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Test Summary Report INEEL Sodium-Bearing Waste Vitrification Demonstration RSM-01-2

R. W. Goles*
J.A. Del Debbio**
R.J. Kirkham**
B.D. MacIsaac*
J. A. McCray**
D. D. Siemer**
N.R. Soelberg**

*Pacific Northwest National Laboratory
**Idaho National Engineering and Environmental Laboratory

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Abstract

A research-scale, liquid-fed, ceramic-melter was used to conduct a flowsheet evaluation of a nonradioactive surrogate of sodium-bearing waste currently being stored in underground tanks at the Department of Energy's Idaho National Engineering and Environmental Laboratory. During this 120-h melter test, the processing characteristics of a glass formulated to have a high sulfur capacity were evaluated with and without reductant (sucrose or glycolic acid) additives. Beyond processing rates, this integrated melter/off-gas system demonstration test evaluated the impacts of reductant type (if used) and concentration upon 1) the partitioning of volatile (sulfur, mercury, and the halogens) and nonvolatile effluents, 2) the oxidation state of the melter glass, 3) the reduction of waste constituent nitrate, 4) the composition of secondary waste streams, and 5) the durability of the melter's glass product.

Summary

Over several decades, site operations, at what is now the U.S. Department of Energy's (DOE's) Idaho National Engineering and Environmental Laboratory (INEEL), have included nuclear reactor testing, reprocessing of spent nuclear fuel, and the storage, treatment, and disposal of the resultant radioactive and mixed wastes generated. Liquid, acidic, and radioactive high-level waste (HLW) and sodium bearing waste (SBW) from spent-fuel reprocessing operations have for the most part been calcined in the New Waste Calcining Facility (NWCF) and the earlier Waste Calcining Facility (WCF) to produce a dry granular waste form that is safer than liquid waste to store. However, about a million gallons of SBW remains uncalcined, and this liquid mixed waste, stored in tanks, does not meet current regulatory requirements for long-term storage and/or disposal. As a part of the Settlement Agreement between DOE and the State of Idaho, the tanks currently containing SBW are to be taken out of service by December 31, 2012, which requires the removal and treatment of the remaining SBW.

Several potential options have been proposed for treating the SBW. Of those considered, vitrification received the highest weighted score against the criteria used. Beginning in Fiscal Year 2000, the INEEL HLW program embarked on a program for technology demonstration and development that would lead to the conceptual design of a vitrification facility, based upon the liquid-fed melter technology, in the event that vitrification is the preferred alternative for SBW disposal. This program consists of several separate activities that include, among others, waste-form development, process feed-stream design, and melter vitrification demonstration testing of the nonradioactive, surrogate SBW flowsheet. The second of two FY 2001 melter flowsheet tests conducted at Pacific Northwest National Laboratory (PNNL) in support of INEEL's vitrification facility design is discussed below.

PNNL's Research-Scale Melter (RSM) was used to conduct these initial melter-flowsheet evaluations. The RSM is a small (1/100-scale, Defense Waste Processing Facility basis) joule-heated melter that is capable of processing melter feed on a continuous basis. This capability is key for:

- developing/evaluating process flowsheets
- characterizing relationships between feed composition and the properties of the final glass produced
- establishing the fate and behavior of process effluent.

This melter system's capability to produce glass in a continuous manner is also essential for estimating the behavior of a full-scale system. Moreover, the size of the RSM allows the impacts of process variables upon melter performance or glass quality to be quickly and efficiently evaluated without undue expense or waste generation.

The experimental scope of this initial, 5-d, 120-h, SBW vitrification test was to evaluate the:

- processing characteristics of the newly formulated SBW-22 surrogate melter feed formulation with and without reductant (sucrose or glycolic acid) additives
- effectiveness of SBW-22 glass to incorporate waste-stream sulfur
- suitability of using sugar or glycolic acid as glass oxidation-state modifiers and nitrate reductants

- emission characteristics of the melter with a special emphasis on volatile effluents such as mercury, sulfur, and the halogens
- impact of flowsheet reductants upon secondary waste stream compositions
- efficacy of a real-time glass oxidation state monitor
- quality and durability of the process's vitreous waste-form product.

During the 120 h of experimental testing, the processing characteristics of a single melter feed formulation, designated SBW-22, at a fixed 20 wt% waste loading was evaluated with and without reductant additives. On the basis of laboratory crucible-scale screening tests, the reductants evaluated during melter testing were limited to sugar and glycolic acid. In addition to reductant chemical type, the impacts of varying reductant concentrations upon processing rates, sulfur glass retention, molten-salt accumulations, glass oxidation state [Fe^{+2}:Fe^{+3}], and glass product durability were evaluated.

The melting kinetics of the SBW-22 feed formulation using a sugar reductant was found to be similar to that of the previously evaluated SBW-9 formulation (Goles 2001), but its cold-cap characteristics were somewhat different. Specifically, a much more developed structure was produced by the SBW-22 formulation, which, upon feed interruption, required significantly more time to burn off than was observed for SBW-9. As in previous testing, it was found that sugar feed concentration could be effectively used to control the oxidation state of the melter's product glass. Specifically, RSM-2 data suggest that SBW-22 sugar concentrations as high as 175 g/L-SBW are adequate for maintaining acceptable glass oxidation conditions: Fe^{+2}:Fe_{tot} < 0.3. Foaming, caused by an inadvertent over-reducing condition, was the only factor that adversely affected processing conditions involving sugar reductant.

The use of glycolic acid reductant with SBW-22 produced a much more developed cold-cap structure than sugar, although it failed as an effective oxidation-state modifier. Due to its ineffectiveness in controlling melter glass oxidation state, relatively high loadings of this reductant were used that further increased cold-cap accumulation and concomitant process-rate reductions. In addition, excessive gas generation at the bottom of the cold cap created glass-foaming conditions that reduced heat-transfer rates and further increased cold-cap accumulations.

Feed was also processed without any reductant modifier during RSM-2 testing. In all cases, inferior processing conditions resulted. Under these strong oxidizing conditions, glass foaming created very significant processing difficulties. In particular, a thick and very persistent insulating foam/cold-cap layer was created that, at the conclusion of an extended campaign, had to be physically broken up and stirred into the bulk glass before subsequent testing conditions could commence. On the basis of the two campaigns conducted, it appears that long-term continuous processing of SBW-22 without a reductant modifier is not a reasonable option.

Beyond controlling glass oxidation state, flowsheet reductants are also used to accelerate feed processing by reducing nitrate to volatile compounds that can be efficiently exhausted from the process. Although the volatile nitrogen oxides produced were directly measured by continuous gas monitors, the degree to which reduction to elemental nitrogen occurs had to be indirectly inferred from process conditions and gas composition data. The results obtained from nitrogen mass balance calculations suggest that on the average, 15% of the nitrogen fed to the melter during RSM testing was reduced to its elemental state. Furthermore, it appears that the reductive N_2 yield is related to reductant concentration.

Melter glass production rates varied from 6.2 to 9.4 lbs/h/ft^2 for the various feed batches processed. These values comfortably exceed the reference (cold-lid) Liquid Fed Ceramic Melter (LFCM) design production rate of 4 lbs/h/ft^2 that is often quoted and used for flowsheet and equipment sizing estimates (Perez 2001). Indeed, this reference-normalized production rate is exceeded even when projections are based upon the overall average feed rate data (7 lbs/h/ft^2) that are inclusive of all melter idling and batching periods.

Average measured Joule heating power was used with corresponding batch feeding rates, reductant loadings, and heat of combustion information to derive specific process energy requirements for SBW feeds. The average value derived for all batches processed, 4.9 kW-h/kg, is slightly greater than typical energy requirements for slurry-fed, Joule-heated ceramic melters (2 to 4 kW-h/kg of glass produced) but is significantly less than that derived during previous testing with the SBW-9 formulation. Recognizing that much of the power required in processing slurry feeds is consumed by boiling away water, the higher than average specific-energy requirement for vitrifying SBW-22 noted above is most likely due to the water weight fraction differences.

