CONF-900809--5

PNL-SA--17928
DE91 004069

Received by OSTI

NOV 26 1990

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June 1990

To be presented at the 21st DOE/NRC Nuclear Air
Cleaning Conference - August 13-16, 1990
San Diego, California

Work Supported by the U.S. Department of Energy
Under Contract DE-AC06-76RLO 1830

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LFCM PROCESSING CHARACTERISTICS OF MERCURY

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Abstract

An experimental-scale liquid-fed ceramic melter was used in a series of tests to evaluate the processing characteristics of mercury in simulated defense waste under various melter operating conditions. This solidification technology had no detectable capacity for incorporating mercury into its borosilicate, vitreous product, and essentially all the mercury fed to the melter was lost to the off-gas system as gaseous effluent. An ejector venturi scrubber condensed and collected 97% of the mercury evolved from the melter. Chemically the condensed mercury effluent was composed entirely of chlorides, and, except in a low-temperature test, Hg_2Cl_2 was the primary chloride formed. As a result, combined mercury accounted for most of the insoluble mass collected by the process quench scrubber. Although macroscopic quantities of elemental mercury were never observed in process secondary waste streams, finely divided and dispersed mercury that blackened all condensed Hg_2Cl_2 residues was capable of saturating the quenched process exhaust with mercury vapor. However, the vapor pressure of mercury in the quenched melter exhaust was easily and predictably controlled with an off-gas stream chiller.

Introduction

Radioactive high-level liquid waste (HLLW), a byproduct of defense nuclear fuel reprocessing activities, is currently being stored in underground tanks at several U.S. sites. Because its mobility poses significant environmental risks, HLLW is not a suitable waste form for long-term storage. High-temperature processes for solidifying and isolating the radioactive components of HLLW have therefore been developed and demonstrated by the U.S. Department of Energy (DOE) and its contractors. Specifically, vitrification using liquid-fed ceramic melters (LFCMs) has become the reference process for converting U.S. HLLW into a borosilicate glass, which is considered a suitably stable matrix for permanent geological storage. Two vitrification plants are currently under construction in the United States: the West Valley Demonstration Plant (WVDP) being built at the former West Valley Nuclear Fuels Services site in West Valley, New York; and the Defense Waste Processing Facility (DWPF), which is nearing completion at DOE's Savannah River Plant (SRP) in Aiken, South Carolina. A third facility, the Hanford Waste Vitrification Plant (HWVP), is being designed at DOE's Hanford Site in southeastern Washington.

The Pacific Northwest Laboratory (PNL) has been developing HLLW immobilization technologies under the sponsorship of the DOE and its predecessor agencies for well over 25 years. Since 1973, PNL has technically supported the design and development of the LFCM vitrification process. Much of this development work has involved nonradioactive experimental studies using a ~1/3-scale LFCM and its associated off-gas processing system. Since 1981, more than 20 LFCM tests involving 3000 h of processing have been conducted at PNL in direct support of the DWPF, the WVDP, and the HWVP.

As a result of the diverse needs and specific processing requirements associated with the HLLW at each of these DOE sites, PNL-LFCM tests have provided a wealth of information regarding the processibility of a variety of waste components under diverse process and operating conditions. The processibility of mercury is of particular importance with respect to melter operability, secondary waste stream management, and environmental compliance modeling due to the (electrical) conductivity, volatility, and toxicity, respectively, of this element and its chemical compounds. Because of the unique challenges the presence of mercury poses to LFCM processing of HLLW, this paper deals exclusively with the empirically determined processing and off-gas behavior of mercury under the various LFCM operating conditions encountered during scaled simulation testing.

Before the operating and processing conditions used during these LFCM tests are discussed, a brief description of the scaled melter and its off-gas processing and sampling systems will be presented along with feed composition data. The nature and magnitudes of melter mercury losses and the impact of these losses on the off-gas processing will then be discussed as a function of test operating conditions. Finally, the results of a destructive post mortem examination of the melter and its off-gas processing system will be used to establish processing system accumulation sites of feed component mercury.

Liquid-Fed Melter Processing and Sampling System Description

This section briefly describes the liquid-fed melter processing system developed at PNL in support of DOE's waste isolation programs. Further information concerning the LFCM technology and its associated off-gas processing requirements is documented elsewhere.(1-3)

Melter

Joule-heated, ceramic-lined melters form the basis of the LFCM process, which is illustrated in Figure 1. This type of glass melter, developed for the commercial glass industry, is composed of a heavily insulated, ceramic-lined cavity that contains the molten glass, and corrosion-resistant electrodes. The electrical resistive heating used to maintain the glass in its molten state and to process feed chemicals into more glass is produced by passing an electric current (AC) through the glass pool via the immersed melter electrodes--this process is known as joule heating. In addition to this primary source of power, auxiliary radiant heaters located in the melter plenum above the glass melt surface are also used to increase or boost processing rates. Because joule-heated ceramic melters can attain higher temperatures than conventional melters, high-silica glasses with exceptional chemical stability can be fabricated. This feature is important to LFCM applications because the vitreous waste form must exhibit low aqueous leaching rates to qualify for long-term geological storage.

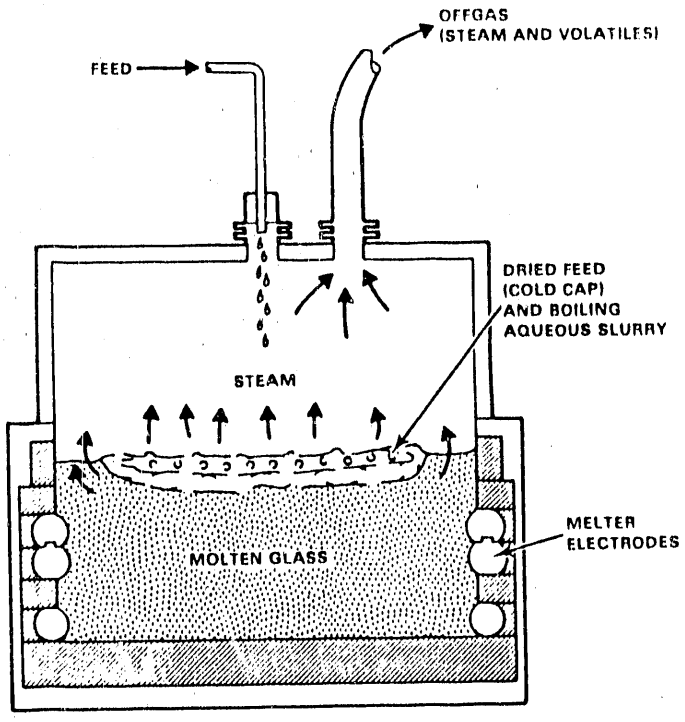

Figure 1. Liquid-Fed Ceramic Melter Process

The use of joule-heated ceramic melters to vitrify HLLW is a straightforward extension of glass-making technology; the waste components are treated merely as a subset of the chemicals needed to produce a specific glass product. In the LFCM process, major glass-forming chemicals and/or glass frit are mixed with the HLLW stream to produce an aqueous slurry that is continuously fed to the joule-heated melter to produce a molten radioactive glass. The molten melter glass product, which commonly has a waste loading of 25 wt%, is transferred via an overflow port to metal canisters that are subsequently cooled and then sealed to further isolate the immobilized waste form.

Because of the health hazards associated with mercury and its chemical compounds, an experimental-scale ceramic melter (ESCM) was exclusively used for all melter tests involving this toxic element. The ESCM was a 1/50-scale melter with a 0.054 m^2 glass surface area and was small enough to be contained within a walk-in hood. Because it is small, melter inleakage could be successfully controlled, allowing plenum oxidizing conditions to be used as a process parameter. Figure 2 illustrates the experimental arrangement involving the ESCM and its ancillary feed and off-gas processing systems.

