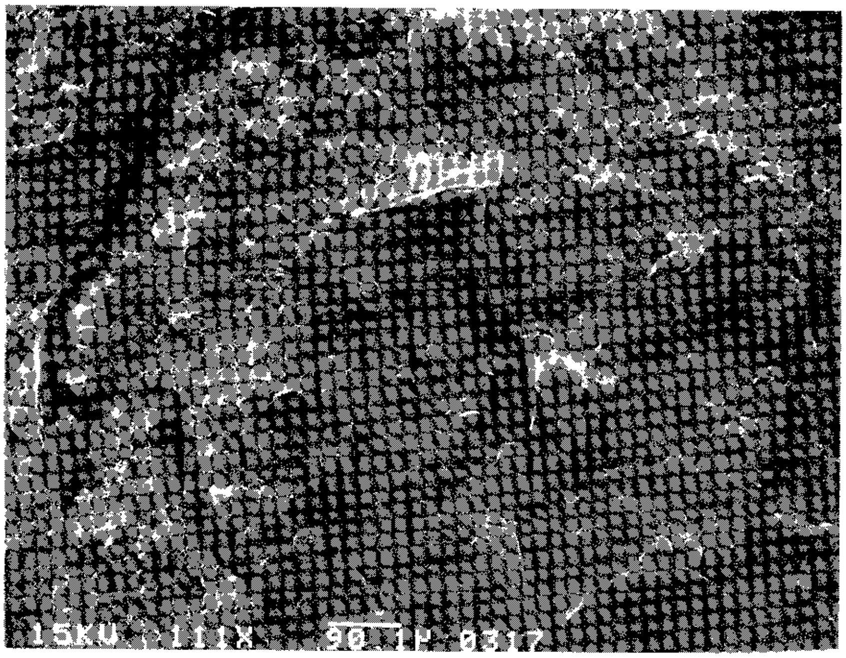

Figure 14.

Spherical Glass Frit Particle Filtered from the Solid Portion of the SGM Condensate.

Analysis of Glass Technology's Mini-melter Off-gas Line Deposits

Samples of off-gas deposits from the Glass Technology Group's (GTG) 1/100th scale minimelter in 774-A were analyzed chemically, and by x-ray diffraction. The minimelter deposits contained about the same quantities of Cl as the SGM deposits but significantly more F and significantly less SO_4 (Table 2). Correspondingly, the x-ray diffraction analysis indicated that NaCl and KCl were the major crystalline phases present but $(Na,K)SO_4$ was present in trace quantities (Table 3). Entrained spinel and Fe_2O_3 were identified in the x-ray spectra as well as $Na_2B_4O_7$. In general, the minimelter deposit chemistry is similar to that of the SGM deposits except for the high F concentration which is an order of magnitude higher than that determined in the SGM off-gas deposits.

Analysis of IDMS Off-gas Line Deposits

Samples of IDMS off-gas pipe deposits removed from close to the exit of the off-gas film cooler after Campaign #1 Run #2 were analyzed. During this campaign, sludge only 165 glass had been melted.[22] Loose deposits as well as deposits which were stuck on the pipe were analyzed chemically and by x-ray diffraction and SEM. The chemical analysis indicated about the same level of chloride as found in the SGM runs but the F concentrations were an order of magnitude higher than those found in SGM (Table 2). Correspondingly, the SO_4 concentrations were lower. The high F and low SO_4 anion distribution is similar to that determined for the GTG minimelter rather than to the deposits in SGM. The morphology of the IDMS deposits differs from that of the SGM deposits as well. When Figure 9 is compared with Figure 15 (top), the IDMS deposits appear looser and more colliform in shape. The SEM analysis revealed enrichment in Na, Cl, S, Ca, Cr, Mn, Ni, and Fe (Figure 15 bottom)

The x-ray diffraction analysis indicated that the major deposit in the IDMS "loose" deposits were entrained spinel. The major deposits in the IDMS "stuck" deposits were NaCl, Na_2SO_4, and stainless steel. The "stuck" deposits also contained significant amounts of $Na_2B_4O_7$, Na_2CrO_4, KBF_4, and sulfur as well.

Additional off-gas line pluggage samples were removed from the IDMS after running with coupled feed (Campaign #2 Run #3). Samples were taken from locations near the OGFC entrance (Deposit #1), below the OGFC brush (Deposit #2), and in the horizontal section of the pipe between the OGFC and the inspection Port (Deposit #3-see Figure 4). These samples, taken from near the off-gas line entrance, were black in color. Thin white deposits were observed further along the off-gas line but were not sampled.[23] Deposits #2 and #3 were enriched in Na, K, Cs, Cr, Fe, Cl, F, and SO_4 (Table 2). Much smaller enrichments in Li and B were observed. These are the same species that materials balance calculations indicate have vaporized from the IDMS melt pool.[24] The F concentrations remained an order of magnitude higher than those observed in SGM as in the previous set of IDMS off-gas deposits. There was insufficient sample of Deposit #1 to do a complete wet chemical analysis but this sample did not show the enrichment of Fe as did the remaining samples.

The XRD analysis of the IDMS Campaign #2 Run #3 samples indicated that the major species were NaCl, KCl, Na_2SO_4, and entrained spinel (Table 3). Likewise the SEM analysis confirmed that Na, K, Cs, Cl, and SO_4 were the major species present (Figure 16 top). For the first time a wide angle detector was used on the SEM-EDAX and the presence of B was confirmed in these deposits in the absence of other glass forming components such as Fe and Si (compare Figure 16 top and bottom). This is confirmatory to the presence of amorphous or poorly crystalline $Na_2B_4O_7$ which would not be observed in the XRD analysis (Table 3) but was predicted from the molar mass balance calculations (Table 2).