The partitioning behavior of sulfur was assessed by conducting post-test analysis of all collected process streams. The results of this assessment indicate that > 95% of the sulfur processed was incorporated within the melter's vitreous product. Indeed, the mass balance results for the overall test suggest that only ~ 1% of the sulfur fed to the RSM partitioned to the off-gas system and collected in secondary waste streams. Moreover this mass closure data for sulfur further validates off-gas monitoring and sampling data that did not detect any significant sulfur partitioning as SO_2 or SO_3. Although process data suggests that higher than average sulfur off-gas partitioning occurred when the highest feed concentrations of sucrose were used, the SO_2 presumably produced, is not persistent and is, apparently, quickly oxidized under RSM processing conditions.

Monitoring for molten salt accumulations (> 90% alkali & alkaline sulfates) was conducted throughout all phases of RSM testing. Initial observations after ~ 15 hrs of processing revealed the presence of small molten salt phase pools on the glass melt surface. Subsequent observations suggested a progressive and continual decline in number and size of observable molten-salt-phase pools. Although never completely absent, the presence of molten salt present at the end of the test was quite difficult to discern.

For the surrogate SBW melter feed used during RSM testing, CO_2 and NO_x (specifically NO) were the major non-condensable (~ 25°C) gases produced by the vitrification process. The combustible gases CO and H_2 were also detected, but at much lower concentrations: 0.08% and 0.03%, respectively. These concentrations are well below the lower flammability limits of these combustible gases, 4.65% for H_2 and 15.5% for CO.

The responses of the total hydrocarbon (THC) analyzer indicated that hydrocarbon compounds with significant room-temperature vapor pressures were present in melter exhaust throughout most periods of testing. Although the off-gas concentrations of these thermal byproducts were relatively low (< 100 ppm on the average), they were, not surprisingly, functionally related to SBW sugar loadings. It also appears that overfeeding and abrupt introduction of feed material into the hot melter are responsible for many of the THC concentration spikes observed during RSM testing.

Condensed-phase effluents were also monitored during SBW melter testing. The melter's aerosol mass decontamination factors (DFs), as measured by isokinetic filter catches, were determined for each of the distinct feeds processed. These melter aerosol mass DFs, which ranged from 30 to 200, were reflective of the uniquely different processing conditions created by the feeds being processed. The sugar-containing feed stream created the highest off-gas partitioning behavior, while the glycolic-containing feed produced the lowest. However, cold-cap growth during partitioning measurements may have artificially elevated the aerosol DF associated with the glycolic-containing, feed-processing campaign.

Melter partitioning for individual feed components was also derived from the off-gas sampling and secondary waste-stream data. With the exception of boron, mercury, and the halogens, essentially all feed constituents (excluding C, N, H_2O, etc) were found to be primarily in a condensed state downstream of the film cooler. Overall, the element-specific DFs recorded during RSM-2 are reasonably close to general expectations and are generally consistent with previous SBW melter-testing results. However, the cesium DFs recorded during the current test, averaging ~ 30, are much more consistent with full-scale expectations than the significantly lower values (~ 5) recorded during the previous RSM/SBW test.

Of the volatile melter effluents mentioned above (excluding C, N, and H_2O), mercury exhibited the highest volatility and overall loss rates (DF~ 1). Only during strongly oxidizing conditions was there evidence (~ 5%) of nonvolatile chemical forms of mercury (HgO) under operating off-gas system temperatures (150°C). When feed reductant was used, mercury off-gas effluent was primarily (90 to 98%) in the elemental state. Only under oxidizing conditions was there any significant indication (30%) of other volatile chemically combined forms of mercury being present ($HgCl_2$).

The halogens also exhibited high volatility loss rates during the current test. Average DFs < 10 were recorded for this group of elements. Although only volatile, gas-phase, halogen DFs were measured during this test, it does not appear that aerosol contributions to overall losses would be very significant, given the high volatilities exhibited by these elements.

The evaluation of the secondary waste streams generated during RSM testing revealed significant differences in scrub-solution elemental concentrations when glycolic acid was used as a feed-stream reductant. Specifically, more organic and inorganic carbon effluent was recovered in off-gas waste streams, a higher fraction of captured effluents were collected by the high-efficiency mist eliminator (HEME), and in general there was a lower fraction of undissolved solids (primarily composed of SiO_2 and Fe_2O_3). The halogens as a group comprised the highest percentage of feed elements collected in the scrub solutions. Fluorine was the highest followed by iodine and chlorine. Mercury was the only other element collected in the scrub solution at greater than 10% of that in the feed.

Representative glass samples generated throughout the duration of RSM-2 testing were subjected to standard durability tests. Specifically, glass samples were subjected to both product consistency test (PCT) (ASTM 1997) and toxicity characteristic leach procedure (TCLP 1992) leach-testing protocols. The PCT results suggest that all RSM-2 glasses were more durable than the standard environmental assessment glass to which they were compared. Furthermore, corresponding TCLP tests indicate that all SBW-22 glasses, even the highly reduced ones, conform to all existing Resource Conservation and Recovery Act (RCRA) land-disposal limits (40 CFR 268).

A real-time electrochemical method for monitoring glass oxidation state was also evaluated during RSM-2. The results obtained were inconclusive due to the glass dissolution of electrode sheathing material. Proper operation of the reference electrode required controlling localized conditions at the electrode tip. Deterioration of the electrode sheathing material prevented the required control conditions to be sustained over a meaningful evaluation period.

During SBW melter-flowsheet evaluation studies, 90-L of SBW simulated waste having a total mass of 110 kg were successfully processed by the RSM, producing 22 L of glass having a total mass of 55 kg. As a result, an overall SBW waste-volume reduction factor of 4.2 was achieved during the current test. This factor is lower than that recorded (7.6) during the previous RSM/SBW melter test because of the significantly lower waste loading used with the SBW-22 formulation during RSM-2 testing.

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Acronyms

AA	atomic adsorption
APEL	Applied Process Engineering Laboratory
CEM	Continuous Emissions Monitor
EA	environmental assessment
DC	direct current
DF	decontamination factor; ratio of input to output rate
DOE	U.S. Department of Energy
dscm	dry standard cubic meter
DWPF	Defense Waste Processing Facility
EPA	U.S. Environmental Protection Agency
EVS	ejector venturi scrubber
FY	Fiscal Year
GC	gas chromatograph
HEME	high-efficiency mist eliminator
HEPA	high-efficiency particulate air (filter)
HLW	high-level waste
iC	inorganic carbon
oC	organic carbon
ICP-AES	inductively coupled plasma-atomic emission spectrometer
INEL	Idaho National Environmental Laboratory
INEEL	Idaho National Engineering and Environmental Laboratory
INTEC	Idaho Nuclear Technology and Engineering Center
LFCM	Liquid Fed Ceramic Melter
LOD	loss on drying
LOI	loss on ignition
MOG	melter off-gas
ND	not detected/measured or no data available
NRTS	Nuclear Reactor Testing Station
NWCF	New Waste Calcining Facility
PCT	Product Consistency Test

PLC	Programmable Logic Controller
PM	particulate matter
PNNL	Pacific Northwest National Laboratory
ppm	parts per million
ppmv	parts per million by volume
POG	process off-gas
RCRA	Resource Conservation and Recovery Act
RPM	revolutions per minute
REDOX	reduction oxidation
RSM	Research-Scale Melter
SBW	sodium-bearing waste
scfm	standard cubic feet per minute
SCR	silicon-controlled rectifier
SEM	scanning electron microscope
SRTC	Savannah River Technology Center
STP	standard temperature and pressure
TCLP	toxicity characteristic leach procedure
TDS	total dissolved solids
TFF	Tank Farm Facility
THC	total hydrocarbon
TOE	total operating efficiency
UTS	Universal Treatment Standard
UV	ultraviolet
VOC	volatile organic compound
WCF	Waste Calcining Facility