<u>Feed Composition</u>

The liquid slurry feed used for the ESCM mercury tests was a uniform mixture of simulated defense waste sludge and glass formers (frit). The composition of this slurry and the projected equivalent oxide composition of the resultant process glass are detailed in Table 1. Because mercury does not possess a stable oxide at glass

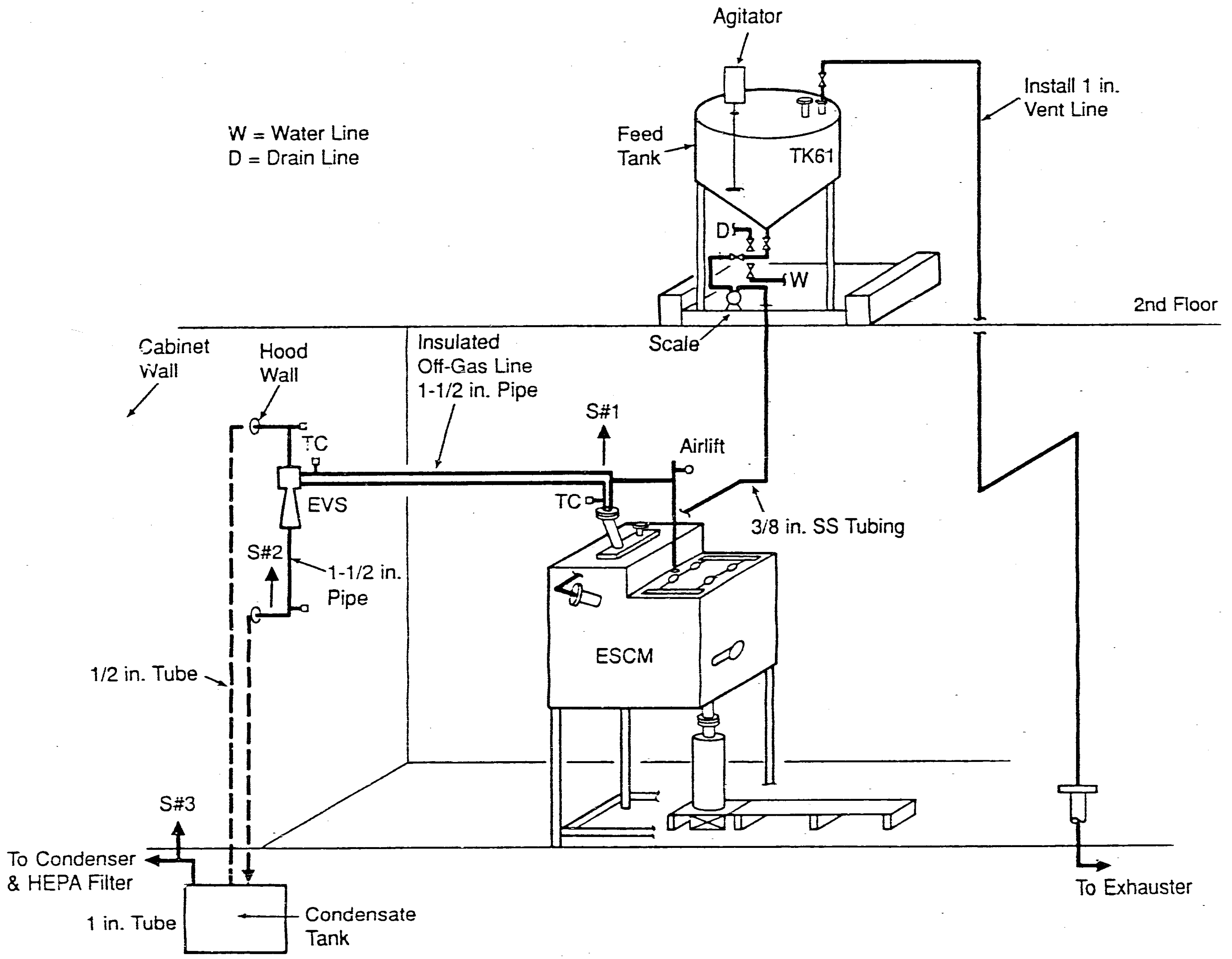

Figure 2. Experimental Scale Ceramic Melter and Off-Gas Processing System

Table 1. Simulated LFCM Defense Waste Feed

Component	Feed*		Product		
	wt%	g/L	Form	Equivalent, g/L	Glass, wt%
$Fe(OH)_3$	12.41	70.1	FeO	15.7	3.25
			Fe_2O_3	34.9	7.22
$Mn(COOH)_2$	3.00	17.0	MnO	11.8	2.45
MnO_2	0.77	4.36			
$Al(OH)_3$	5.84	33.0	Al_2O_3	25.1	5.18
$Ca(COOH)_2$	1.29	7.28			
$CaCO3$	0.25	1.40	CaO	6.44	1.25
$CaSO_4$	0.16	0.90	$CaSO_4$	0.90	0.19
CaF_2	0.057	0.32			
$Ca_3(PO_4)_2$	0.047	0.26	$Ca_3(PO4)_2$	0.26	0.054
$Na(COOH)_2$	2.85	16.1	NiO	3.13	0.65
$Ni(OH)_2$	0.76	4.30			
NaCOOH)	2.91	16.4			
Na_2SO_4	0.079	0.45	Na_2SO_4	0.45	0.093
NaC	0.51	2.90	NaCl	2.9	0.60
NaI	0.0055	0.031	NaI	0.03	0.006
Na_2O	7.71	43.6	Na_2O	54.2	11.19
Na_3PO_4	0.0055	0.031	Na_3PO_4	0.03	0.006
NaF	0.030	0.17	NaF	0.52	0.11
$NaNO_3$	1.26	7.13			
RuO_2	0.13	0.71	RuO_2	0.71	0.15
$Cr(OH)_3$	0.12	0.67	Cr_2O_3	0.49	0.10
Cu_2O	0.10	0.54	CuO	0.60	0.12
Hg	0.15	0.83			
Zeolite	3.31	18.7			
KCOOH	0.16	0.90	K_2O	0.51	0.10
$Sr(COOH)_2$	0.036	0.204	SrO	0.119	0.025
Rare Earths	0.063	0.36	RE_2O_3	0.36	0.074
$CsNO_3$	0.099	0.56	Cs_2O	0.40	0.083
B_2O_3	6.28	35.5	B_2O_3	35.5	7.33
Li_2O	4.39	24.8	Li_2O	24.8	5.13
ZrO_2	0.63	3.55	ZrO_2	3.55	0.73
SiO_2	43.96	248.0	SiO_2	257.0	53.18
MgO	0.63	3.55	MgO	3.55	0.73

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*Feed Density = 1.363; Feed Solids = 565 g/L; Glass Product = 484 g/L.

processing temperatures, the process efficiency for vitrifying this element, as shown in the table, was expected to be quite low; this was in fact one of the primary reasons for conducting the ESCM tests in the first place. The effective waste loading of the slurry used was 25 wt% of the total oxides present in the feed.

Although the composition of the liquid feed remained nominally constant throughout the period of testing covered in this report, the quantities of formic acid, NaCl and Na_2SO_4 used in the feed formulations were altered during the testing program to establish their influence upon feed processing rates, glass quality, and effluent emission characteristics. These as well as other process variables used during ESCM testing are summarized in Table 2 along with related concurrent processing conditions.

Off-Gas Processing System

Because of the turbulent conditions produced by introducing an aqueous slurry into a melter whose plenum and glass temperatures are greater than 350°C and 1000°C, respectively, a contaminated gas phase waste (off-gas) stream is generated that must be treated before it can be released to the environment. A melter off-gas (MOG) treatment system provides this function by pulling off these hot, contaminated, steam-laden emissions and decontaminating them. As discussed previously,(1) LFCM process exhaust consists primarily of steam, volatilized effluent, and suspended condensed-phase matter (aerosols). Since most waste components that require long-term environmental isolation are lost to the off-gas system as aerosols, LFCM process off-gas (POG) treatment systems are composed primarily of aerosol emission abatement devices and ancillary support equipment.