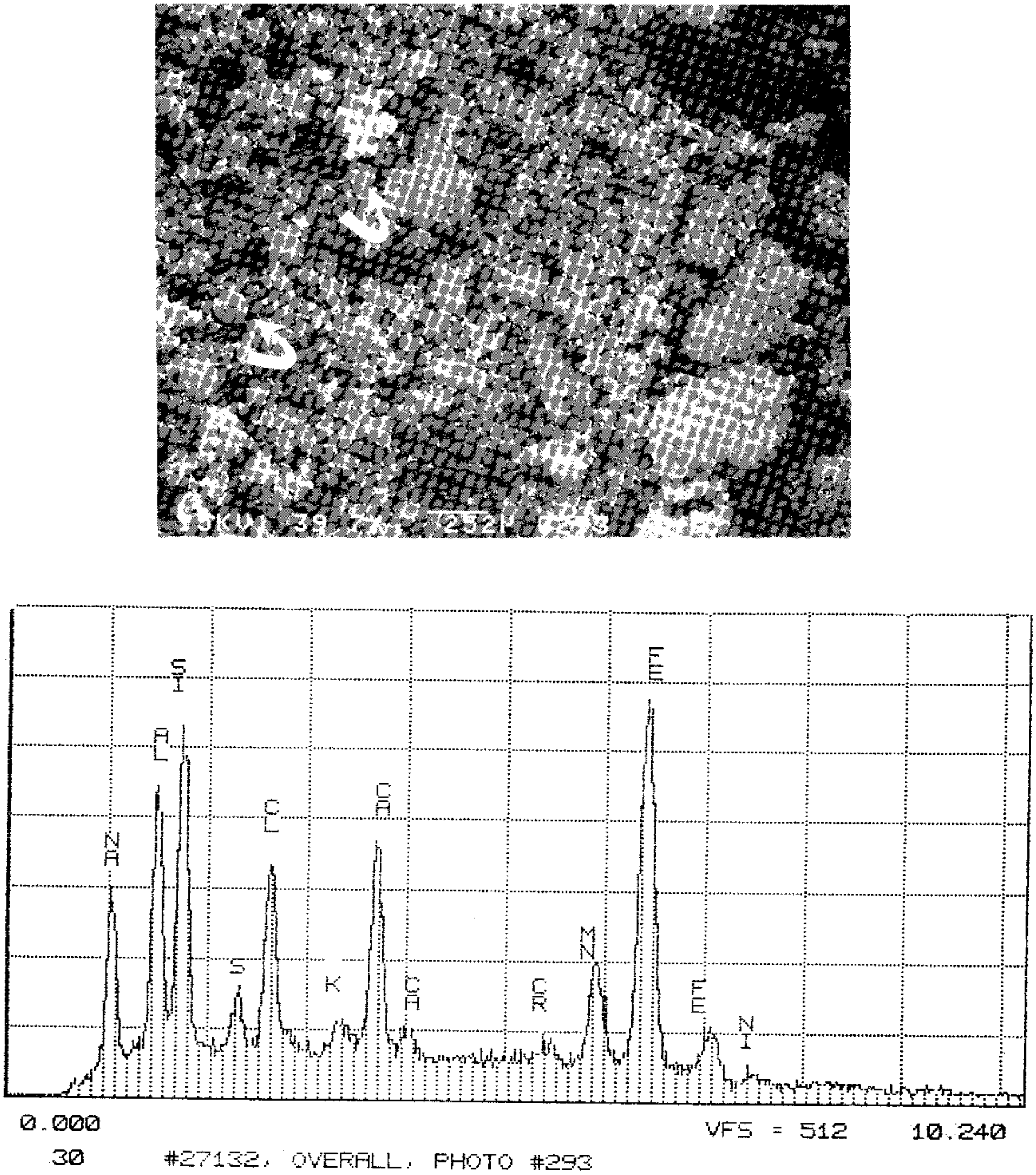

Figure 15.

Morphology of "Loose" IDMS Off-Gas System Deposits (Top). Note the Looser Colliform Shape of the Deposits Compared to the SGM Deposits (Figure 9). Note the White Arrows Point to Entrained Frit Particles. The Elemental Chemistry of the "Loose" Deposits is Shown in the EDAX Spectra (Bottom).

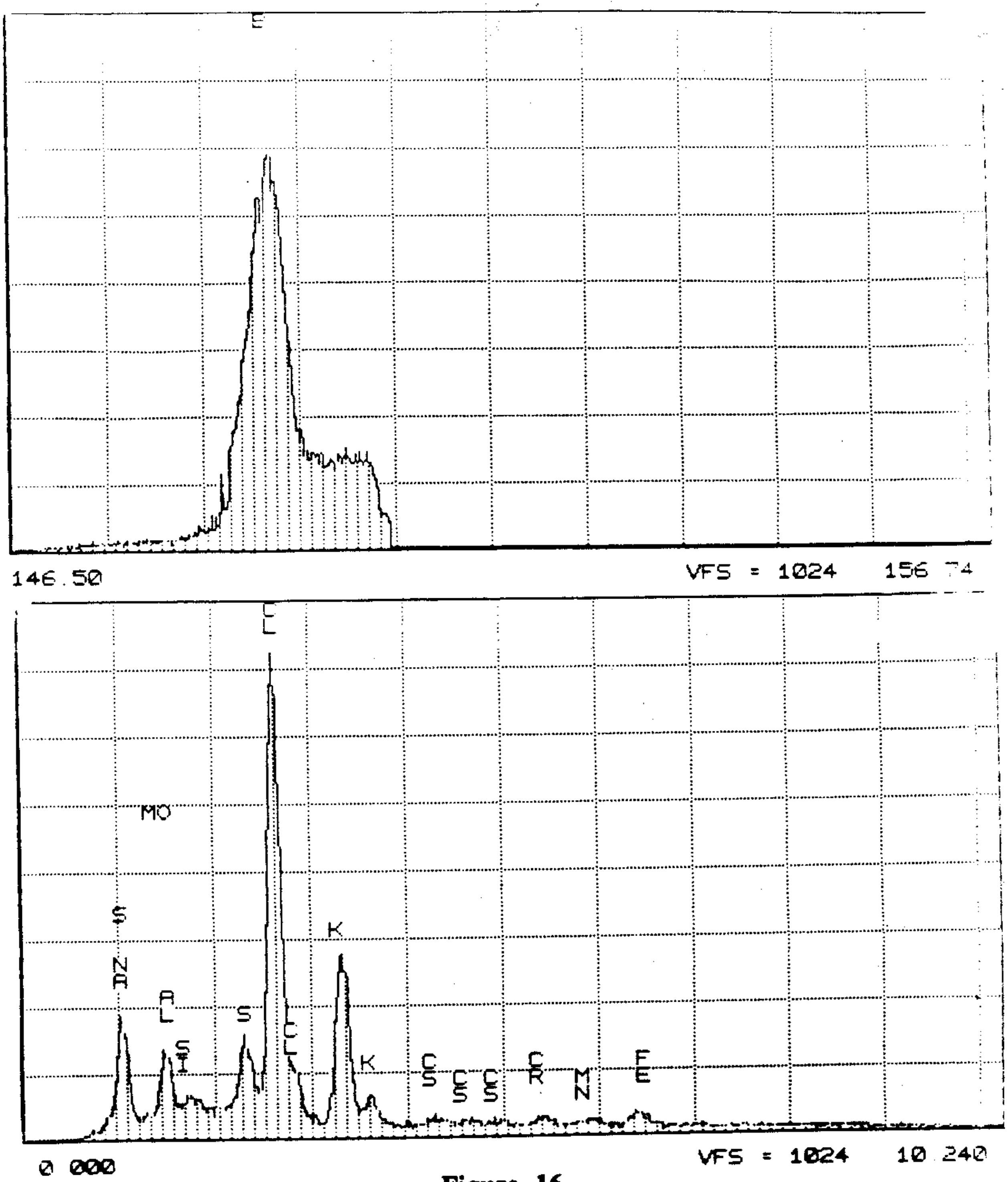

Figure 16.

SEM-EDAX Spectra of IDMS Campaign #2 Run #3 Deposits From Near the OGFC Entrance (Top). Compared to the Analysis of Campaign #1 Run #2 Deposits Given in Figure 15, This Sample is Greatly Enriched in Alkali, Chloride and Sulfate and Not in Splattered or Entrained Glass Correspondingly, the Bottom Wide Angle SEM-EDAX Indicates the Presence of Boron in the Absence of Other Entrained Glass Species Such as Fe and Si.

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DISCUSSION

Deposition Mechanisms

Similarity to Commercial Borosilicate Melters

Sodium borate, sodium sulfate, sodium chloride, and sodium fluoride deposits have been observed to form in commercial melters by condensation.[25-28] During melting of sodium borosilicate glass in commercial melters, sodium borate deposits occur in the middle sections of glass regenerators while the sodium sulfate and sodium chloride deposits form in the higher sections.[26-27] Potassium[25] and rubidium[28] behave similarly to sodium but condense at somewhat lower temperatures.