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7.5. Stream-Dependent % Accumulation Per Liter of SBW Processed 7.9

1.0 Introduction

Over several decades, operations at the Department of Energy (DOE) Idaho National Engineering and Environmental Laboratory (INEEL, formerly called the Idaho National Engineering Laboratory, INEL, and before that the Nuclear Reactor Testing Station, NRTS) have involved nuclear reactor testing, reprocessing of spent nuclear fuel, and the storage, treatment, and disposal of the resultant radioactive and mixed wastes generated. Liquid, acidic, and radioactive, high-level waste (HLW) and sodium-bearing waste (SBW) from spent-fuel reprocessing operations have been temporarily stored at the Idaho Nuclear Technology and Engineering Center (INTEC) Tank Farm Facility (TFF). All of the stored HLW and some of the SBW have been calcined in the New Waste Calcining Facility (NWCF) and the earlier Waste Calcining Facility (WCF) to convert the liquid waste into a dry granular calcine that is safer to store. DOE determined to close the NWCF calciner in Fiscal Year (FY) 2000 rather than upgrade and permit this facility to meet new regulatory requirements, in part because even the calcine is not expected to meet long-term disposal requirements.

The TFF presently contains about 3.8×10^6 L (1-million gal) of SBW that was not calcined. The SBW is an aqueous, highly acidic (1 to 3 molar nitric acid) solution containing dissolved and suspended radionuclides, heavy metals, and other species, including halogens. This waste is a listed, mixed waste, containing radionuclides, hazardous characteristics (corrosivity and characteristic metals), and small amounts of listed organic constituents. This liquid mixed waste, stored in tanks, does not meet current regulatory requirements for long-term storage or disposal.

In January 1990, the U.S. Environmental Protection Agency (EPA) issued to DOE a Notice of Noncompliance because the TFF did not meet the secondary containment requirements of the Resource Conservation and Recovery Act (RCRA). As a part of the Settlement Agreement between DOE and the State of Idaho, the TFF tanks are to be taken out of service by December 31, 2012. An obvious element of the TFF tanks closure is the removal and treatment of the remaining SBW.

Several potential options have been proposed for treating the SBW. Of those considered, vitrification received the highest weighted score against the criteria used. Beginning in FY 2000, the INEEL HLW program embarked on a program for technology demonstration and development that would lead to conceptual design of a vitrification facility, in the event that vitrification, based upon the liquid-fed melter technology, is the preferred alternative for the SBW. This program consists of several separate activities that include, among others, waste-form development, process feed-stream design, and melter vitrification demonstration testing of the nonradioactive, surrogate SBW flowsheet.

This summary report documents the second of two FY 2001 melter flowsheet tests conducted at the Pacific Northwest National Laboratory (PNNL) in support of INEEL's vitrification facility design. Specifically, PNNL's Research-Scale Melter (RSM) was used to evaluate 1) the processing characteristics of the SBW-22 glass formulation under oxidizing, slightly reduced, and reduced processing conditions using two different chemical reductants, 2) the capability of the SBW-22 glass formulation to accommodate the sulfur present in a simulated SBW melter feed stream at 20% waste-loading conditions, 3) the off-gas-emission characteristics of the melter, 4) the physical/chemical fate and off-gas behavior of important feed constituents, including Hg, S, the halides, hazardous elements, and radioactive surrogates,

5) the ejector-venturi wet (acid) scrubbing efficiencies for Hg, S, the halides, and aerosolized feed material, 6) the effectiveness of sugar and glycolic acid as a glass-oxidation-state modifier and a nitrate reductant, 7) the quality and durability of the process's vitreous waste-form product, and 8) the performance of an online glass oxidation-state monitor.

2.0 Test Objectives

The primary objective of RSM testing was to characterize the melter-process flowsheet based upon SBW waste and a target glass composition. The targeted vitreous-product composition, SBW-22, was established from a series of laboratory crucible tests involving SBW surrogate material and suitable glass-forming additives that were chosen to maximize the sulfur capacity of the vitreous product. To avoid the possibility of exceeding the solubility limit of sulfur, the RSM-01-2 test was conducted at a single 20% waste-loading level.

Although waste loading was an invariant test parameter, reductant type and concentration were parameterized during RSM-01-2. Specifically, various concentrations of both sugar and glycolic acid were used as glass oxidation-state modifiers and nitrate reductants. In addition, processing campaigns were also conducted in the absence of reductant. The objective was to create processing conditions that ranged from strongly oxidizing, where the Fe^{+2}/Fe_{tot} ratio of the product glass was 0, to the maximum acceptable reducing conditions ($Fe^{+2}/Fe_{tot} = 0.3$).

Given that the scale of the RSM is ideally suited for conducting parametric flowsheet assessments of processing conditions and glass-product quality, the experimental objectives of the second FY 2001 RSM test involving a surrogate formulation of INEEL's SBW were established to:

1. Determine the best reasonably achieved melt rate and melter-system performance under three different melter redox conditions, using two different reducing agents.

2. Confirm the presence or lack of a separate sulfate layer during all operating conditions. Perform corrective actions if, in the opinion of the test leader, the presence of a sulfate layer becomes excessive.

3. Evaluate the fate and speciation of mercury in the feed mixture for the different melter redox conditions and reductants.

4. Determine the fate of the surrogate SBW feed constituents, including the reductant, halides, nitrates, sulfur, metals that are hazardous or are surrogates for hazardous metals, and elements that are surrogates for radionuclides.

5. Characterize wet scrubber performance for scrubbing water, Hg species, halides, NO_x species, SO_2, and particulate matter, under the different melter operating conditions, but at a single specified scrubber pressure drop.

6. Determine the composition, properties, and amounts of all process streams, including the product glass, scrub solution (including dissolved and undissolved solids), off-gas (including particulate matter [PM] in the off-gas), and PM deposits recovered from the off-gas piping.

7. Measure leachability (based on toxicity characteristic leach procedure [TCLP] testing) and oxidation state (based on the Fe^{+2}/Fe_{tot} ratio) of the product glass, as it is constrained to meet waste-acceptance criteria for HLW borosilicate glass.

8. Establish glass production rates and power requirements based on stable operation at maximum feed rates under all reductant test conditions.

9. Demonstrate and determine the performance of an online redox electrode compared with the traditional wet-chemistry redox analysis technique.

Testing objectives, sampling and data-recording requirements, quality assurance requirements, and system configuration are documented in the RSM Test Plan that was prepared and approved before the start of testing. A copy of the test plan is presented in Appendix A for reference.

3.0 RSM System Description

Pacific Northwest National Laboratory's RSM facility is located in the Applied Process Engineering Laboratory (APEL) building in Richland, Washington. Figure 3.1 is a photograph of the RSM system as it nominally appeared during melter testing, and Figure 3.2 schematically illustrates the system components and their relationships to one another. Because mercury was part of the RSM-01-2 (henceforth referred to as RSM-2) flowsheet evaluation test, a temporary enclosure was constructed around the RSM to contain and exhaust any melter emissions created by inadvertent melter pressurizations. A plan and elevation view of this enclosure is illustrated in Figure 3.3, while exterior and interior photographs of this walk-in hood appear in Figures 3.4 and 3.5, respectively.

The RSM processing system provides a continuous, Joule-heated vitrification capability, which is key for

- developing process flowsheets
- characterizing relationships between feed composition and the properties of the final glass produced
- establishing the fate and behavior of feed constituents under various melter processing conditions.

This melter system's capability to produce glass in a continuous manner is also essential for modeling the behavior of a full-scale system. Moreover, the size of the RSM allows the impacts of process variables upon melter performance or glass quality to be quickly and efficiently evaluated without undue expense or waste generation.