Like other high-temperature combustion processes, vitrification produces two distinct and independent classes of aerosol effluent: entrained and semivolatile condensed matter.(1) Because many radioactive semivolatiles such as ^{137}Cs form condensation aerosols that are

Table 2. ESCM Operation and Production Summary

Process Parameter	Units	Test 1	Test 2	Test 3	Test 4	Test 5
Duration	h	236	107	103	104	112
Feed Processed	kg	1198	344	187	280	450
Average Feed Rate	$l/h\text{-}m^2$	70	46	25	36	54
Glass Produced	kg	426	129	66	103	165
Average Glass Rate	$kg/h\text{-}m^2$	35	23	12	18	27
Hg Processed	kg	0.76	0.23	0.12	0.18	0.29
Plenum Temperature	°C	750	750	550	740	740
Air Inleakage	kg/h	0.67	0.82	0.37	0.46	0.90
Feed NaCl	wt%	0.51	0.51	0.51	0.51	0.76
Feed Na_2SO_4	wt%	0.079	0.079	0.079	0.079	0.36
Feed HCOOH	%ST*	90	86	86	110	110

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*The amount of formic acid used is specified in terms of the percent of the stoichiometric amount required to complete acid/base reaction with the waste sludge, plus that required to decompose all carbonates and nitrates.

primarily of submicron size, high-efficiency aerosol removal devices are essential components of LFCM processing systems. However, these devices are not, in general, compatible with high off-gas aerosol and steam loadings or with high melter exhaust temperatures; consequently, aqueous quench scrubbers are universally used to precondition the process exhaust by cooling the exhaust stream, condensing the steam, and removing large-diameter (>1 μm) entrained debris. Because entrained matter can also present an off-gas line blockage problem, off-gas quenchers are usually close coupled to the melter source to minimize or at least localize this problem.

In general, additional stages of off-gas treatment would be required if one or more radioactive waste components formed a gaseous effluent that was thermodynamically stable and was little affected by the aqueous quench scrubbing stage. However, radionuclides such as these (^3H, ^{14}C, and ^{129}I) are usually not significant HLLW components (on a curie basis). Process generation of regulated gaseous, chemical effluents such as NO_x, SO_2, and Hg may also require additional treatment stages, depending upon the concentrations of these effluents penetrating the primary quench scrubber.

The process off-gas system used in support of the ESCM testing discussed here utilized three stages of off-gas treatment consisting of an ejector venturi (quench) scrubber (EVS), a water-cooled off-gas condenser, and a final high efficiency particulate air (HEPA) filter, arranged sequentially in that order. The off-gas condenser was employed to reduce off-gas water loadings before final HEPA filtration and to assess the ability of an off-gas chiller to reduce potential off-gas concentrations of mercury vapor. Since the purpose of the ESCM tests was primarily to characterize the melter effluent behavior of mercury, the performance of the generic off-gas processing system was not exhaustively characterized as was the melter source; however, sufficient data were collected to establish the average off-gas operating and performance characteristics.

Off-Gas Effluent Sampling System

The sampling network, which was developed in support of melter emission characterization studies, is conceptually illustrated in Figure 3. The components making up this network were designed to selectively determine the composition of melter exhaust with regard to particulate matter (Fe, Si, Sr, etc.), semivolatile effluent (compounds of B, Cs, Ru, Hg, etc.), and noncondensible gases (O_2, CO, CO_2, SO_2, and Hg).

Sampling for mercury effluent in the unquenched (~350°C) melter exhaust was conducted using a differential effluent sampling system to assess the pathways and magnitudes of condensible and/or scrubbable melter-generated emissions (semivolatile and particulate effluent). This sampling system was composed of a heated (~200°C) particulate filter, a heat-traced sampling line, a condenser, and a series arrangement of three gas scrubbing units sequentially configured in the order listed (Figure 3). The distribution of semivolatiles across the five discrete sample fractions generated by the system was determined using inductively coupled plasma (ICP) spectrometry, atomic absorption (AA), ion chromatography (IC), and

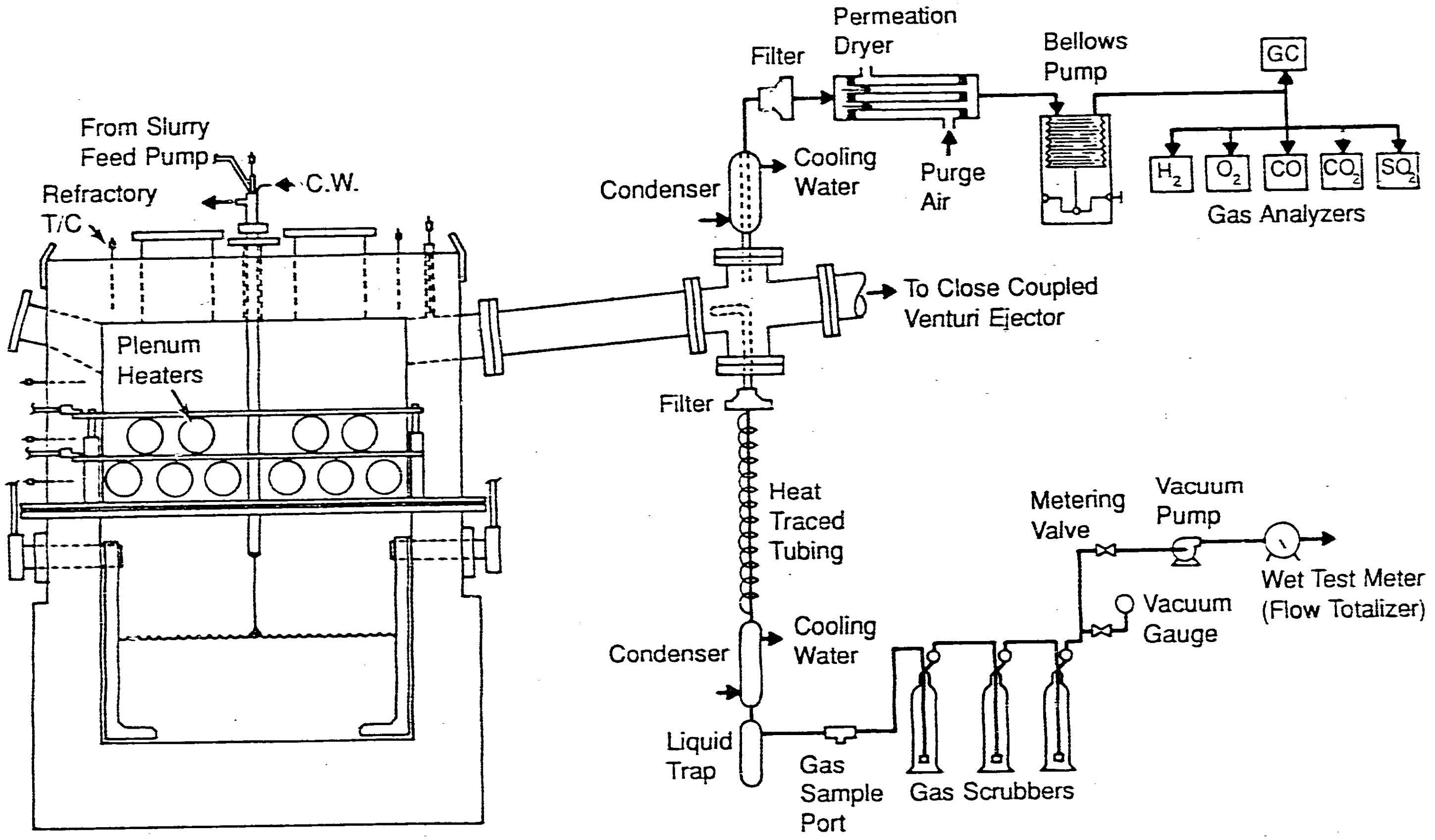

Figure 3. Melter Off-Gas Sampling Network

x-ray fluorescence (XRF). Chemical identities of crystalline effluents collected were established by x-ray diffraction techniques.