The mode of formation of borate and sulfate deposits in commercial melters has been extensively studied.[25-28] When melting soda-lime-silica (flat) glass, gaseous Na_2SO_4, NaOH, and NaCl have been identified at temperatures between 727°C and 1527°C.[29] Gaseous $NaBO_2$, $Na_2(BO_2)_2$, and $Na,Rb(BO_2)_2$ have been observed by mass spectrometric techniques over sodium borosilicate glass melts at temperatures between 533-880°C.[28] Condensation of salt components or metal oxides on the cold channel surfaces or on regenerator bricks has been defined as the operative mechanism of deposit formation at >627°C during borosilicate glass melting.[26-27] Sodium, sulfur, and chloride-containing molecules are vapor phase transported toward the deposition surface. At the cold surface chemical reactions and condensation occur, causing a growing deposition layer of Na_2SO_4 and/or NaCl, NaF, and $Na_2B_4O_7$. Deposition of fine particles entrained from earlier condensation reactions is the operative mechanism of deposit formation at <527°C.[26-27] The condensation mechanisms have been modeled based on thermodynamics and boundary layer transport. The models have been validated for commercial borosilicate melters based on both laboratory experiments and melter experience.[26]

Similarity to Other Nuclear Waste Glass Melters

Alkali metaborates have been observed in the off-gas systems of melters at the Pacific Northwest Laboratory.[30] The mechanism of formation has been attributed to feed entrainment and volatilization/condensation.[31] The porosity of the off-gas system deposits have been attributed to revolatilization of condensed boron compounds.[30] Remediation measures have been taken to maintain velocities of 15 m/s (49.2 fps) in the off-gas line to minimize solids deposition. Off-gas velocity was felt to be more effective in eliminating off-gas line deposition than off-gas temperature.[32] This is the same minimum velocity, 50 fps, recommended by Colven et al. during the LSFM campaigns.[4] These were not the velocities adhered to in the early SGM campaigns and in IDMS when off-gas line pluggages occurred.

Studies with High-Level Waste Glass (J-10) at the Japan Atomic Energy Research Institute on the vaporization of ^{137}Cs and ^{106}Ru were conducted in a closed steel canister which was used as both the glass melting and annealing chamber.[33] Cesium was found to vaporize as $CsBO_2$ and ruthenium as RuO_4. These volatile species were found to condense and react with the steel chamber in the plenum, again confirming that gaseous alkali borate phases volatilize when borosilicate glasses are melted and then condense on colder substrates.

Vaporization and Vapor Pressure Studies of SRP Glasses

Vaporization of the alkali species (Na, Cs, and Li) from SRL glasses was extensively studied by Wilds in 1978.[34] This study demonstrated that the ratio of (Na+Li)/B that vaporized from an Na and Li containing glass was ~1.0 while the ratio of Na/B in a non Li containing glass was also ~1.0. This indicated that the primary vapor species was $Li_2O \bullet B_2O_3$ and $Na_2O \bullet B_2O_3$. Increased vaporization of these alkali metaborates was observed with increased melt temperature, increased Na and Li concentrations in the glass, and increased time at temperature. No $Cs_2O \bullet B_2O_3$ was observed to vaporize and it was hypothesized that the Cs species vaporizing was gaseous Cs and CsO.

Vapor phase transport is directly related to the elemental and/or chemical vapor pressure properties of specific melter feed species. Work done on SRL 165 glass at the National Bureau of Standards in 1983 indicated that alkali metaborates were the dominant vapor species which would undergo vapor transport under the reduced pressure conditions expected during DWPF processing.[35-36] Mass spectrometric analyses indicated that gaseous Na, Li, Cs, O_2, $NaBO_2$, and $LiBO_2$ were present above the melt at temperatures between 800-1150°C. The 165 glass did not contain K_2O and so KBO_2 was not observed, but is predicted to form similarly to $NaBO_2$ and $LiBO_2$. In the presence of water vapor, which would be present in steam from the OGFC and/or from slurry feeding in DWPF, HBO_2 was the principal gaseous species detected. It was concluded that the off-gas atmosphere was of secondary importance to the condensed phase composition in controlling vapor transport since SO_4, Cl, and F were absent in the glass studied. The thermodynamic activity of boron oxide in the glass is the major glass species controlling metaborate vapor transport.

Summary of Parameters of Deposition/Non-Deposition

During LSFM operation, off-gas line temperatures between 350-450°C were recommended to stop adhesion of partially melted off-gas particulates.[1-9] Colven, and others[3,4] indicated that both temperature control and off-gas velocities of 50 fps were necessary during LSFM to stop off-gas system deposit buildup. During LSFM-7 and -9 when high steam velocities of 60 fps were used, no deposits were found (Table 5).[4,7]

During SGM Campaigns 7, 8, and 9 off-gas pluggages occurred. The off-gas temperatures measured beyond the off-gas film cooler during SGM-6,-7, and -8 were in excess of 450°C.[10,11] The highest temperatures were observed during the dry feed run (SGM-8) which also had the most significant off-gas pluggages.[12] Pluggage had been partially attributed to the excursions above 400°C and the off-gas velocities were not reported.[11]

Partial and complete removal of the SGM off-gas deposits were found to occur during surges in off-gas flow, indicating that high velocity is an important mechanism by which deposits can be partially removed. The temperature surges in the off-gas line during SGM-6 and SGM-7 were found to correspond to the initiation of melter feeding.[10] The temperature rise was attributed to the effect of the steam generated by slurry feeding, which cools the melter plenum but heats the off-gas line. The apparent cyclic nature of the SGM (Figure 9) melter deposits may, therefore, be due to periods when the melter was run and off-gas line temperatures were higher so that vapor phase transport was more active.

The last part of the SGM-9 campaign was run in a temperature control mode at 350 ± 25°C by addition of steam (Table 5).[12,14] Off-gas velocities were not reported but were later calculated to be 70 ± 5 fps.[15] During IDMS operation in the temperature control mode (350 ± 25°C) off-gas deposits accumulated. The off-gas velocity was only 40 fps (Table 5).

Experience with waste glass melters run at PNL has suggested that the minimum velocity recommended by SRL of 50 fps is more important than temperature in controlling off-gas deposition.[30] West Valley Nuclear Fuel Services (WVNFS) have never experienced off-gas line pluggages in the jumper section of their off-gas line. They attribute the lack of pluggage to never allowing their off-gas line velocity to fall below the 50 fps recommended by SRL in the early 1980s (Table 5).[37]

In summary, the critical off-gas line operating parameter appears to be velocity (Table 5). Although the temperatures of the LSFM, SGM, PNL, and WVNFS melters varied, all have remained free of off-gas line deposition when the off-gas line velocity was >50 fps (Table 5).

Table 5.

Summary of Conditions of Off-gas System Deposition and Non-Deposition.

Melter	Velocity (fps)	Steam	Temp. (°C)	Off-gas Deposits
LSFM-7	60	Yes	350-450	No
LSFM-9	60	Yes	350-450	No
SGM 6,7,8	not reported	No	450	Yes
SGM-9	70±5	Yes	325 ± 25	No
IDMS	40	No	325 ± 25	Yes
PNL	>50	?	?	No
WVNFS	>50	?	?	Never

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CONCLUSIONS

The chemistry and distribution of the alkali phases found in the SGM and IDMS off-gas deposits is the same as that found in commercial borosilicate glass melters. Sodium borate phases deposit closer to the off-gas line entrance while the more volatile alkali sulfates, chlorides, and fluorides form at greater distances from the entrance. The mode of formation is vapor phase transport and condensation.

Deposition by vapor phase transport indicates that alkali species, B, SO_4, and Cl form alkali metaborates and salts which act as the cementitious portion of the off-gas deposits. The alkali rich deposits condense and cement the entrained spinel and spherical frit particles to the off-gas line.

The chemistry and distribution of phases in the SGM and IDMS off-gas systems is inconsistent with the hypothesis put forth by Colven and others[3,4] that off-gas line pluggages are caused by adhesion of partially melted particulates to insufficiently cooled off-gas system pipes. The remediation suggested,[12] to operate the off-gas system in a temperature control mode, e.g. 350 ± 25°C, was determined by choosing the lowest melt temperature of any constituent of the off-gas system deposits. The lowest melting constituents were $NaBF_4$ and KBF_4. However, there is insufficient F present in the off-gas system deposits to form significant amounts of $NaBF_4$ and KBF_4 to act as the cementitious portion of the deposits.