3.1 Melter

The RSM itself is a small joule-heated melter that is capable of processing melter feed on a continuous basis. The body of the RSM is an Inconel® closed-ended cylinder lined with Alfrax® refractory and containing a Monofrax® K3 refractory melt cavity. An Inconel overflow tube discharges molten glass into a stainless steel canister. An electric kiln surrounds the melter body and minimizes heat loss from the melter body during operation, and auxiliary heaters are used to heat the melter's discharge section to facilitate pouring of the glass. The stainless steel glass receipt canister sits inside a clamshell furnace maintained between 700°C and 900°C to promote uniform canister filling. A platform scale forming part of a hydraulic jack assembly that supports the canister inside the furnace allows glass-canister accumulations to be monitored as necessary. Two top-entering Inconel 690 electrodes (7.6-cm square × 0.64-cm thick [3-in. square × ¼-in. thick]) that are suspended in the glass supply joule-heating power to the RSM. The electrode's connecting tubular busbars also serve as thermowells that allow continuous measurement of the glass-pool temperatures. Figure 3.6 provides a cross-sectional view of the melter vessel illustrating its refractory makeup while Table 3.1 summarizes the RSM's dimensions and other operational features.

Figure 3.1. Photograph of the Research-Scale Melter Demonstration Unit

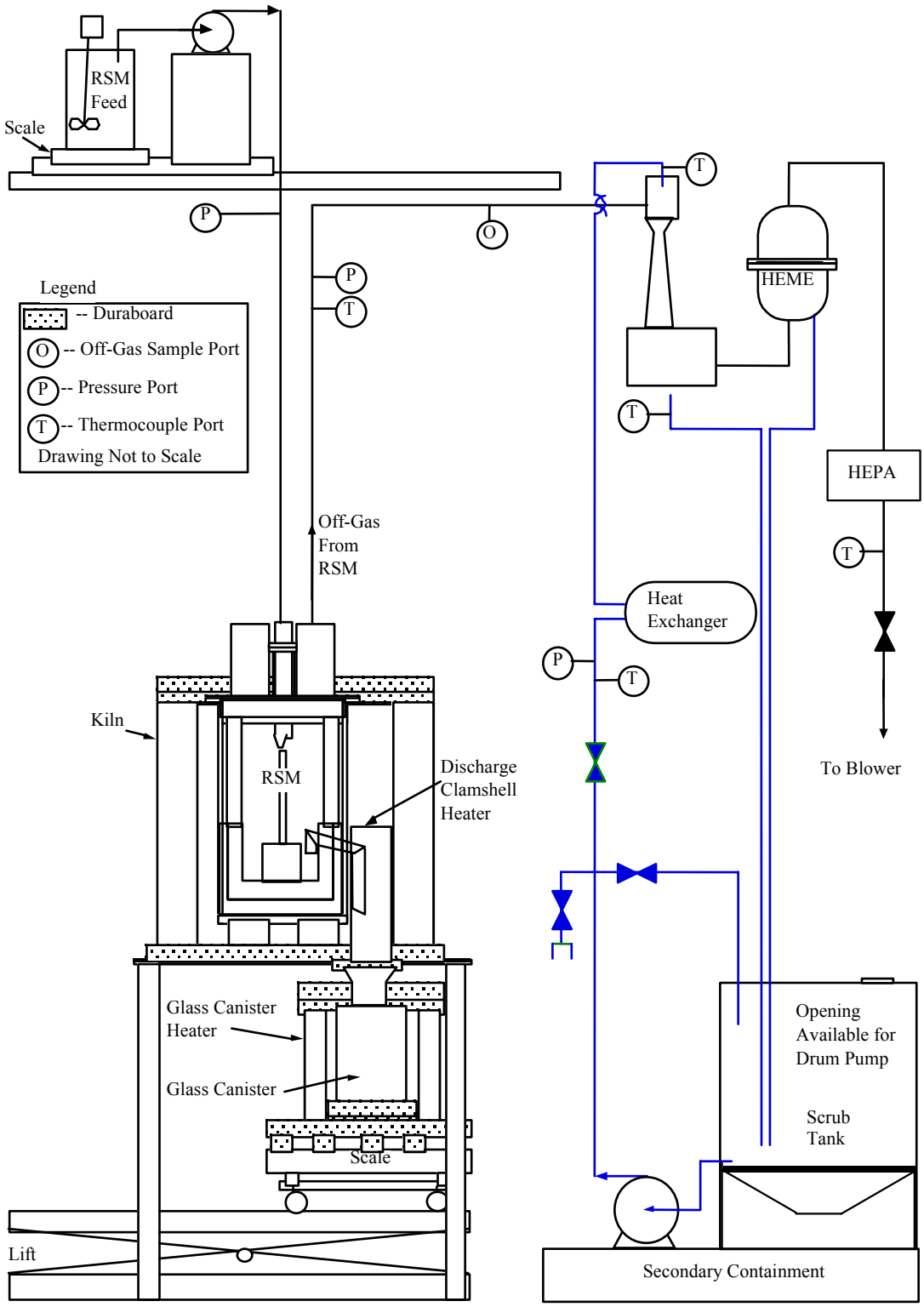

Figure 3.2. Schematic of the Research-Scale Melter Processing System

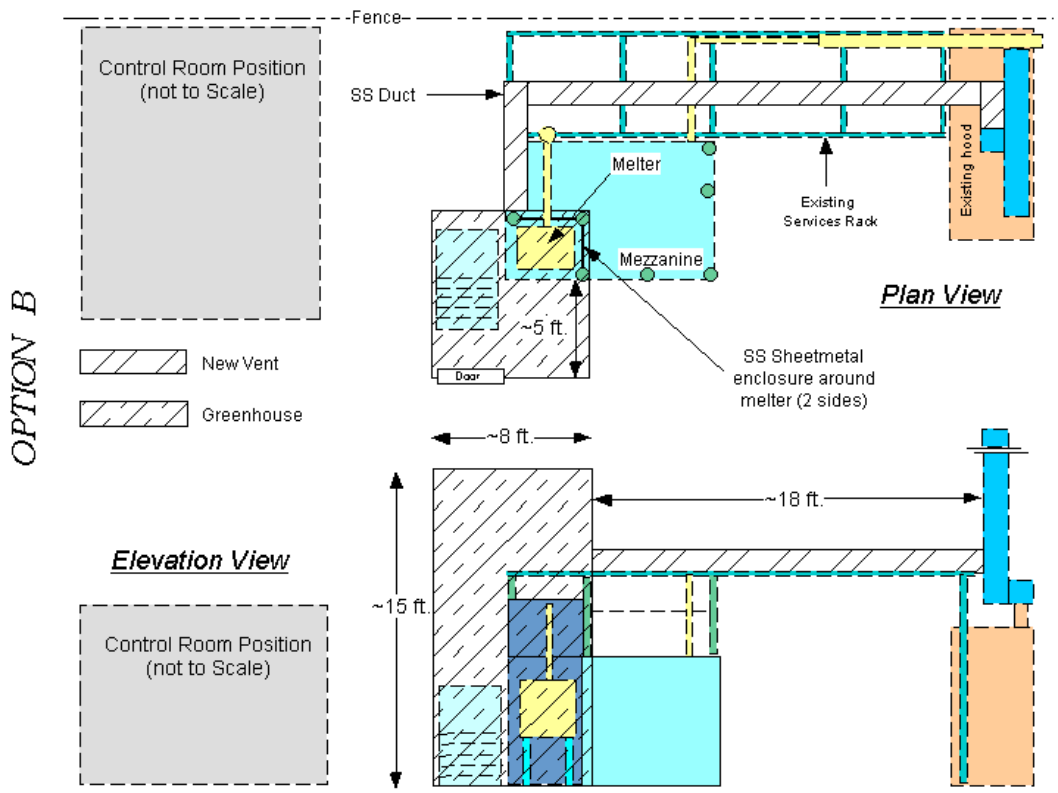

Figure 3.3. Plan and Elevation View of Temporary Melter Hood Enclosure

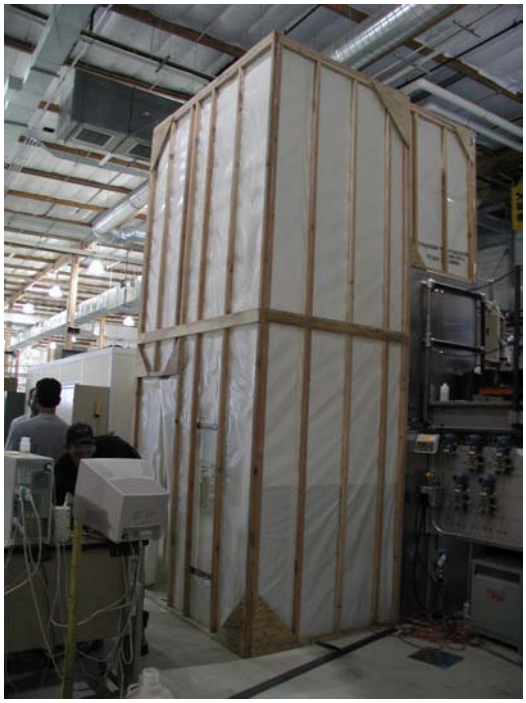

Figure 3.4. Exterior View of Melter Enclosure

Figure 3.5. Interior View of Enclosed Melter

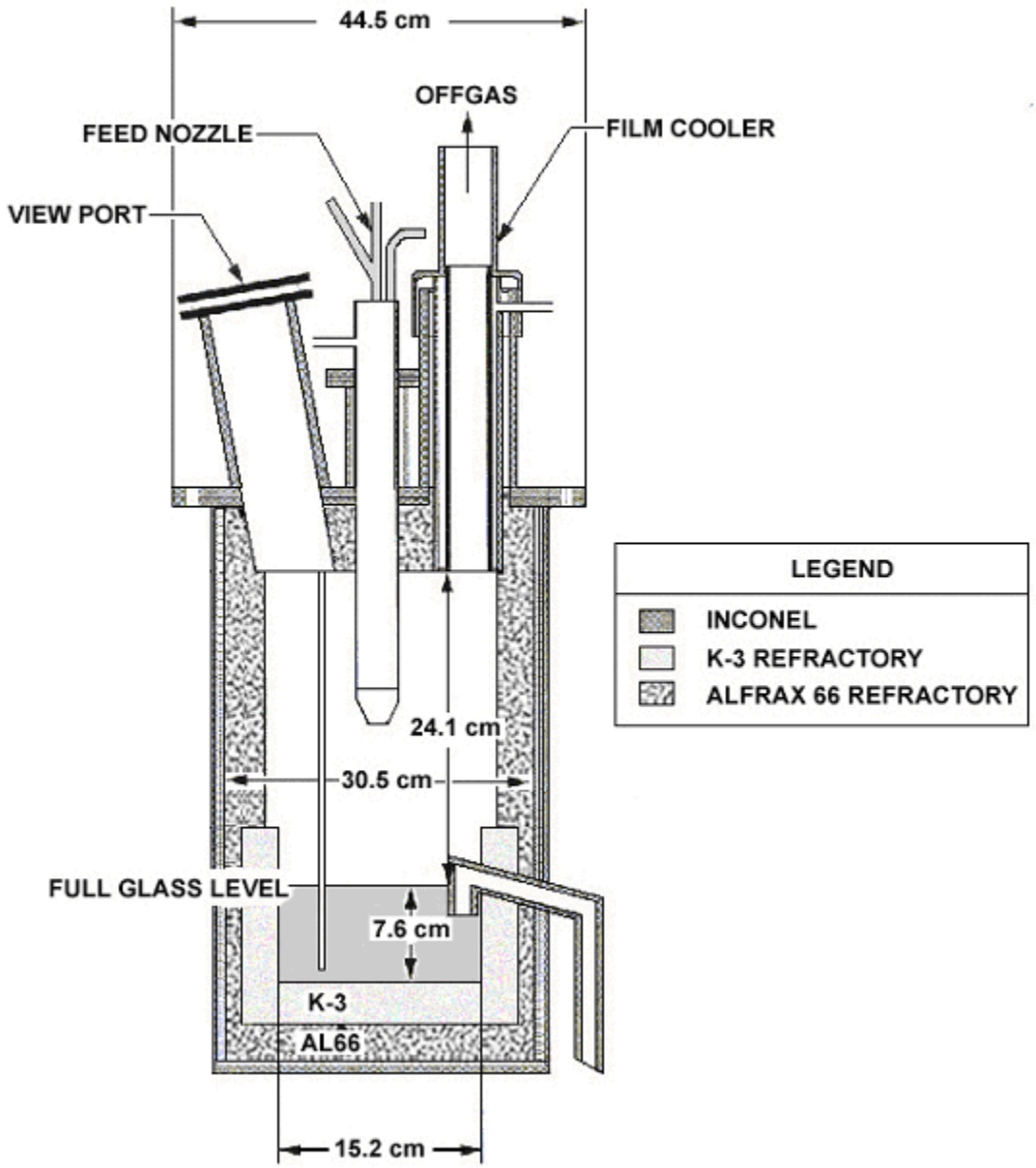

Figure 3.6. Cross Section View of the Research-Scale Melter (not drawn to scale)

3.2 Feed System