With the exception of mercury, the gaseous composition of melter off-gas emissions was established using continuous real-time gas analyzers. The gas stream sampled was extracted after the off-gas quencher (site S2 of Figure 2) in the two-phase off-gas line leading to the EVS condensate tank. The moisture-laden gas sample stream was passed through a heated filter assembly to remove airborne particulate matter and then through a permeation dryer to reduce the moisture loading of the gas before it was distributed to the individual gas analyzers by a stainless steel bellows pump.

Noncontinuous batch sampling for gaseous mercury was measured at three locations: after the venturi (S2), downstream of the quench tank (S3) and, finally, after the exhaust had passed through a condenser and final HEPA filter. The presence of mercury vapor at these off-gas system sites was measured by two separate types of Hg° monitors. One instrument was based upon UV excitation, while the other operated on the basis of gold film amalgamation. Being low-level instruments, neither monitor was suitable for direct sampling of the melter exhaust stream. However, by using instrument calibration techniques that involve controlled dilution of a room-temperature $Hg_{(l)}$ source, off-gas system vapor pressure measurements of mercury were obtained using off-gas grab samples.

In addition to effluent data collected by the sampling network, the glass product and the secondary aqueous waste stream generated by the MOG EVS were routinely sampled and analyzed throughout ESCM testing. Post-test examination of the melter and off-gas system was also conducted in order to establish the presence and magnitudes of mercury accumulation sites, if present.

Melter Performance and Effluent Characterization

Melter performance with regard to effluent emission is commonly expressed in terms of a unitless decontamination factor or DF. By definition, a feed component DF is the ratio of the rate at which that particular component enters a device to the rate at which it exits. Consequently, melter DFs are related to LFCM process efficiencies (1 - 1/DF) for converting feed components into a borosilicate glass. Particulate melter DFs are similarly defined in terms of mass ratios without reference to elemental composition, where only contributions of airborne particulate matter to overall melter effluent losses are considered. Since particulate DFs are strongly influenced by physical entrainment losses, they are very good indicators of processing stability. More importantly, large deviations of individual DFs from corresponding particulate DFs are direct indicators of the semivolatile or volatile properties of the element and/or its chemical compounds.(3)

Table 3 summarizes process and operating conditions used during ESCM testing and the resultant performance parameters obtained. In accordance with pre-testing expectations, processing efficiency for mercury is quite low. No trace of mercury could be found in the glass product under any of the processing conditions used, and

Table 3. ESCM PROCESSING AND PERFORMANCE PARAMETERS

			Plenum					FEED			Melter DF		
				Composition (%)				wt%					
Test Number	Hours	Prod Rate kg/hm^2	Temp (°C)	(O_2)	(CO_2)	(CO)	(H_2O)	NaCl	Na_2SC_4	% HCOOH*	Hg	Fe	Part
1	236	35.3	750	1.28	1.05	0.01	92.5	0.51	0.079	90	<1.4	430	820
2	107	23.4	750	9.78	0.47	<0.05	52.3	0.51	0.079	86	<1.4	230	520
3	103	12.3	550	3.26	0.52	0.05	82.8	0.51	0.079	86	<1.4	230	460
4	104	18.3	740	3.33	1.03	0.02	82.9	0.51	0.079	110	<1.1	230	560
5	113	27.0	740	3.11	2.07	0.35	82.7	0.76	0.36	110	<1.1	290	600

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*The amount of formic acid used is specified in terms of the percent of the stoichiometric amount required to complete acid/base reaction with the waste sludge, plus that required to decompose all carbonates and nitrates.

analytical sensitivity limits were used with feed makeup data to place an upper bound on melter DFs for this element.

Off-gas sampling results were totally consistent with the low DFs established by the glass composition data; that is, mercury was found to be a major contributor to the overall mass of condensible off-gas effluent exiting the ESCM. This result reflects the fact that a minor feed constituent with very low DF can produce a greater effluent mass loss rate than a major feed component with a significantly higher process DF. Table 4 presents melter effluent loss rates of selected feed components as a percentage of observed mercury losses. These emission rate data clearly show that, on a mass basis, mercury is the major contributor to overall process losses of nonvolatile and condensible effluent. Indeed, the average mass emission rate of mercury observed during all phases of ESCM testing was found to be only 17% lower than the average overall particulate (aerosol) loss rate that has been shown to be the predominant LFCM process loss mechanism.(1)

Although melter DFs for mercury derived from off-gas sampling data were somewhat higher than glass values previously presented (2 vs <1.1), the uncertainties in the documented inhomogeneity of mercury in the feed made the short-term off-gas sampling values indistinguishable from the glass-derived result; i.e., DF ~ 1. Nevertheless, a search for potential accumulation sites for mercury and its compounds within the melter and its off-gas system were conducted upon completion of ESCM testing, as will be discussed later in this paper.

Nature of Melter Mercury Losses

Although it was never expected that the variable process parameters evaluated during ESCM testing would influence melter processing performance for mercury, it was recognized that these variables could be quite influential in establishing the physical and chemical nature of the mercury effluent produced. In order to characterize the pathways and magnitudes of process losses of mercury, a differential sampling system composed of an aerosol filter, condenser, and a series arrangement of three gas-scrubbing units was employed. The

Table 4. Comparative LFCM Effluent Loss Rates

	Relative Melter Mass Loss Rates					
Effluent	Test 1	Test 2	Test 3	Test 4	Test 5	Average
Al	0.82	3.13	2.38	2.84	2.33	2.30
B	14.12	10.45	13.06	9.67	11.87	11.84
Cr	0.23	0.60	0.19	0.12	0.23	0.27
Fe	12.99	24.12	24.12	24.34	19.00	20.92
Hg	100.00	100.00	100.00	100.00	100.00	100.00
Mn	3.42	6.65	6.65	6.30	4.79	5.56
Na	9.25	12.02	16.83	6.57	6.94	10.32
Ni	0.06	0.00	0.00	0.01	0.01	0.02
Si	4.21	11.98	10.48	11.98	10.16	9.76
Part.	81.20	128.70	143.37	120.12	112.04	117.09

manner in which any given element is distributed across the five discrete sampling fractions of this device is indicative of the physical states assumed by the effluent and their relative importance. Sampling to characterize ESCM-generated effluents was conducted in the process exhaust line connecting the melter plenum to the EVS (site S1). The temperature in this jumper was maintained between 300°C and 400°C, using steam injection cooling when necessary.

The sampling results obtained were invariant throughout all phases of ESCM testing. Specifically, all mercury-containing effluent was collected in the condensate fraction of the differential sampling system. Since mercury was never detected in the filter catch, the above sampling results indicate that mercury is primarily a gas phase effluent in the unquenched process exhaust under all the process operating conditions examined. A typical x-ray fluorescence spectrum of insoluble matter collected in the condenser coils of the sampler is displayed in Figure 4. This insoluble matter is almost entirely composed of mercury halides, of which the chloride is clearly the dominant chemical form. The insoluble nature of the halides combined with the relative intensities of mercury and chlorine fluorescent spectral lines establish the major chemical form of the condensible mercury effluent collected as calomel or Hg_2Cl_2. The spectrum also indicates that Hg_2I_2 and/or the mixed halide HgCl·HgI are also present. It should be noted that iodine was a minor halide constituent in the simulated feed stream used during all phases of ESCM testing (see Table 1).