Since the operative mechanism is vapor phase transport and condensation, cooling of the off-gas line[3,4,12] is not the correct remediation for control of off-gas deposition. Ideally, hotter off-gas line temperatures would retain the alkali compounds as vapors until they reach the quencher. Hotter off-gas line temperatures would necessitate redesign of the quencher to take the increased heat load and would limit the off-gas velocity which cools the off-gas system.

The identification of vapor phase transport as the operational mechanism causing off-gas system pluggages beyond the entrance, indicates that deposition can be effectively controlled by high velocity air and/or steam. Velocities higher than 50 fps are necessary to transport the volatile species to the quencher rather than having them condense in the off-gas line. Velocity is, therefore, considered a more important criterion for controlling off-gas pluggage than temperature control.

The DWPF melter off-gas velocity design basis[38] is 47 fps and the velocities at 350-450°C are calculated to be between 43-46 fps.[39] It is, therefore, recommended that DWPF design velocities be increased to >50 fps by increasing the air/steam flow entering the off-gas film cooler (OGFC) entrance, by reducing the pipe diameter, or by upgrading the process chiller capacity. Since the recommended process chiller design upgrade for DWPF[40] is based on keeping the off-gas system cool, it is recommended that the chiller design upgrade be re-evaluated based on a velocity criterion rather than on temperature control.

The cementitious alkali borates, halides, and sulfates comprising the off-gas line deposits were determined to be water soluble. If pluggages occur beyond the off-gas line entrance, then the deposits can be effectively removed with water and/or steam. It is recommended that removable sections of the off-gas pipe be water cleaned and that engineering designs be investigated to take advantage of the water soluble nature of the off-gas deposits so that the need for mechanical disassembly and cleaning of the highly radioactive (^{137}Cs and ^{106}Ru) off-gas line could be eliminated in DWPF.

SUMMARY OF RECOMMENDATIONS

Based on the results of the off-gas deposit analysis, the following are recommended:

- Re-evaluate the design basis for the recommended DWPF off-gas process chiller upgrade[40] based on a velocity criterion (>50 fps) rather than on temperature control.
- Re-design the DWPF off-gas system to increase the off-gas line velocities from the design basis of 47 fps to >50fps.
- Investigate water or steam cleaning of off-gas line deposits from the removable jumper section of the off-gas line during the remaining IDMS campaigns and/or the DWPF Cold Runs
- Investigate engineering designs to take advantage of the water soluble nature of the off-gas deposits to eliminate mechanical disassembly and cleaning of the off-gas line jumper section. For example, an auxiliary off-gas line could be used while cleaning the plugged line in situ with hot water. The alkali rich hot water could be recycled to the melt pool in a manner similar to slurry feeding or recycled to the Slurry Mix Evaporator.

ACKNOWLEDGEMENTS

Paul D. Guidotti and Jeff A. Voogd requested the analyses of the SGM-7 and SGM-8 off-gas deposits. The analyses from SGM-9, the spray nozzle, quencher nozzle, and condensate tank were requested by Glenn F. Rabon. The analysis of the IDMS samples were requested by Don Miller who also supplied the calculated DWPF off-gas velocities. Alex S. Choi calculated the off-gas velocity used during the SGM-9 campaign. Nick D. Hutson provided the IDMS material balance which indicated which species had volatilized during melting. Dennis F. Bickford is gratefully acknowledged for his helpful discussions regarding cold cap and melter chemistry.

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APPENDIX I

Model Mass Balance Calculation

	A	B	C	D	E	F
1						
2	SAMPLE ID=	104228/OFFGAS PLUG ENTRANCE TOP				
3						
4		ELEMENT	GRAV	OXIDE		
5		WT%	FACTOR	WT%		
6	Al2O3	3.29	1.8895	=B6*C6		
7	CaO	1.7	1.3992	=B7*C7		
8	Fe2O3	18 9	1.4297	=B8*C8		
9	MgO	0.209	1.6583	=B9*C9		
10	MnO	0.989	1.2912	=B10*C10		
11	Na2O	12 18	1.348	=((B42/2)*22.98)*C11		
12	Li2O	0.888	2.1525	=B12*C12		
13	NiO	1.55	1.2726	=B13*C13		
14	SiO2	2.56	2.1393	=B14*C14		
15	Cr2O3	0 2837	1.4616	=B15*C15		
16	B2O3	7	3.2201	=(B46*10 81)*C16		
17	SrO	0.1178	1.1826	=B17*C17		
18	ZrO2	0.12	1.3508	=B18*C18		
19	TiO2	0 03	1.668	=B19*C19		
20	K2O	6 9256	1.2046	=((B42/2)*39 1)*C20		
21	Cs2O	0 9813	1.0602	=B21*C21		
22						
23	Cl	4.2949				
24	F	0.1169				
25	SO4	9 07				
26						
27	SUMS			=SUM(D6:D22)		
28						NORMALIZED
29	MOLAR BALANCE (M)			MOLAR SUMS	MOLE %	MOLE %
30						
31	(Na,K)2SO4	=(B25/96 08)*((174.26+142.02)/2)		SALTS	=SUM(B31:B33)	=(E31/E36)*100
32	(Na,K)Cl	=(B23/35 45)*((58.43+74.5)/2)		BORATES	=B34	=(E32/E36)*100
33	(Na,K)BF4	=((B24/19)/4)*((125.91+109.79)/2)		CHROMATES	=B35	=(E33/E36)*100
34	(K,Na)2B4O7	=(((B16/10.81)-(B33/117.85))/4)*((201.21+233.42)/2)		OXIDES	=D27	=(E34/E36)*100
35	Na2CrO4	=(B15/52)*161.95				
36	OXIDES	=D27		TOTAL	=SUM(E31:E34)	=SUM(F31:F34)
37						
38	SUM	=SUM(B31:B36)				
39						
40	MOLES					
41	K,Na excess*	=((B20/39.1)+(B11/22.98))-((B25/96.06)*2)-(B23/35.45)-((B24/19)/4)				
42	K,Na excess**	=B41-((B34/217.32)*2)-((B35/161.95)*2)				
43	SO4 excess	=(B25/96.06)-(B31/158.14)				
44	Cl excess	=(B23/35.45)-(B32/66 47)				
45	F excess	=((B24/19)/4)-(B33/117.85)				
46	B excess	=(B16/10.81)-(B33/117.85)-((B34/217.31)*4)				
47						
48		* Excess after assignment with Cl, SO4 and F				
49		** Excess after assignment with Cr and B				

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APPENDIX II

Mass Balance Calculations for Off-gas Deposits

SAMPLE ID= 104226/OFFGAS PLUG ENTRANCE TOP

	ELEMENT WT%	GRAV FACTOR	OXIDE WT%
Al2O3	3.29	1.89	6.22
CaO	1.70	1.40	2.38
Fe2O3	18.90	1.43	27.02
MgO	0.21	1.66	0.35
MnO	0.99	1.29	1.28
Na2O	12.18	1.35	0.96
Li2O	0.69	2.15	1.48
NiO	1.55	1.27	1.97
SiO2	2.56	2.14	5.48
Cr2O3	0.28	1.46	0.41
B2O3	7.00	3.22	0.00
SrO	0.12	1.18	0.14
ZrO2	0.12	1.35	0.16
TiO2	0.03	1.67	0.05
K2O	6.93	1.20	1.45
Cs2O	0.98	1.06	1.04
Cl	4.29		
F	0.12		
SO4	9.07		
SUMS			50.39