The melter-feed system is located on the elevated steel platform adjacent to the melter (see Figure 3.1.). The main feed tank used during current testing was a conical bottom tank with a maximum capacity of 57 L (15 gal). However, to add experimental flexibility, reductant was batched with the SBW-22 feed formulation in a 20-L day tank that served as the melter's feed source. The melter feed tanks, the variable-speed agitator, the peristaltic feed pump, and the valve-control station are attached to a steel pallet that allows the melter-feed-tank system, which includes a secondary containment vessel and a load-cell platform scale, to be lifted from the platform with a forklift. The electronic record of time-

dependent feed-tank weight, generated by the system's load-cell platform scale, provides for a direct measure of the melter mass-feeding rate. Figure 3.7 provides a photographic perspective of this elevated tank and feed-delivery system.

Table 3.1. RSM Dimensions and Operational Specifications

Parameter	**Value**
Melter cavity diameter	15 cm
Melter cavity height	17 cm
Melter inside volume	4.5 L
Glass pool surface area	182 cm^2
Nominal glass depth	7.6 cm
Melter glass inventory volume	1.4 L
Nominal molten glass mass	3.6 kg
Glass turnover rate @ nominal feed rate of 1.5 L/h of feed with 0.6 kg/L oxides	4.5 h
Maximum operating temperature	1,200°C
Nominal operating temperature	1,150°C
Electrode Dimensions	7.6 cm × 7.6 cm
Electrode Material	Inconel 690
Electrode melt-cavity bottom clearance	0 cm
Electrode current (average)	90 A
Electrode voltage (average)	25 V
Electrode current density (average/maximum)	1.6/2.0 A/cm^2

A peristaltic pump, located in a stainless steel enclosure designed to contain any electrometric pump-line slurry leaks, was used to extract feed from the 20-L agitated day tank. The feed was pumped through a valve-control station that allowed feed to be either sampled or delivered to the water-cooled feed nozzle that extended through the melter lid into the melter's plenum. The valve station, illustrated in Figures 3.8 and 3.9, also permits feed lines to be flushed with water without resorting to disassembly. A computer/pump interface allowed the pump's feeding rate to be controlled remotely, thus facilitating necessary adjustments required to maintain a steady melting process.