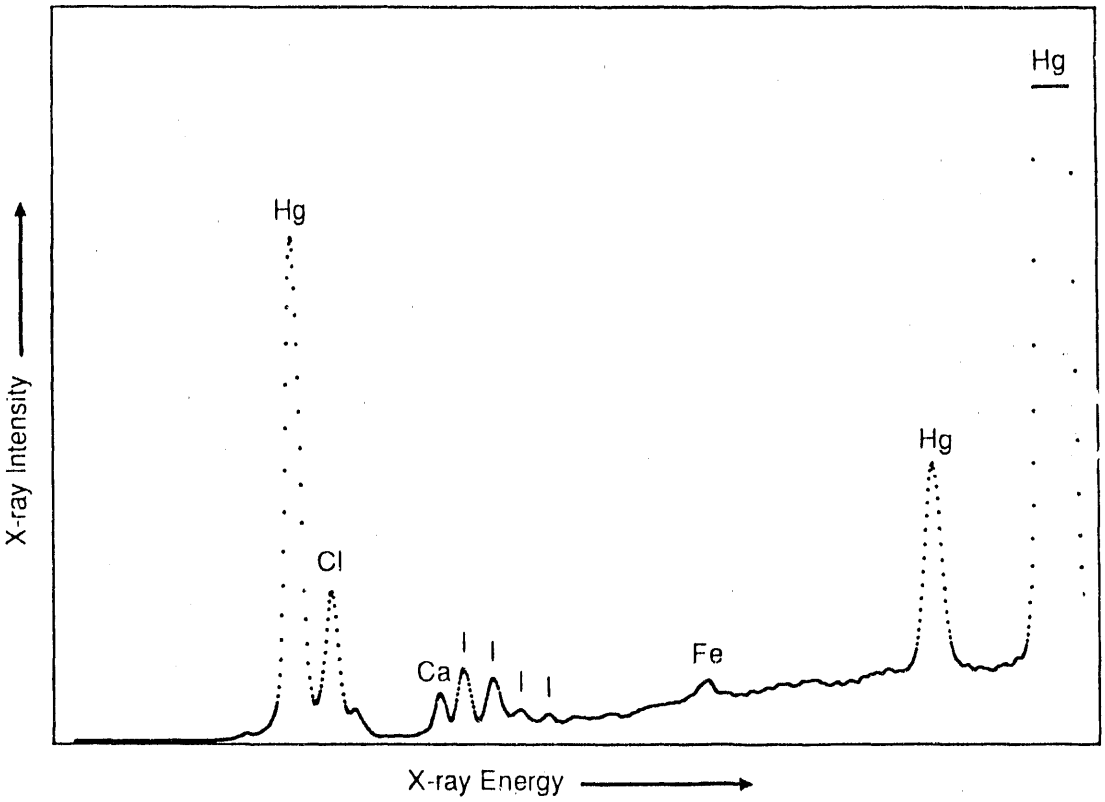

Figure 4. X-Ray Fluorescence Spectrum of Condensed Off-Gas Effluent

The presence of corrosive sublimate, $HgCl_2$, in the condensate sample fraction was also assessed by examining the concentration of soluble mercury in chloride-rich condensate liquor. The ratio of soluble to insoluble mercury in the condensate sample fractions taken throughout ESCM testing are summarized in Table 5. These data clearly show that insoluble Hg_2Cl_2 is the primary mercury-containing effluent species collected by the off-gas sampling system except during high inleakage and low plenum temperature operating conditions. Since oxidizing conditions are required to produce mercury halides from the Hg° and halide components of the feed, increased oxygen concentrations produced by high melter inleakage conditions should result in conditions favoring production of the higher (mercuric) halide; i.e., $HgCl_2$, in accordance with observations made during ESCM test 2. The effect of plenum temperature upon the relative yields of the two mercury halides is more difficult to interpret, but must necessarily be related to the kinetics of mercury halide formation or, more probably, to effective halide oxidation (X_2 production) rates.

Beyond the mercury halides, condensate samples contained little else other than anions formed from aqueous interactions with reactive, acid gas effluents; e.g., X_2, HX, HBO_2, NO_x, and SO_x. No evidence for the presence of HgO was found in any of the samples collected during ESCM testing. Moreover, essentially all mercury effluent collected was chemically combined as a halide. However, the presence of Hg° vapor above all process-generated condensates, detected by environmental mercury analyzers previously described, unmistakably established the presence of elemental mercury in these samples. However, in no instance was macroscopic, agglomerated mercury (spheres) ever observed in condensate sample fractions or, as will be subsequently discussed, in process quench (EVS) tank residues. It is believed that the presence of the Hg° in these samples was primarily due to the disproportionation reaction, governed by the following equilibrium expression:

$$Hg_2^{++} \rightleftarrows Hg^{++} + Hg; \; K = 2 \cdot 10^{-2} \qquad (1)$$

Because the mercurous ion is only moderately stable to disproportionation in solution, any reagent which preferentially reduces

Table 5. Sampler Mercury Effluent Composition

Test Number	Air Inleak kg/h	Plenum C°	Mercury Halide Composition, % Hg_2Cl_2	$HgCl_2$	Hg_2I_2*
1	0.73	750	86	14	Trace
2	6.8	750	38	62	Trace
3	0.73	550	33	67	Trace
4	0.76	740	82	18	Trace
5	0.76	740	85	15	Trace

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*Or mixed halide HgCl-HgI.

(complexation, precipitation, etc.) the activity of Hg^{++} relative to Hg_2^{++} will drive the above reaction to the right. Indeed the tendency for Hg_2Cl_2 to disproportionate is so great that its name, calomel, is derived from a Greek word which means "to blacken." Gradual darkening of condensate sample residues (Hg_2Cl_2) was in fact observed during all ESCM sampling campaigns, further supporting the proposed origin of Hg° in these samples. The very strong vapor signature of this element in all condensate samples collected was a direct result of the extremely large surface area over which the limited (massless) quantities of finely divided mercury were deposited. X-ray diffraction studies of frozen sampler and process condensate residues, which will be discussed in the next section, further confirmed that only microscopic quantities of Hg° were present in these samples.

EVS Scrubbing Liquor Composition

The sampling data just described have shown melter-generated mercury effluent to be in the gaseous state prior to the quenching process. Furthermore, mercury halides were found to be the predominant chemical forms of mercury recovered when the sample stream was quenched. Since these off-gas sampling data cannot conclusively establish that mercury halides were present in the unquenched melter exhaust, it is important to verify that process quenching of the ESCM exhaust will produce the same chemical effluent results that were observed under controlled sampling conditions.

Unlike the differential sampling system that segregated effluents on the basis of their physical states in the unquenched melter exhaust stream, the EVS collects aerosols, condensibles, and chemically scrubbable effluents in a single effluent waste stream--the EVS scrubbing liquid. Because this waste stream is composed of both soluble and insoluble effluents, representative sampling is troublesome, as uniformity of insolubles throughout the bulk of the solution cannot be guaranteed. Nevertheless, samples of the EVS scrub solution effluents were collected and analyzed, assuming uniform stirred tank conditions for ESCM tests 1 through 3. Loss of tank agitation during tests 4 and 5 effectively invalidated the uniform stirred tank approximation and guaranteed nonrepresentative sampling conditions for these last two tests.

Table 6 presents the distribution of EVS mercury effluent between soluble and insoluble forms for the first three ESCM tests. With the exception of the second test, these results are quite comparable to the previously described off-gas sampling results if the soluble and insoluble forms of mercury are assumed to be $HgCl_2$ and Hg_2Cl_2, respectively. Since the chloride ion concentration in EVS solutions was always much greater than the mercury content of these solutions, there is little doubt that the weakly dissociated $HgCl_{2(aq)}$ is the major soluble form of mercury present in EVS solutions.