MOLAR BALANCE (M)	
(Na,K)2SO4	14.93
(Na,K)Cl	8.05
(Na,K)BF4	0.18
(K,Na)2B4O7	35.10
Na2CrO4	0.88
OXIDES	50.39
SUM	109.53

MOLAR SUMS	MOLE %	NORMALIZED MOLE %
SALTS	23.17	21.15
BORATES	35.10	32.04
CHROMATES	0.88	0.81
OXIDES	50.39	46.00
TOTAL	109.53	100.00

MOLES	
K,Na excess*	**0.40**
K,Na excess**	**0.06**
SO4 excess	**0.00**
Cl excess	**0.00**
F excess	**0.00**
B excess	**0.00**

* Excess after assignment with Cl, SO4 and F

** Excess after assignment with Cr and B

APPENDIX II

Mass Balance Calculations for Off-gas Deposits

SAMPLE ID= 104227/OFFGAS PLUG ENTRANCE BOTTOM

	ELEMENT WT%	GRAV FACTOR	OXIDE WT%
Al2O3	3.32	1.89	6.27
CaO	3.49	1.40	4.88
Fe2O3	18.06	1.43	25.82
MgO	0.19	1.66	0.32
MnO	1.46	1.29	1.88
Na2O	13.18	1.35	0.45
Li2O	1.19	2.15	2.57
NiO	0.91	1.27	1.16
SiO2	4.21	2.14	9.01
Cr2O3	0.61	1.46	0.89
B2O3	5.73	3.22	0.00
SrO	0.12	1.18	0.14
ZrO2	0.02	1.35	0.03
TiO2	0.03	1.67	0.05
K2O	7.72	1.20	0.69
Cs2O	2.01	1.06	2.13
Cl	3.49		
F	0.13		
SO4	17.01		
SUMS			56.28

MOLAR BALANCE (M)	
(Na,K)2SO4	28.00
(Na,K)Cl	6.54
(Na,K)BF4	0.20
(K,Na)2B4O7	28.71
Na2CrO4	1.89
OXIDES	56.28
SUM	121.63

MOLAR SUMS	MOLE %	NORMALIZED MOLE %
SALTS	34.74	28.57
BORATES	28.71	23.61
CHROMATES	1.89	1.55
OXIDES	56.28	46.27
TOTAL	121.63	100.00

MOLES	
K,Na excess*	**0.32**
K,Na excess**	**0.03**
SO4 excess	**0.00**
Cl excess	**0.00**
F excess	**0.00**
B excess	**0.00**

* Excess after assignment with Cl, SO4 and F

** Excess after assignment with Cr and B

APPENDIX II

Mass Balance Calculations for Off-gas Deposits

SAMPLE ID= 104228/OFFGAS PLUG ±1/3 TOP

	ELEMENT WT%	GRAV FACTOR	OXIDE WT%
Al2O3	1.81	1.89	3.42
CaO	1.84	1.40	2.57
Fe2O3	14.00	1.43	20.02
MgO	0.11	1.66	0.19
MnO	3.11	1.29	4.02
Na2O	13.52	1.35	2.53
Li2O	0.54	2.15	1.16
NiO	1.34	1.27	1.71
SiO2	2.49	2.14	5.33
Cr2O3	0.31	1.46	0.46
B2O3	1.80	3.22	0.00
SrO	0.06	1.18	0.07
ZrO2	0.04	1.35	0.05
TiO2	0.21	1.67	0.35
K2O	6.77	1.20	3.85
Cs2O	0.73	1.06	0.77
Cl	4.08		
F	0.09		
SO4	18.58		
SUMS			46.49

MOLAR BALANCE (M)	
(Na,K)2SO4	30.59
(Na,K)Cl	7.66
(Na,K)BF4	0.14
(K,Na)2B4O7	8.98
Na2CrO4	0.97
OXIDES	46.49
SUM	94.83

MOLAR SUMS	MOLE %	NORMALIZED MOLE %
SALTS	38.38	40.48
BORATES	8.98	9.47
CHROMATES	0.97	1.03
OXIDES	46.49	49.02
TOTAL	94.83	100.00

MOLES	
K,Na excess*	**0.26**
K,Na excess**	**0.16**
SO4 excess	**0.00**
Cl excess	**0.00**
F excess	**0.00**
B excess	**0.00**

* Excess after assignment with Cl, SO4 and F

** Excess after assignment with Cr and B

APPENDIX II

Mass Balance Calculations for Off-gas Deposits

SAMPLE ID= 104229/OFFGAS PLUG VARIOUS SCRAPINGS-BOTTOM

	ELEMENT WT%	GRAV FACTOR	OXIDE WT%
Al2O3	1.70	1.89	3.21
CaO	1.79	1.40	2.50
Fe2O3	13.80	1.43	19.73
MgO	0.13	1.66	0.21
MnO	2.90	1.29	3.74
Na2O	13.46	1.35	1.44
Li2O	0.78	2.15	1.67
NiO	0.98	1.27	1.25
SiO2	3.47	2.14	7.42
Cr2O3	0.49	1.46	0.71
B2O3	3.56	3.22	0.00
SrO	0.04	1.18	0.05
ZrO2	0.06	1.35	0.08
TiO2	0.27	1.67	0.44
K2O	4.71	1.20	2.19
Cs2O	1.61	1.06	1.71
Cl	4.64		
F	0.42		
SO4	14.23		
SUMS			46.36

MOLAR BALANCE (M)	
(Na,K)2SO4	23.43
(Na,K)Cl	8.69
(Na,K)BF4	0.65
(K,Na)2B4O7	17.59
Na2CrO4	1.51
OXIDES	46.36
SUM	98.23

MOLAR SUMS	MOLE %	NORMALIZED MOLE %
SALTS	32.77	33.36
BORATES	17.59	17.91
CHROMATES	1.51	1.54
OXIDES	46.36	47.20
TOTAL	98.23	100.00

MOLES	
K,Na excess*	**0.27**
K,Na excess**	**0.09**
SO4 excess	**0.00**
Cl excess	**0.00**
F excess	**0.00**
B excess	**0.00**

* Excess after assignment with Cl, SO4 and F

** Excess after assignment with Cr and B

APPENDIX II

Mass Balance Calculations for Off-gas Deposits

SAMPLE ID= 104230/OFFGAS PLUG +2/3 TOP

	ELEMENT WT%	GRAV FACTOR	OXIDE WT%
Al2O3	1.79	1.89	3.38
CaO	1.82	1.40	2.55
Fe2O3	14.50	1.43	20.73
MgO	0.10	1.66	0.16
MnO	3.02	1.29	3.90
Na2O	13.93	1.35	2.32
Li2O	0.65	2.15	1.39
NiO	1.19	1.27	1.51
SiO2	2.56	2.14	5.48
Cr2O3	0.41	1.46	0.59
B2O3	3.34	3.22	0.00
SrO	0.05	1.18	0.06
ZrO2	0.04	1.35	0.06
TiO2	0.22	1.67	0.36
K2O	5.88	1.20	3.52
Cs2O	1.16	1.06	1.23
Cl	4.24		
F	0.11		
SO4	15.20		
SUMS			47.24

MOLAR BALANCE (M)

(Na,K)2SO4	25.02
(Na,K)Cl	7.95
(Na,K)BF4	0.18
(K,Na)2B4O7	16.70
Na2CrO4	1.26
OXIDES	47.24
SUM	98.35

MOLAR SUMS

SALTS	33.15	33.70
BORATES	16.70	16.98
CHROMATES	1.26	1.29
OXIDES	47.24	48.03
TOTAL	98.35	100.00

MOLES	
K,Na excess*	**0.32**
K,Na excess**	**0.15**
SO4 excess	**0.00**
Cl excess	**0.00**
F excess	**0.00**
B excess	**0.00**