(a)

(b)

Figure 3.7. Elevated Feed Tank, Secondary Containment, and Load Cell Platform Scale

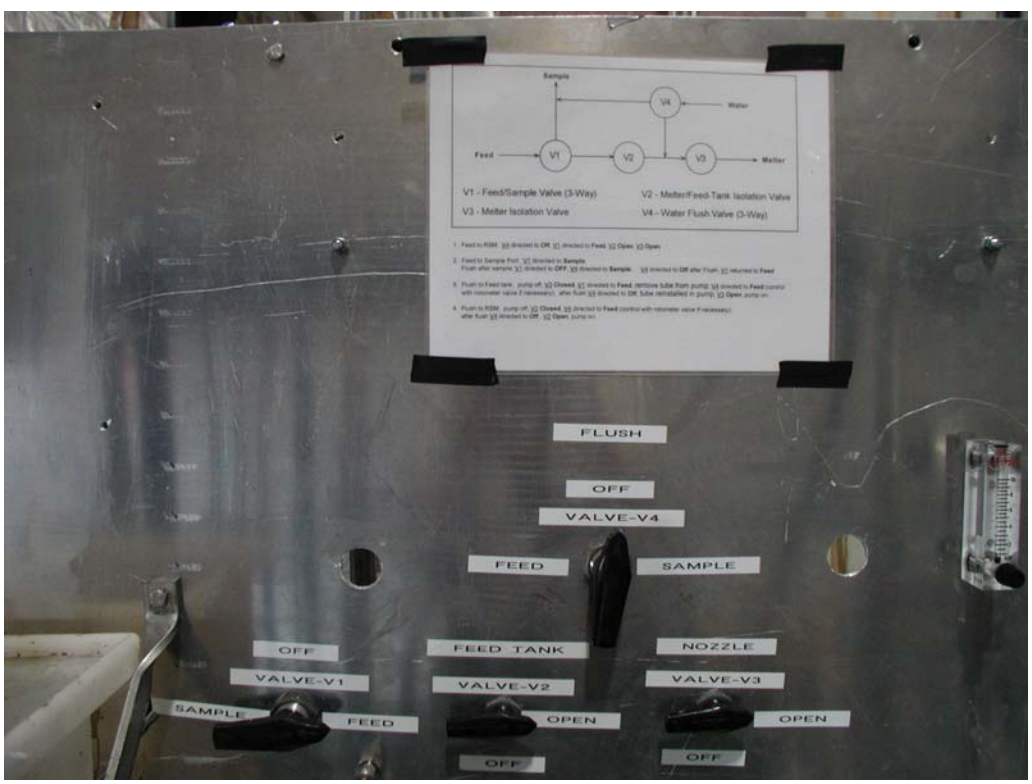

Figure 3.8. Melter Feed Delivery Valve Station

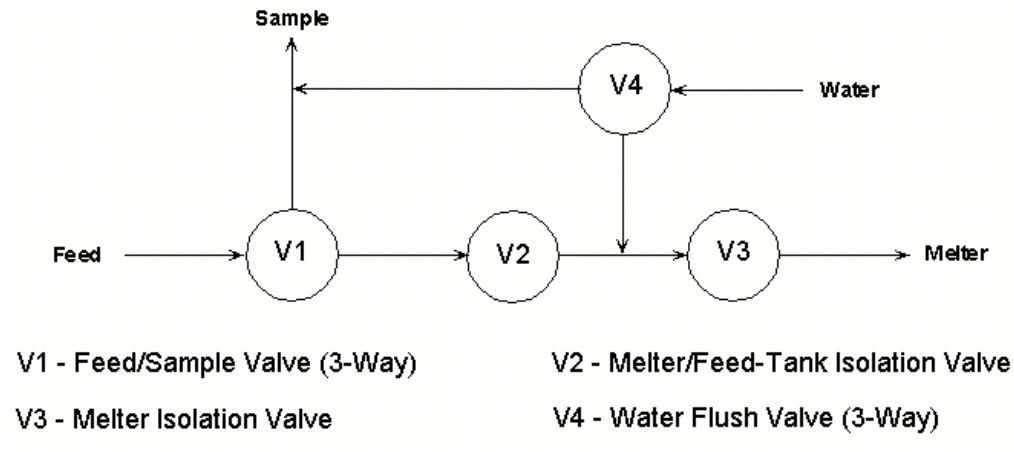

Figure 3.9. Melter Valve Station Configuration and Flow Logic

3.3 Off-Gas Processing System

Melter off-gas is treated by an off-gas treatment system consisting of a film cooler, ejector venturi scrubber (EVS), high-efficiency mist eliminator (HEME), and high-efficiency particulate arrestor (HEPA) filter (see Figure 3.2). The film cooler, located at the melter's exhaust port, injects room-temperature building air into the off-gas pipe to

- cool and solidify entrained vitreous matter to minimize pipe-wall particle adhesion
- speed aerosol transport to the EVS quench scrubber to minimize aerosol-settling losses in horizontal off-gas line (2 in.) pipe runs.

The EVS used an acidic, high-pressure aqueous scrubbing liquor spray to contact the process exhaust stream to quench it and to remove steam, large-diameter aerosols, and some condensable and/or acid gases. A 60-L (16-gal) charge of 1 M HNO_3 was put in the scrubbing liquor/condensate collection tank at the start of each test campaign. Off-gas condensate and all collected solids that accumulated within the condensate tank throughout the duration of the campaign were fully collected at the conclusion of each discrete test segment. A water-cooled heat exchanger located in the EVS's spray circuit was used to maintain nominal room-temperature scrubbing liquor conditions.

The HEME uses a deep, regenerable fibrous-bed to remove both liquid aerosols generated by the high-pressure EVS spray and submicron condensed-phase aerosols that successfully penetrate the low-efficiency quench (EVS) scrubber. The demisted and relatively clean HEME exhaust is then heated before being treated with a certified HEPA filter to remove all significant remaining concentrations of aerosol matter before the process exhaust is released to the environment.

3.4 Off-Gas Sampling System

Process off-gas sampling during the current test was limited to characterizing the melter source term and establishing the performance of the melter's aqueous quench scrubber and HEME as a function of processing conditions, i.e., reductant type and concentration. Melter effluents with significant room-temperature vapor pressures were nominally monitored continuously with gas analyzers, while discrete

sampling campaigns were conducted to characterize condensed-phase effluents and condensable/srubbable acid gases.

The continuous emission monitors employed during RSM testing along with the gases they were designed to detect are summarized in Table 3.2. The sample stream presented to each of these analyzers was, with the exception of the mercury analyzer, always extracted downstream of the film cooler but upstream of the EVS. A heated quartz filter, close-coupled to the process off-gas (POG) extraction point, was used to remove condensed matter from the sample stream flow. Since the POG sample source was unquenched, the filtered sample stream was, with the exception of the mercury sampling leg, diluted (~ 5x) with argon gas to reduce the dew point of the gas below the ambient operating-temperature conditions of the gas analyzers. Although a dryer was used to condition the gas chromatograph's (GC's) inlet stream, the off-gas effluent concentrations derived by the GC have been corrected to represent actual conditions in the unquenched, post-film cooler process exhaust (wet basis).

Table 3.2. RSM's Continuous Emissions Monitors (CEMs)

Analyzer	**Targeted Effluent Gases**
Gas Chromatograph	H_2, He, N_2, CO, O_2, CO_2, & N_2O
Nitrogen Oxide	NO & NO_2
Sulfur Oxide Monitor	SO_2
Total Hydrocarbon	Volatile Hydrocarbons
Total Mercury Analyzer	Hg (oxidized and elemental)

As alluded to earlier, the inlet source of the Hg analyzer was variable and was changed, when appropriate, during the course of the testing. Specifically, three off-gas sampling sites were available to the mercury analyzer: a post melter site located between the film cooler and the EVS, a post EVS site located between the EVS and the HEME, and a post HEME location sited between the HEME and HEPA. A conceptual schematic of the sample gas-distribution system used to support the continuous emission monitoring conducted during RSM-2 is illustrated in Figure 3.10.