The insoluble forms of mercury present in the EVS scrubbing liquor were assessed using x-ray diffraction techniques. The results of these studies for all ESCM tests conducted are summarized in Table 7. In all cases Hg_2Cl_2 was found to be not only the major mercury compound present but the major (~90%) crystalline compound in the EVS insoluble residues. Moreover, low-temperature x-ray diffraction examinations of the insoluble matter failed to reveal any evidence

Table 6. EVS Mercury Effluent Characteristics

Sample No.	Percent Insoluble		
	Test 1	Test 2	Test 3
1	94	85	67
2	93	89	72
3	89	86	73
4	88	89	66
5	90	94	52
6	87	88	61
7	86	95	31
8	85	96	59
9		88	43
10		93	45
11		87	27
12		90	25
13			32

Table 7. X-Ray Diffraction Data for Insoluble Residues Collected During ESCM Tests

ESCM-I Condenser Residue			ESCM-I Venturi Residue			ESCM-II Venturi Residue 8/5/83		
Intensity	Interplanar Distance, °A	ID	Relative Intensity	Interplanar Distance, °A	ID	Relative Intensity	Interplanar Distance, °A	ID
3	5.11	?	82	4.19	HgCl	90	4.17	HgCl
3	4.52	HgI	16	3.36	?	3	3.34	?
80	4.15	HgCl	100	3.195	HgCl	100	3.17	HgCl
7	3.48	HgI	10	2.98	?	2	2.96	?
100	3.17	HgCl	15	2.85	HgCl	20	2.83	HgCl
1	2.96	?	45	2.75	HgCl	50	2.728	HgCl
2	2.90	HgI	18	2.53	?	10	2.523	?
10	2.82	HgCl	50	2.35	Al	2	2.348	?
90	2.72	HgCl	23	2.25	HgCl	25	2.247	HgCl
2	2.46	HgI	65	2.08	HgCl	70	2.070	HgCl
17	2.35	Al	100+	2.03	Al	50	1.963	HgCl
20	2.25	HgI, HgCl	20	1.98	HgCl	2	1.842	?
27	2.07	HgCl	40	1.97	HgCl	7	1.757	HgCl
100	2.03	Al	11	1.76	HgCl	24	1.734	HgCl
25	1.97	HgCl	25	1.735	HgCl	2	1.697	?
	1.96	HgCl	10	1.596	HgCl	10	1.588	HgCl

ESCM-II Venturi Residue 8/12/83			ESCM-III Venturi Residue 10/1/83		
Relative Intensity	Interplanar Distance, °A	ID	Relative Intensity	Interplanar Distance, °A	ID
4	5.11	?	100	4.19	HgCl
75	4.16	HgCl	2	3.70	?
3	3.35	?	100	3.20	HgCl
100	3.17	HgCl	2	2.98	?
20	2.83	HgCl	8	2.85	HgCl
55	2.726	HgCl	20	2.75	HgCl
20	2.526	?	5	2.71	?
28	2.244	HgCl	12	2.53	?
70	2.065	HgCl	20	2.26	HgCl
5	1.99	?	30	2.08	HgCl
55	1.975	HgCl	5	1.99	HgCl
	1.963	HgCl	15	1.97	HgCl
2	1.84	?	2	1.84	?
3	1.757	HgCl	3	1.76	HgCl
20	1.731	HgCl	6	1.74	HgCl
4	1.697	?	2	1.70	?
			4	1.595	HgCl

for the presence of elemental mercury in these samples. The composition of suspended matter in EVS scrubbing liquor, summarized in Table 8 further verifies the conclusions drawn by the x-ray diffraction studies of tank sludges, although, as discussed previously, this suspended matter may not be totally representative of tank insolubles. These compositional results clearly illustrate how a minor process feed stream component with a low melter DF can influence secondary waste stream composition and process recycle strategies.

Given the demonstrated chemical similarities between EVS and off-gas samples, Tables 5 and 6 can be directly compared, keeping in mind that the predominant soluble and insoluble compounds of mercury in the EVS samples are $HgCl_2$ and Hg_2Cl_2, respectively. In general, good agreement between sample results is observed except that the proportion of calomel in the EVS waste stream tends to be higher than in corresponding off-gas sampler effluent. This is especially true for ESCM test 2, which involved high inleakage and plenum temperatures. The fact that such a large difference exists between sample results for this test suggests that stable chemical forms of mercury in the unquenched melter exhaust probably did not exist and that the chemical nature of mercury effluent collected may have been a function of quenching conditions. Since the residence time of off-gas effluents was minimized during this test, reaction kinetics may also have been responsible for transitory off-gas compositions and resultant variable effluent results.

Like the mercury effluent collected by the off-gas sampler, the insoluble EVS matter exhibited a very strong vapor pressure of elemental mercury, but no evidence of agglomerated mercury could be found. However, due to the density of elemental mercury, sampling of the scrub solutions may not have been representative for the elemental form of this effluent. Consequently, a search for macroscopic quantities of pooled elemental mercury was conducted within the EVS quench tank. This was accomplished by allowing the scrub solution to settle, pumping out the supernate to within a few inches of the tank bottom, and allowing the remaining water to evaporate. The tank residues were subsequently examined. No sign of the elemental form of this effluent could be found through visual examinations, and low-temperature x-ray diffraction examination of these

Table 8. Major and Minor Feed Components in EVS Suspended Solids, wt%

Element	Test 1	Test 2	Test 3	Test 4	Test 5
Al	0.77	0.45	1.06	0.10	0.00
Cr	0.51	1.09	1.34	1.99	0.60
Fe	33.89	29.30	40.10	46.71	32.87
Hg	51.48	59.91	45.47	43.96	60.58
Mn	4.68	5.02	6.42	0.00	0.00
Na	0.51	0.38	0.60	1.85	1.60
Ni	0.00	3.26	3.69	3.87	3.91
Si	8.15	0.60	1.31	1.51	0.43

residues also failed to detect any significant concentrations of Hg° in these tank samples. Nevertheless, like all process samples, these tank residues exhibited an elemental mercury partial pressure that was nominally equivalent to the temperature-dependent vapor pressure of this element. The impact of these process sources of mercury vapor upon off-gas concentrations of this element will be discussed in the following section.

Off-Gas Concentrations of Mercury Vapor

Since mercury is toxic, controlled effluent mercury vapor concentrations were measured at various off-gas system locations during ESCM testing in order to assess the impact of gas phase losses upon off-gas process requirements. Mercury vapor concentrations were measured at three different off-gas locations: after the venturi in the two-phase off-gas line leading to the condensate tank (site S2), downstream of the quench tank (site S3), and, finally, after the exhaust had passed through a condenser and final HEPA filter (see Figure 2). These sampling points will be referred to as the venturi, quench tank, and HEPA locations, respectively. Table 9 presents a summary of the mercury data collected at these points during tests 2 and 3, which represented typical off-gas conditions during normal processing operations.