* Excess after assignment with Cl, SO4 and F

** Excess after assignment with Cr and B

APPENDIX II

Mass Balance Calculations for Off-gas Deposits

SAMPLE ID= **104231/OFFGAS PLUG +2/3 BOTTOM**

	ELEMENT WT%	GRAV FACTOR	OXIDE WT%
Al2O3	1.60	1.89	3.02
CaO	1.64	1.40	2.29
Fe2O3	12.79	1.43	18.29
MgO	0.09	1.66	0.16
MnO	2.66	1.29	3.43
Na2O	12.58	1.35	3.24
Li2O	0.55	2.15	1.18
NiO	1.25	1.27	1.59
SiO2	2.65	2.14	5.67
Cr2O3	0.41	1.46	0.59
B2O3	1.80	3.22	0.00
SrO	0.04	1.18	0.05
ZrO2	0.05	1.35	0.06
TiO2	0.21	1.67	0.36
K2O	7.90	1.20	4.93
Cs2O	1.95	1.06	2.07
Cl	4.76		
F	0.15		
SO4	14.70		
SUMS			46.94

MOLAR BALANCE (M)

(Na,K)2SO4	24.20
(Na,K)Cl	8.92
(Na,K)BF4	0.23
(K,Na)2B4O7	8.94
Na2CrO4	1.27
OXIDES	46.94
SUM	90.49

MOLAR SUMS

SALTS	33.35	36.85
BORATES	8.94	9.88
CHROMATES	1.27	1.40
OXIDES	46.94	51.87
TOTAL	90.49	100.00

MOLES

K,Na excess*	**0.31**
K,Na excess**	**0.21**
SO4 excess	**0.00**
Cl excess	**0.00**
F excess	**0.00**
B excess	**0.00**

* Excess after assignment with Cl, SO4 and F

** Excess after assignment with Cr and B

APPENDIX II

Mass Balance Calculations for Off-gas Deposits

SAMPLE ID= 104232/OFFGAS PLUG END SCRAPINGS

	ELEMENT WT%	GRAV FACTOR	OXIDE WT%
Al2O3	1.65	1.89	3.12
CaO	1.54	1.40	2.15
Fe2O3	12.90	1.43	18.44
MgO	0.09	1.66	0.16
MnO	2.74	1.29	3.54
Na2O	13.40	1.35	1.42
Li2O	0.60	2.15	1.30
NiO	1.13	1.27	1.44
SiO2	2.34	2.14	5.01
Cr2O3	0.50	1.46	0.74
B2O3	3.44	3.22	0.00
SrO	0.04	1.18	0.05
ZrO2	0.04	1.35	0.06
TiO2	0.03	1.67	0.05
K2O	7.11	1.20	2.16
Cs2O	1.61	1.06	1.71
Cl	6.72		
F	0.14		
SO4	14.62		
SUMS			41.33

MOLAR BALANCE (M)		MOLAR SUMS	MOLE %	NORMALIZED MOLE %
(Na,K)2SO4	24.07	SALTS	36.88	38.04
(Na,K)Cl	12.60	BORATES	17.19	17.73
(Na,K)BF4	0.22	CHROMATES	1.57	1.62
(K,Na)2B4O7	17.19	OXIDES	41.33	42.62
Na2CrO4	1.57			
OXIDES	41.33	TOTAL	96.97	100.00
SUM	96.97			

MOLES	
K,Na excess*	**0.27**
K,Na excess**	**0.09**
SO4 excess	**0.00**
Cl excess	**0.00**
F excess	**0.00**
B excess	**0.00**

* Excess after assignment with Cl, SO4 and F

** Excess after assignment with Cr and B

APPENDIX II

Mass Balance Calculations for Off-gas Deposits

SAMPLE ID= 104233/OFFGAS PLUG FLANGE SCRAPINGS

	ELEMENT WT%	GRAV FACTOR	OXIDE WT%
Al2O3	1.67	1.89	3.16
CaO	1.55	1.40	2.17
Fe2O3	12.40	1.43	17.73
MgO	0.12	1.66	0.20
MnO	2.46	1.29	3.18
Na2O	14.39	1.35	2.95
Li2O	0.73	2.15	1.57
NiO	1.10	1.27	1.40
SiO2	4.33	2.14	9.26
Cr2O3	0.46	1.46	0.68
B2O3	1.57	3.22	0.00
SrO	0.03	1.18	0.04
ZrO2	0.09	1.35	0.12
TiO2	0.25	1.67	0.41
K2O	6.21	1.20	4.49
Cs2O	2.15	1.06	2.28
Cl	3.10		
F	0.10		
SO4	19.96		
SUMS			49.63

MOLAR BALANCE (M)

(Na,K)2SO4	32.86
(Na,K)Cl	5.82
(Na,K)BF4	0.16
(K,Na)2B4O7	7.82
Na2CrO4	1.44
OXIDES	49.63
SUM	97.72

MOLAR SUMS	MOLE %	NORMALIZED MOLE %
SALTS	38.84	39.74
BORATES	7.82	8.00
CHROMATES	1.44	1.48
OXIDES	49.63	50.78
TOTAL	97.72	100.00

MOLES

K,Na excess*	**0.28**
K,Na excess**	**0.19**
SO4 excess	**0.00**
Cl excess	**0.00**
F excess	**0.00**
B excess	**0.00**

* Excess after assignment with Cl, SO4 and F

** Excess after assignment with Cr and B

APPENDIX II

Mass Balance Calculations for Off-gas Deposits

SAMPLE ID= 104234/OFFGAS PLUG VARIOUS SCRAPINGS

	ELEMENT WT%	GRAV FACTOR	OXIDE WT%
Al2O3	1.62	1.89	3.06
CaO	0.56	1.40	0.78
Fe2O3	3.54	1.43	5.06
MgO	0.21	1.66	0.34
MnO	0.70	1.29	0.90
Na2O	13.94	1.35	1.71
Li2O	0.88	2.15	1.88
NiO	1.13	1.27	1.44
SiO2	7.18	2.14	15.36
Cr2O3	0.91	1.46	1.32
B2O3	3.99	3.22	0.00
SrO	0.00	1.18	0.01
ZrO2	0.08	1.35	0.11
TiO2	0.13	1.67	0.22
K2O	7.37	1.20	2.60
Cs2O	1.31	1.06	1.39
Cl	6.43		
F	0.15		
SO4	13.56		
SUMS			36.20

MOLAR BALANCE (M)		**MOLAR SUMS**	**MOLE %**	**NORMALIZED MOLE %**
(Na,K)2SO4	22.32	SALTS	34.63	37.00
(Na,K)Cl	12.06	BORATES	19.94	21.31
(Na,K)BF4	0.24	CHROMATES	2.82	3.01
(K,Na)2B4O7	19.94	OXIDES	36.20	38.68
Na2CrO4	2.82			
OXIDES	36.20	TOTAL	93.59	100.00
SUM	93.59			

MOLES	
K,Na excess*	**0.33**
K,Na excess**	**0.11**
SO4 excess	**0.00**
Cl excess	**0.00**
F excess	**0.00**
B excess	**0.00**

* Excess after assignment with Cl, SO4 and F

** Excess after assignment with Cr and B

APPENDIX II

Mass Balance Calculations for Off-gas Deposits

SAMPLE ID= **104235/HAIR FROM VACUUM CYCLONE SEPERATOR**

	ELEMENT WT%	GRAV FACTOR	OXIDE WT%
Al2O3	1.69	1.89	3.19
CaO	1.79	1.40	2.50
Fe2O3	13.50	1.43	19.30
MgO	0.11	1.66	0.18
MnO	1.94	1.29	2.50
Na2O	15.13	1.35	1.57
Li2O	0.67	2.15	1.43
NiO	0.49	1.27	0.62
SiO2	2.94	2.14	6.29
Cr2O3	0.47	1.46	0.68
B2O3	3.65	3.22	0.00
SrO	0.44	1.18	0.52
ZrO2	0.06	1.35	0.08
TiO2	0.24	1.67	0.41
K2O	6.14	1.20	2.38
Cs2O	3.86	1.06	4.09
Cl	13.43		
F	5.89		
SO4	5.28		
SUMS			45.75