Discrete sampling for process-generated off-gas effluents was also conducted using manual sampling trains based on the modified EPA Method 5 sampling train illustrated in Figure 3.11. The actual system used, however, did not employ the standard pitot tube equipped stack probe. Rather, helium dilution process flow measurements (see below) were used in conjunction with process line and sampling tube geometrical factors to establish isokinetic sampling flow rates, where gas velocities of sample and process streams are matched. This off-gas sampling system was composed of an appropriately sized quartz sampling probe, an aerosol collection device, a condenser to remove condensable vapors, and a series arrangement of aqueous chemical gas scrubbers used to collect reactive non-condensable gases. Photographs of the sample-train collection and flow-control equipment used during RSM-2 are shown in Figures 3.12 and 3.13, respectively. Since all gas scrubbers were usually immersed in an ice-bath container, the first scrubber vessel also served as the system's condenser.

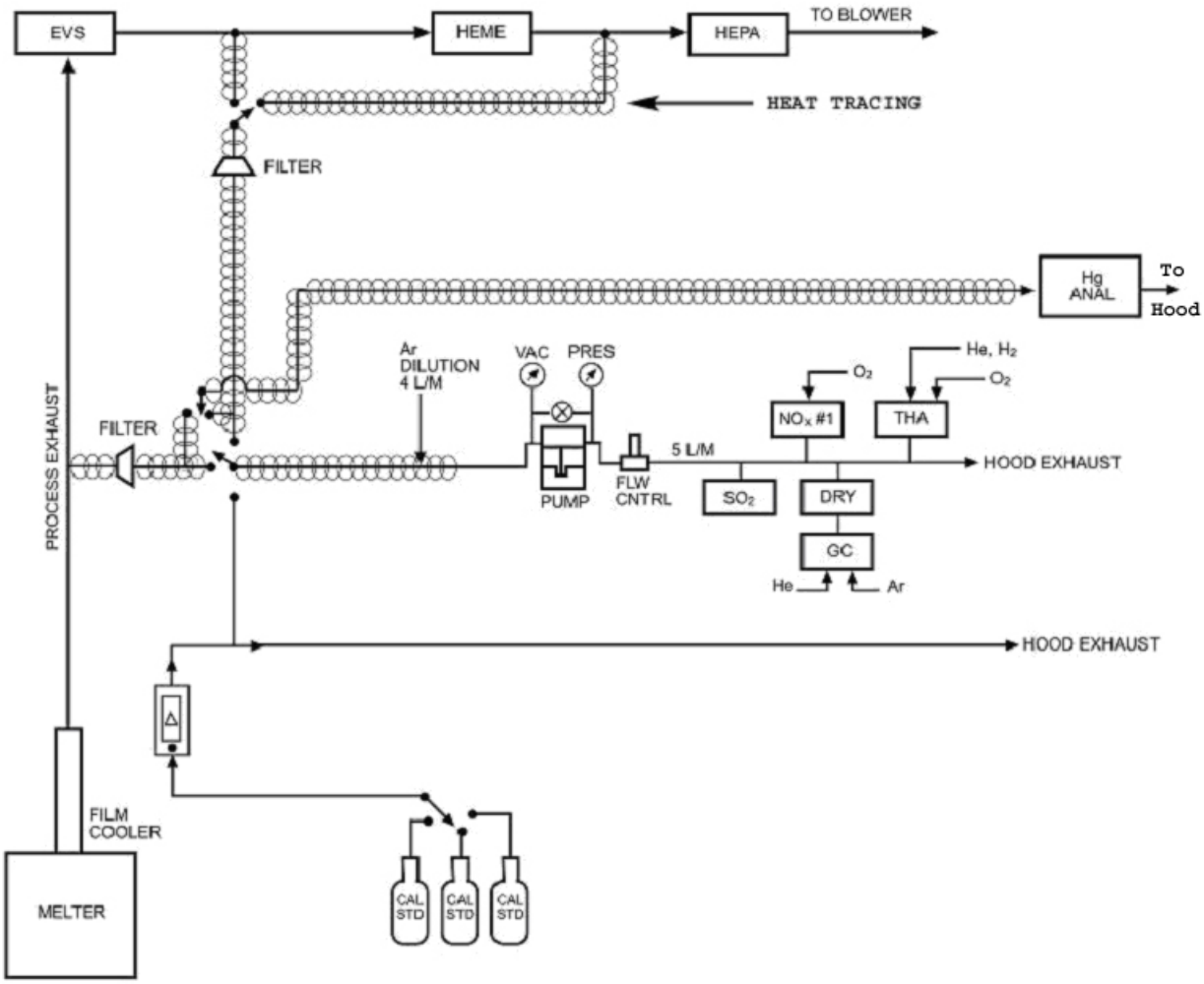

Figure 3.10. Gas Distribution System Supplying Continuous Emission Monitors

In operation, the sampling probe was inserted coaxially along the centerline of the off-gas pipe into the process off-gas line at an elbow between the film cooler and the EVS (see Figure 3.14). The diameter of the beveled sampling-probe inlet was chosen to allow isokinetic-sampling conditions to be achieved with reasonable sampling flow rates. The heated filter assembly employed a quartz-filter media to quantitatively collect condensed effluents entrained in the unquenched (150°C), post film-cooler melter exhaust stream. The filtered gas stream was subsequently quenched (to 20°C or lower, depending on the application) and chemically washed to remove reactive gases by a series arrangement of several gas washing vessels.

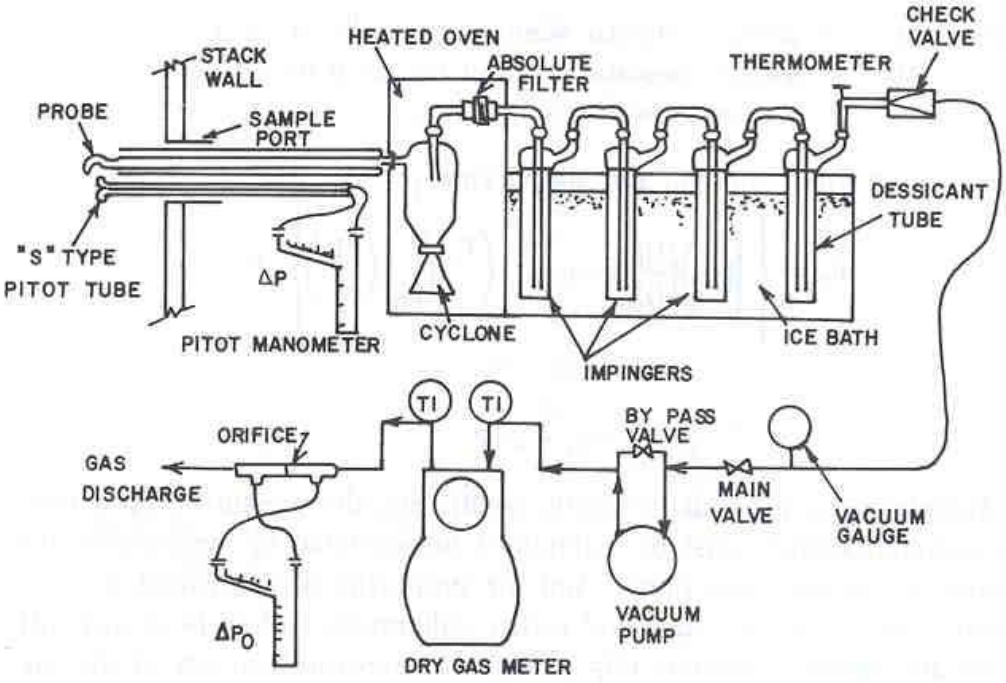

Figure 3.11. Modified EPA Method-5 Sampling Train

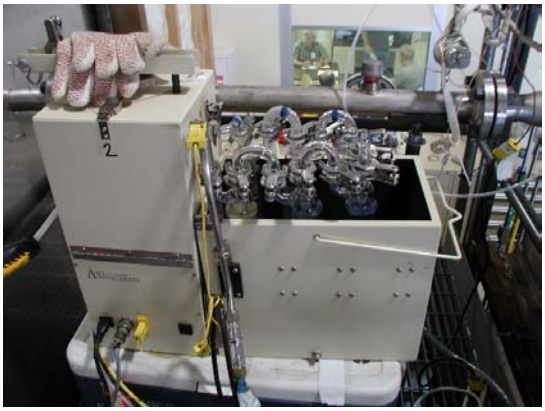

Figure 3.12. Sample Train Collection Module

Figure 3.13. Sample Train Flow Control Module

3.5 Data Acquisition and Process Control System

The RSM is controlled and monitored with a Square D, SY/MAX® 400 Programmable Logic Controller (PLC). Operators interface with the PLC using a PC running FIX DMACS® software on a Microsoft NT platform that is serially linked to the PLC. FIX32 provides user-control inputs as well as history logging of the RSM system-process variables.