The measurements made after the venturi suggest that a near-saturation condition existed in the quenched exhaust for the duration of both ESCM tests. That is, the partial pressure of mercury measured at sampling site 2 was found to be equivalent to the equilibrium vapor pressure of this element at the temperature of the observation. This result is also found to be independent of the range of off-gas flow rates used during LFCM mercury testing. Nominal saturation conditions were also observed at the other two sampling sites, although the concentrations were significantly lower due to reduced off-gas temperatures. Specifically, the exhaust concentrations of this element were nominally determined by the EVS tank and off-gas condenser exhaust temperatures, ~20°C and 5°C, respectively. However, accumulation of mercury-bearing aerosols on the HEPA filter, which will be described later, definitely influenced

Table 9. ESCM Quenched Exhaust Gas Concentrations of Hg

			Hg Concentration and Vapor Pressure Temperature, Torr x 10^3, °C						Venturi Exhaust, °C
			Venturi		Quench Tank		HEPA		
Test	Date	Time	Pres	Temp	Pres	Temp	Pres	Temp	°C
ESCM-2	08/02/83	1000	6.7	41	--	--	--	--	40
	08/03/83	1400	6.5	41	--		0.45	9	40
	08/04/83	1730	4.1	35	1.7	24	0.48	10	40
	08/05/83	1100	5.6	39	1.7	24	0.53	11	41
ESCM-3	08/08/83	2220	3.1	32	0.88	17	0.33	6	32
	08/09/83	1015	2.0	26	1.0	18	0.57	12	30
	08/10/83	0940	2.1	27	1.2	20	0.41	8	30
	08/11/83	1415	1.3	21	0.96	17	0.55	11	30

measurements collected downstream of this device. The simple temperature dependence of these vapor measurements suggests that an off-gas chiller is apparently all that is required to control off-gas mercury vapor emissions.

As previously mentioned, the EVS waste stream solids were found to exhibit a strong Hg° vapor pressure even though only microscopic quantities of mercury were present. In an attempt to determine the influence of the EVS waste stream upon the overall mercury source term, the Hg° concentration in the venturi exhaust was measured as a function of time after melter feeding was terminated at the conclusion of ESCM test #3. The data collected, which are summarized in Table 10, clearly establish that the Hg-loaded scrub liquor is a major off-gas emission source of Hg° vapor effluent and that EVS operations are capable of maintaining saturation conditions in the dual-phase venturi exhaust line independent of the melter source term.

EVS tank exhaust concentrations of Hg° have also been found to be strongly linked to the turbulence associated with tank agitation. Without agitation, the EVS jet spray provides the only intimate contact between mercury-coated tank residues and the off-gas stream. When the EVS tank agitator failed during tests 4 and 5, tank exhaust concentrations of mercury dramatically dropped below the levels previously achieved with the 5°C off-gas condenser. This result demonstrates that the agitated EVS tank was a significantly greater source of mercury vapor than was the quenched melter exhaust stream. It should be noted that the large effect of tank agitation upon off-gas concentrations of mercury in these tests was somewhat enhanced by the fact that EVS tank inleakage significantly (~2X) diluted the process exhaust stream composition before measurements were made. However, this condition in no way alters the conclusions drawn about the importance of the agitated EVS tank to the overall off-gas concentrations of mercury vapor.

Since the mercury in the melter feed stream was primarily in an uncombined, elemental form, the impact of the agitated feed tank upon mercury vapor concentrations in the vessel ventilation system was also investigated. Unlike the process off-gas system, much greater

Table 10. Time Dependence of Hg Vapor Pressure in the Venturi Exhaust Upon Feed Termination

Time	Hg Partial Pressure, Torr x 10^3	Exhaust, °C	Vapor Pressure Temp, °C
0935	2.2	27	27
0944	2.2	27	27
1004	2.1	27	27
1025	2.0	26	26
1125	1.7	24	24
1315	1.5	22	22
1415	1.8	25	25
1650	1.6	23	23

tank ventilation flow rates were used in characterizing $Hg_{(g)}$ in the vessel vent system. The results obtained from these measurements over a two-day period are summarized in Table 11. As with the EVS tank, the agitated feed tank's exhaust exhibited a partial pressure of mercury vapor that approached the characteristic vapor pressure of this element under low flow conditions. However, with an air purge of ~300 scfm, mercury vaporization rates were insufficient to maintain saturation conditions in the vessel exhaust stream. The average partial pressure of mercury under these high flow rate conditions is equivalent to saturation conditions at 11°C. As shown in Table 11, the observed concentrations are purge-rate-dependent variables, and reductions in tank ventilation rates will be accompanied by corresponding increases in exhaust concentrations of $Hg_{(g)}$.

Process Accumulation Sites of Mercury-Containing Effluent

Physical examinations of the ESCM and its associated off-gas processing system were conducted at the conclusion of the five LFCM processing tests in order to identify unanticipated sites of accumulation of mercury and its associated compounds. Although mercury and its halogenated compounds have significant vapor pressure at or below LFCM operating temperatures, cool surfaces within the melter cavity and/or melter off-gas system can condense gaseous mercury effluent and act as a sink or accumulation point for mercury and its volatile compounds. Since the condensation of volatile can and will produce small, highly penetrating aerosols, the final off-gas HEPA filter can also become a significant accumulation site for mercury-bearing compounds. Consequently, the melter, the melter-EVS off-gas line, the quench tank, the quench tank exhaust line, and the final HEPA filter were all examined to establish potential buildup sites of mercury and to measure the magnitudes of mercury accumulations, if possible.

Melter

In order to prepare the ESCM melter for visual examination at the conclusion of the fifth and final test, the remaining glass in the melter had to be gravity drained through an inductively heated freeze valve located in the bottom of the melt chamber. To accomplish this, plenum heaters were used to heat the melter cavity to ~1000°C after ancillary equipment (such as the feed nozzle) was removed. A water-cooled condenser was placed in the melter exhaust port to collect any mercury volatilized from the melter during the

Table 11. Feed Tank Hg Emissions

Sampling Period		Feed Tank Exhaust Conditions		Vapor Pressure	Exhaust Flow,
Date	Time	Hg°, Torr x 10^3	°C	Temperature °C	scfm
10/11/83	1545	0.44	30	10	330
10/12/83	1650	1.4	31	22	low
10/12/83	1730	0.79	31	15	330
10/12/83	1738	0.86	31	16	330
10/12/83	2000	0.61	31	13	330
10/13/83	1600	0.64	28	13	330

heatup and/or draining process, and an environmental mercury monitor was used to measure mercury vapor emanating from the molten glass as it was discharged from the melter. The melter itself was disassembled and destructively examined for mercury content subsequent to the glass draining process.

Table 12 summarizes the location and quantity of mercury found in the melter. The largest source of mercury found in the melter examination was observed in the melter exhaust condenser and represents the quantity of mercury that resided within the melter after processing that distilled away during the draining process. As with literally all samples collected during ESCM testing, the volatilized mercury collected was primarily chemically combined, presumably as a halogen. However, the sample exhibited a strong elemental mercury vapor pressure, but no evidence for macroscopic quantities of agglomerated mercury could be found in the condensed effluent. Nozzle and viewport deposits collected after processing but before melter heatup and glass drainage accounted for nominally 5% of the sample mass of mercury collected from the melter.

No elemental mercury vapor could be detected emanating from the molten glass draining from the melter. This was also found to be the case for glass transfers conducted during all phases of ESCM processing of mercury-containing feeds. This result is in accord with previously described compositional data that found no detectable quantities of this element in the vitreous glass product.

Very little evidence of mercury was found within the melter itself after glass drainage. Only nozzle and off-gas ports in the melter lid exhibited any detectable elemental mercury vapor pressure and this emanated from massless deposits. All cracks and penetrations in the melter refractories which extended out to the melter shell were closely examined for deposits of condensed mercury. No evidence of mercury accumulations could be found and no characteristic vapor emanations could be detected from any site other than the nozzle locations previously described.

Since the total quantity of mercury residing in the melter after processing is clearly an insignificant fraction of quantity fed to it during the three processing campaigns (1.5 kg), it is clear that an LFCM is not a significant point of accumulation for mercury or its associated compounds. Moreover, the melter effluent characterization and post-test examination studies have shown that feed component mercury does not present a credible electrical shortage threat to LFCM operations.