MOLAR BALANCE (M)

(Na,K)2SO4	8.69
(Na,K)Cl	25.18
(Na,K)BF4	9.13
(K,Na)2B4O7	14.13
Na2CrO4	1.45
OXIDES	45.75
SUM	104.34

MOLAR SUMS	MOLE %	NORMALIZED MOLE %
SALTS	43.01	41.22
BORATES	14.13	13.55
CHROMATES	1.45	1.39
OXIDES	45.75	43.85
TOTAL	104.34	100.00

MOLES	
K,Na excess*	**0.25**
K,Na excess**	**0.10**
SO4 excess	**0.00**
Cl excess	**0.00**
F excess	**0.00**
B excess	**0.00**

* Excess after assignment with Cl, SO4 and F

** Excess after assignment with Cr and B

APPENDIX II

Mass Balance Calculations for Off-gas Deposits

SAMPLE ID= 107270/SGM-8 PLUGGAGE

	ELEMENT WT%	GRAV FACTOR	OXIDE WT%
Al2O3	1.22	1.89	2.31
CaO	0.85	1.40	1.19
Fe2O3	6.57	1.43	9.39
MgO	0.23	1.66	0.39
MnO	1.37	1.29	1.77
Na2O	13.50	1.35	-0.87
Li2O	1.18	2.15	2.54
NiO	0.37	1.27	0.47
SiO2	9.52	2.14	20.37
Cr2O3	0.80	1.46	1.16
B2O3	4.64	3.22	0.00
SrO	0.01	1.18	0.02
ZrO2	0.25	1.35	0.33
TiO2	0.11	1.67	0.19
K2O	3.51	1.20	-1.32
Cs2O	0.44	1.06	0.47
Cl	11.85		
F	0.13		
SO4	7.35		
SUMS			38.39

MOLAR BALANCE (M)	
(Na,K)2SO4	12.10
(Na,K)Cl	22.22
(Na,K)BF4	0.20
(K,Na)2B4O7	23.23
Na2CrO4	2.48
OXIDES	38.39
SUM	98.61

MOLAR SUMS	MOLE %	NORMALIZED MOLE %
SALTS	34.52	35.00
BORATES	23.23	23.56
CHROMATES	2.48	2.51
OXIDES	38.39	38.93
TOTAL	98.61	100.00

MOLES	
K,Na excess*	0.19
K,Na excess**	-0.06
SO4 excess	0.00
Cl excess	0.00
F excess	0.00
B excess	0.00

* Excess after assignment with Cl, SO4 and F

** Excess after assignment with Cr and B

APPENDIX II

Mass Balance Calculations for Off-gas Deposits

SAMPLE ID= 110547/110539/31835/31836/SGM-9 PLUGGAGE

	ELEMENT WT%	GRAV FACTOR	OXIDE WT%
Al2O3	1.43	1.89	2.70
CaO	1.07	1.40	1.49
Fe2O3	9.79	1.43	14.00
MgO	0.11	1.66	0.18
MnO	1.57	1.29	2.03
Na2O	12.56	1.35	-1.51
Li2O	1.18	2.15	2.54
NiO	0.89	1.27	1.13
SiO2	6.94	2.14	14.85
Cr2O3	0.35	1.46	0.50
B2O3	1.10	3.22	0.00
SrO	0.27	1.18	0.32
ZrO2	0.07	1.35	0.09
TiO2	0.14	1.67	0.23
K2O	5.92	1.20	-2.30
Cs2O	1.00	1.06	1.06
Cl	5.84		
F	0.26		
SO4	27.14		
SUMS			37.31

MOLAR BALANCE (M)	
(Na,K)2SO4	44.68
(Na,K)Cl	10.95
(Na,K)BF4	0.40
(K,Na)2B4O7	5.34
Na2CrO4	1.07
OXIDES	37.31
SUM	99.76

MOLAR SUMS	MOLE %	NORMALIZED MOLE %
SALTS	56.03	56.17
BORATES	5.34	5.36
CHROMATES	1.07	1.08
OXIDES	37.31	37.40
TOTAL	99.76	100.00

MOLES	
K,Na excess*	**-0.04**
K,Na excess**	**-0.10**
SO4 excess	**0.00**
Cl excess	**0.00**
F excess	**0.00**
B excess	**0.00**

* Excess after assignment with Cl, SO4 and F

** Excess after assignment with Cr and B

APPENDIX II

Mass Balance Calculations for Off-gas Deposits

SAMPLE ID= 105020/GLASS TECHNOLOGY GROUP MINIMELTER DEPOSITS

	ELEMENT WT%	GRAV FACTOR	OXIDE WT%
Al2O3	2.00	1.89	3.78
CaO	1.72	1.40	2.41
Fe2O3	10.38	1.43	14.84
MgO	0.17	1.66	0.29
MnO	3.70	1.29	4.78
Na2O	8.75	1.35	-2.17
Li2O	1.19	2.15	2.55
NiO	7.40	1.27	9.42
SiO2	6.28	2.14	13.43
Cr2O3	1.22	1.46	1.78
B2O3	2.38	3.22	0.00
SrO	0.04	1.18	0.05
ZrO2	0.09	1.35	0.12
TiO2	0.17	1.67	0.29
K2O	1.37	1.20	-3.30
Cs2O		1.06	0.00
Cl	10.53		
F	12.77		
SO4	0.89		
SUMS			48.25

MOLAR BALANCE (M)	
(Na,K)2SO4	1.47
(Na,K)Cl	19.74
(Na,K)BF4	19.80
(K,Na)2B4O7	2.81
Na2CrO4	3.79
OXIDES	48.25
SUM	95.86
MOLES	
K,Na excess*	**-0.07**
K,Na excess**	**-0.14**
SO4 excess	**0.00**
Cl excess	**0.00**
F excess	**0.00**
B excess	**0.00**

MOLAR SUMS	MOLE %	NORMALIZED MOLE %
SALTS	41.01	42.78
BORATES	2.81	2.93
CHROMATES	3.79	3.95
OXIDES	48.25	50.33
TOTAL	95.86	100.00

* Excess after assignment with Cl, SO4 and F

** Excess after assignment with Cr and B

APPENDIX II

Mass Balance Calculations for Off-gas Deposits

SAMPLE ID= 2000027137/200031837/200031838
IDMS PIPE "STUCK" DEPOSIT-SLUDGE ONLY CAMPAIGN #1

	ELEMENT WT%	GRAV FACTOR	OXIDE WT%
Al2O3	1.83	1.89	3.46
CaO	3.70	1.40	5.18
Fe2O3	11.06	1.43	15.81
MgO	0.14	1.66	0.24
MnO	2.29	1.29	2.95
Na2O	13.31	1.35	1.88
Li2O	0.83	2.15	1.79
NiO	0.98	1.27	1.24
SiO2	5.17	2.14	11.05
Cr2O3	1.42	1.46	2.08
B2O3	2.37	3.22	0.00
SrO	0.01	1.18	0.01
ZrO2	0.08	1.35	0.11
TiO2	0.04	1.67	0.06
K2O	1.67	1.20	2.85
Cs2O		1.06	0.00
Cl	5.84		
F	2.18		
SO4	7.57		
SUMS			48.71

MOLAR BALANCE (M)	
(Na,K)2SO4	12.47
(Na,K)Cl	10.95
(Na,K)BF4	3.39
(K,Na)2B4O7	10.33
Na2CrO4	4.42
OXIDES	48.71
SUM	90.26

MOLAR SUMS	MOLE %	NORMALIZED MOLE %
SALTS	26.80	29.70
BORATES	10.33	11.44
CHROMATES	4.42	4.90
OXIDES	48.71	53.96
TOTAL	90.26	100.00

MOLES	
K,Na excess*	**0.27**
K,Na excess**	**0.12**
SO4 excess	**0.00**
Cl excess	**0.00**
F excess	**0.00**
B excess	**0.00**

* Excess after assignment with Cl, SO4 and F

** Excess after assignment with Cr and B

APPENDIX II

Mass Balance Calculations for Off-gas Deposits

SAMPLE ID= **2000027138/200031839/200031840**
IDMS PIPE LOOSE DEPOSITS-SLUDGE ONLY CAMPAIGN #1

	ELEMENT WT%	GRAV FACTOR	OXIDE WT%
Al2O3	2.95	1.89	5.58
CaO	4.15	1.40	5.80
Fe2O3	17.63	1.43	25.21
MgO	0.20	1.66	0.32
MnO	3.35	1.29	4.33
Na2O	8.75	1.35	2.15
Li2O	1.01	2.15	2.18
NiO	1.57	1.27	2.00
SiO2	7.89	2.14	16.87
Cr2O3	0.73	1.46	1.07
B2O3	2.70	3.22	0.00
SrO	0.01	1.18	0.01
ZrO2	0.13	1.35	0.18
TiO2	0.07	1.67	0.12
K2O	0.74	1.20	3.27
Cs2O		1.06	0.00
Cl	2.06		
F	1.29		
SO4	1.99		
SUMS			69.08

MOLAR BALANCE (M)	
(Na,K)2SO4	3.27
(Na,K)Cl	3.86
(Na,K)BF4	2.00
(K,Na)2B4O7	12.64
Na2CrO4	2.27
OXIDES	69.08
SUM	93.13

MOLAR SUMS	**MOLE %**	**NORMALIZED MOLE %**
SALTS	9.13	9.81
BORATES	12.64	13.57
CHROMATES	2.27	2.44
OXIDES	69.08	74.18
TOTAL	93.13	100.00

MOLES	
K,Na excess*	**0.28**
K,Na excess**	**0.14**
SO4 excess	**0.00**
Cl excess	**0.00**
F excess	**0.00**
B excess	**0.00**

* Excess after assignment with Cl, SO4 and F
** Excess after assignment with Cr and B

APPENDIX II

Mass Balance Calculations for Off-gas Deposits

SAMPLE ID= 2000038736/200038737

IDMS FILM COOLER DEPOSIT #1

	ELEMENT WT%	GRAV FACTOR	OXIDE WT%
Al2O3	0.70	1.89	1.32
CaO	0.24	1.40	0.34
Fe2O3	3.04	1.43	4.35
MgO	0.02	1.66	0.03
MnO	0.66	1.29	0.85
Na2O	NO DATA	1.35	0.00
Li2O	1.09	2.15	2.34
NiO	0.23	1.27	0.29
SiO2	0.67	2.14	1.42
Cr2O3	1.18	1.46	1.73
B2O3	1.50	3.22	4.84
SrO	0.01	1.18	0.01
ZrO2	NO DATA	1.35	
TiO2	0.05	1.67	0.09
K2O	NO DATA	1.20	0.00
Cs2O	NO DATA	1.06	
Cl	NO DATA		
F	NO DATA		
SO4	NO DATA		
SUMS			

MOLAR BALANCE (M)	
(Na,K)2SO4	
(Na,K)Cl	
(Na,K)BF4	
(K,Na)2B4O7	
Na2CrO4	3.68
OXIDES	
SUM	3.68
MOLES	
K,Na excess*	
K,Na excess**	
SO4 excess	
Cl excess	
F excess	
B excess	**0.14**

MOLAR SUMS	MOLE %	NORMALIZED MOLE %
SALTS	0.00	0.00
BORATES	0.00	0.00
CHROMATES	3.68	100.00
OXIDES	0.00	0.00
TOTAL	3.68	100.00

* Excess after assignment with Cl, SO4 and F

** Excess after assignment with Cr and B

APPENDIX II

Mass Balance Calculations for Off-gas Deposits

SAMPLE ID= 2000038739/
IDMS FILM COOLER DEPOSIT #2

	ELEMENT WT%	GRAV FACTOR	OXIDE WT%
Al2O3	2.57	1.89	4.86
CaO	1.58	1.40	2.21
Fe2O3	12.83	1.43	18.34
MgO	0.12	1.66	0.21
MnO	2.82	1.29	3.64
Na2O	10.83	1.35	-0.74
Li2O	0.68	2.15	1.46
NiO	1.08	1.27	1.37
SiO2	2.92	2.14	6.25
Cr2O3	1.09	1.46	1.59
B2O3	1.62	3.22	0.00
SrO	0.03	1.18	0.04
ZrO2	0.04	1.35	0.06
TiO2	0.22	1.67	0.37
K2O	5.93	1.20	-1.13
Cs2O	1.10	1.06	1.17
Cl	13.42		
F	1.11		
SO4	8.07		
SUMS			39.69

MOLAR BALANCE (M)	
(Na,K)2SO4	13.29
(Na,K)Cl	25.16
(Na,K)BF4	1.72
(K,Na)2B4O7	7.35
Na2CrO4	3.39
OXIDES	39.69
SUM	90.60

MOLAR SUMS	MOLE %	NORMALIZED MOLE %
SALTS	40.17	44.33
BORATES	7.35	8.11
CHROMATES	3.39	3.75
OXIDES	39.69	43.81
TOTAL	90.60	100.00

MOLES	
K,Na excess	**0.06**
K,Na excess	**-0.05**
SO4 excess	**0.00**
Cl excess	**0.00**
F excess	**0.00**
B excess	**0.00**

* Excess after assignment with Cl, SO4 and F
** Excess after assignment with Cr and B

APPENDIX II

Mass Balance Calculations for Off-gas Deposits

SAMPLE ID= 2000038740/2000038741
IDMS FILM COOLER DEPOSIT #3

	ELEMENT WT%	GRAV FACTOR	OXIDE WT%
Al2O3	2.75	1.89	5.20
CaO	1.74	1.40	2.43
Fe2O3	14.58	1.43	20.84
MgO	0.13	1.66	0.22
MnO	3.07	1.29	3.96
Na2O	10.64	1.35	-1.63
Li2O	0.75	2.15	1.61
NiO	3.19	1.27	4.06
SiO2	3.21	2.14	6.86
Cr2O3	2.06	1.46	3.01
B2O3	1.39	3.22	0.00
SrO	0.03	1.18	0.04
ZrO2	0.04	1.35	0.05
TiO2	0.23	1.67	0.38
K2O	6.51	1.20	-2.48
Cs2O	1.40	1.06	1.48
Cl	11.57		
F	3.87		
SO4	11.50		
SUMS			46.04

MOLAR BALANCE (M)	
(Na,K)2SO4	18.93
(Na,K)Cl	21.69
(Na,K)BF4	6.00
(K,Na)2B4O7	4.21
Na2CrO4	6.42
OXIDES	46.04
SUM	103.29

MOLAR SUMS	MOLE %	NORMALIZED MOLE %
SALTS	46.63	45.14
BORATES	4.21	4.08
CHROMATES	6.42	6.21
OXIDES	46.04	44.57
TOTAL	103.29	100.00

MOLES	
K,Na excess	0.01
K,Na excess	-0.11
SO4 excess	0.00
Cl excess	0.00
F excess	0.00
B excess	0.00

* Excess after assignment with Cl, SO4 and F
** Excess after assignment with Cr and B