Melter-EVS Off-Gas Jumper

The process exhaust line conveying melter off-gases to the EVS quench scrubber was operated at ~350°C throughout all phases of ESCM testing. Consequently, condensation of mercury compounds was not a problem over most of its length. However, the temperature of this line at the inlet of the EVS quench scrubber was sufficiently low to condense volatile mercury halides which, once formed, acted as accumulation sites for other airborne condensed effluent. Off-gas line obstructions instigated by mercury effluent condensation were observed during all phases of ESCM testing and are a reflection of

Table 12. Mercury Content of Post-Test Samples

Source	Sample		Location	Composition	Hg, g	% Total
	Number	wt,g				
Melter	1	0.213	Lower Feed Nozzle	50% HgCl, 50% Fe_3O_4	0.0064	4×10^{-3}
	2	1.609	Upper Feed Nozzle	50% HgCl, 50% Fe_3O_4	0.0193	1.1×10^{-2}
	3	1.232	Feed Nozzle Port	SiO_2, Weak HgCl and Fe_3O_4	0.0296	1.7×10^{-2}
	5	39.04	Viewport	--	0.109	6.3×10^{-2}
	9	--	Thermal Well Top 2"	HgCl	--	--
	10	--	Thermal Well Top 6"	Fe_3O_4 and NaCl	--	--
	11	--	Thermal Well Port	HgCl, Moderate Fe_3O_4	--	--
	19-21	--	Exhaust Quencher	--	3.68	2.1
Quench Tank	4	10.8	Bottom Drain Residue	--	0.824	0.4
	30	--	Tank Bottom Residue	--	141.01	81.2
HEPA	31	--	Filter Sheets	--	28.00	16.2
			TOTALS		173	100

the fact that a temperature transition between 350°C and 40°C has to occur at the off-gas inlet of the process quench scrubber. Because of this and the insoluble nature of calomel, mercury in LFCM feeds will exacerbate off-gas blockage problems associated with the LFCM technology.

Use of high off-gas velocities has been somewhat successful in reducing off-gas effluent deposition rates with non-mercury-containing feeds. Air or steam injection used to boost melter exhaust velocities may, along with resultant volumetric dilution, also be useful in controlling mercury deposition rates, but unless the observed process chemistry changes, mercury-containing feeds will create additional unquenched off-gas system challenges.

Condensate Tank

An ejector venturi was used to scrub and quench the melter exhaust during the LFCM testing with mercury-containing feed. Analysis of melter exhaust and EVS scrub solution compositions has shown calomel, Hg_2Cl_2, to be a major melter effluent and a primary EVS condensate tank insoluble, respectively. If a nominal EVS DF of 10 for ostensibly insoluble iron effluent is assumed,[1-2] the melter DF data for mercury and iron, when combined with the x-ray diffraction results of condensate tank sludges, suggest an EVS scrubbing efficiency of >90% for mercury effluent. Post-test examination of the quench tank contents confirmed this vessel to be a major accumulation point for melter-generated mercury effluent (see Table 12). The quantity of mercury-rich sludge found in this vessel was not at all unusual in that the tank's contents were pumped out without the benefit of tank agitation; as a result, the settled solids quantified during post-test examination represented a large portion of the insoluble solids collected during the final ESCM test. These insoluble tank residues were found to be friable and easily suspended, and no evidence of macroscopic quantities of high-density elemental mercury could be found in these settled solids.

Quench Tank Exhaust Line

The exhaust line leading from the quench tank to upstream processing equipment was examined for mercury-containing residues. This process line was extremely clean; no collectible macroscopic residues were present. This finding is not at all surprising in that the condensation process, which has been shown to be almost entirely responsible for generating mercury-bearing airborne particulate matter, produces submicron aerosols which have no appreciable tendency to settle or accumulate on surfaces.

Final Off-Gas HEPA Filter

Since vapor condensation can act to produce penetrating submicron aerosols, the final HEPA filter used in the ESCM off-gas processing system was examined to determine the total mass of mercury that successfully penetrated the EVS quench scrubber as a submicron aerosol throughout all phases of ESCM testing. Samples of the filter media were extracted across the HEPA and were subsequently analyzed for mercury. The average value from these samples was used to estimate the total mercury loading on the HEPA filter. These results are also listed in Table 12. Since ~1.5 kg of mercury was fed to the melter during the five tests conducted, submicron aerosol transport of mercury is not a major source of off-gas system losses of mercury and its associated compounds, although it is significantly greater than elemental vapor losses. Based upon the observed filter loading, feed composition data, and off-gas vapor pressure measurements, the average mercury DF achieved by the EVS throughout all phases of ESCM testing is estimated to be ~40. This corresponds to an EVS removal efficiency of 97%, which is in accord with previous estimates based upon condensate sample compositions.

Discussion/Summary

The processing and off-gas emission characteristics of mercury in LFCM feeds have been studied under various operating conditions, including melter inleakage, plenum temperature, and feed composition. The results of these studies have shown typical LFCM-produced borosilicate glasses to have no detectable capacity for mercury. Essentially all the mercury fed to the melter was lost to the process off-gas system in the gaseous state; as a result, mercury, on the average, accounted for greater than 85% of condensible process off-gas system effluent losses. Mercury halides are the primary chemical forms of mercury condensed and collected after process exhaust quenching. Since chlorine was the major halogen in the melter feed, mercury effluent was predominantly composed of its two chloride forms.

Although $HgCl_2$ is the thermodynamically favored chloride over the entire processing temperature range,(4) Hg_2Cl_2 was found to be the predominant halide condensed and collected after off-gas quenching except under low plenum temperature conditions. Since Hg(II) is readily reduced to the Hg(I) state by elemental mercury if stoichiometric quantities of oxidants are not available, the instability of mercury oxides in combination with limited concentrations of halogens is most likely responsible for the thermodynamically unexpected result. In any event, the fact is that Hg_2Cl_2, on the average, was the predominant chemical form of mercury that was condensed and collected by the process quench scrubber during ESCM testing. This result was also observed previously when a similar waste stream was solidified using the related spray calciner in-can melter technology.(5) Unlike the present result, however, small quantities of agglomerated elemental mercury were identified in quench scrubber insoluble residues. Only finely divided surface deposits of mercury were observed in process samples collected and examined in these LFCM tests. The highly dispersed nature of these deposits strongly suggests that the source of Hg° in these samples is due to disproportionation of Hg(I) (see Equation 1).

In spite of the fact that large sources of elemental mercury are not generated by the processing of mercury-containing feed, the quenched melter exhaust stream was found to be saturated with mercury vapor under normal operating conditions. The agitated quench (condensation) tank was found to be the major source of mercury vapor downstream of the EVS. However, it was also found that the partial pressure of mercury in the off-gas stream could be effectively and predictably controlled through use of an off-gas chiller.

The major point of accumulation of LFCM processed mercury is the process condensate tank. Overall, the EVS collected 97% of the mercury processed (DF ≈ 40) during ESCM testing, and on the average most of this was in the form of insoluble Hg_2Cl_2. Since the LFCM process has no capacity for incorporating mercury (melter DF ≈ 1), Hg_2Cl_2 always represented the largest fraction of insoluble matter in ESCM condensate tank residues. Typically, 90% of the insoluble crystalline matter collected in the EVS condensate tank during ESCM testing was specifically due to Hg_2Cl_2.

The proportion of soluble $HgCl_2$ produced varied with processing conditions. With low plenum temperatures, ~70% of the ESCM mercury effluent that was collected in the EVS condensate tank and in off-gas samples was in the form of $HgCl_2$. Although off-gas sampling data indicated similar effluent compositions under high inleakage conditions, condensate tank compositional data indicated otherwise. These differing results suggest that quenching conditions may influence the chemical composition of the mercury halides collected. However, the process realities are that except for the test conducted without auxiliary lid heat, Hg_2Cl_2 was the primary chemical form of mercury collected by the process quench scrubber, independent of all other operating conditions.

Acknowledgments

The Pacific Northwest Laboratory is operated for the U.S. Department of Energy by Battelle Memorial Institute under Contract DE-AC06-76RLO 1830.

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