This data-acquisition and control system monitors and controls the electrodes, the melter and discharge canister kilns, the heater for the discharge section, and the peristaltic pump for the feed system. Data collected include the voltage and current for major electrical components, temperature at various locations in the system (e.g., molten glass, plenum space in melter, melter kiln, off-gas treatment system), pressures in the melter and across all off-gas system components, and the weight of the feed tank. Data are typically archived every minute, but are displayed at more frequent intervals to assist the operators.

Figure 3.14. Melter Off-Gas Line Sampling Port

4.0 SBW Simulant, Melter Feed, and Product Glass

The primary objective of the Liquid Fed Ceramic Melter (LFCM) waste-vitrification process is to isolate the toxic and/or hazardous elements and/or radionuclides from the environment. The vitrification technology achieves this by incorporating and thereby immobilizing these hazardous waste constituents within a durable glass matrix. To create a vitreous waste product, glass-forming chemicals have to be added to the waste before it can be vitrified (calcined and melted) in a high-temperature melter. However, to meet stringent waste-form durability criteria, an appropriate glass composition has to be formulated, and its chemical properties (multi-valent oxidation states) have to be carefully controlled.

During RSM testing of the SBW flowsheet, melter-feed material of specified waste loading was prepared in batches by mixing a preformulated SBW stock solution/slurry with appropriate quantities of glass-forming chemicals to which reductant material of variable concentration was added as a glass-oxidation-state modifier. The following discussion provides detailed information concerning these feed-stream constituent additives.

4.1 SBW Surrogate

The SBW surrogate solutions prepared for RSM testing were, with few exceptions, physically and chemically representative of the characterized material contained in INTEC TFF tank WM-180. The make-up procedure was prepared based on information provided by INEEL's Dr. Jerry Christian (Christian 2001). The procedure used to prepare the SBW surrogate solutions for this RSM test is provided in Appendix B. Table 4.1 compares the defined target SBW composition with the surrogate material used during RSM testing. As is clear from these tabular data, the surrogate recipe does, however, exclude the chemically hazardous constituents, As and Be, and all unstable radioactive components. Some of these radionuclides were, nevertheless, represented by their stable isotopes or chemical analogs, but not necessarily at SBW reference concentrations. Table 4.2 summarizes the major radionuclides present in SBW and identifies the stable isotope/surrogate substitutes used to represent them.

Following the waste makeup procedure presented in Appendix B, 160 L (42 gal) of SBW surrogate was prepared in two separate batches, and the resultant batch composition data are compared to the defined SBW target in Table 4.3. The analytical agreement with few exceptions is quite good, and the lack of adequate analytical detection sensitivity can easily explain the few larger variances that are observed. Based on these analytical data and a review of the SBW preparation documentation, it was determined that the surrogate material prepared in support of the RSM's SBW flowsheet adequately represented the INTEC TFF waste source, and no chemical adjustments were deemed necessary.

Beyond chemical composition, the physical properties of the SBW surrogate were also measured. Specifically, density and weight loss on ignition (LOI) and equivalent oxides/L measurements were

Table 4.1. INTEC TFF Tank WM-180 Waste and Simulant Compositions

INEEL Waste Definition[a]		Simulant Con (M)
Element	**Reagent**	
Aluminum	$Al(NO_3)_3 \cdot 9H_2O$	6.3E-01
Arsenic*	$Na_2HAsO_4 \cdot 7H_2O$	2.4E-4
Barium	$Ba(NO_3)_2$	5.6E-05
Beryllium*	BeF_2	7.3E-6
Boron	H_3BO_3	1.2E-2
Cadmium	$Cd(NO_3)_2 \cdot 4H_2O$	7.5E-4
Calcium	$Ca(NO_3)_2 \cdot 4H_2O$	4.7E-2
Cerium	$Ce(NO_3)_3 \cdot 6H_2O$	3.4E-4
Cesium	$CsNO_3$ (Rad Surrogate)	2.2E-3
Chromium	$Cr(NO_3)_3 \cdot 9H_2O$	2.7E-3
Cobalt	$Co(NO_3)_2 \cdot 6H_2O$	1.9E-5
Copper	$Cu(NO_3)_2 \cdot 3H_2O$	7.0E-4
Gadolinium	$Gd(NO_3)_3 \cdot 5H_2O$	1.8E-4
Iron	$Fe(NO_3)_3 \cdot 9H_2O$	2.2E-2
Lead	$Pb(NO_3)_2$	1.3E-3
Lithium	$LiNO_3$	3.4E-4
Magnesium	$Mg(NO_3)_2 \cdot 6H_2O$	1.2E-2
Manganese	$Mn(NO_3)_2$	1.4E-2
Mercury	$Hg(NO_3)_2 \cdot 2H_2O$	2.0E-3
Molybdenum	H_2MoO_4	1.9E-4
Nickel	$Ni(NO_3)_2 \cdot 6H_2O$	1.5E-3
Potassium	KNO_3	2.0E-1
Ruthenium	$RuNO(NO_3)_3$	1.2E-4
Sodium	$NaNO_3$	2.1E+0
Strontium	$Sr(NO_3)_2$	6.8E-3
Titanium	TiO_2	5.8E-5
Uranium*	$UO_2(NO_3)_2 \cdot 6H_2O$	3.2E-4
Zinc	$Zn(NO_3)_2 \cdot 6H_2O$	1.0E-3
Zirconium	ZrF_4	6.6E-5
Chloride	HCl	2.9E-2
Fluoride	HF	3.4E-2
Iodide	KI	1.3E-4
Nitrate	HNO_3	8.0E-1
Phosphate	H_3PO_4	1.4E-2
Sulfate	H_2SO_4	7.0E-2

(a) Unstable elements of insignificant mass not included.
* Not included in surrogate.

Table 4.2. Stable Isotope Chemical Analog Surrogates

SBW Radionuclide	Substitute/Chemical Surrogate
^{3}H	Stable Isotope
^{60}Co	Stable Isotope
^{90}Sr	Stable Isotope
^{99}Tc	None
^{129}I	Stable Isotope
^{134}Cs	Stable Isotope
^{137}Cs	Stable Isotope
^{154}Eu	Cerium
^{234}U	None
^{235}U	None
^{236}U	None
^{238}U	None
^{237}Np	None
^{238}Pu	None
^{239}Pu	None
^{241}Am	None

conducted on the SBW surrogate and resulting melter feed. These values along with the defined SBW oxide loading value are summarized in Table 4.4. The measured oxide loading data, if sampling errors are neglected, suggest a slightly diluted SBW surrogate, but only by ~ 10%.

4.2 Target Glass Composition

The targeted vitreous product composition for the current RSM-2 flowsheet evaluation was established from a series of laboratory crucible tests involving SBW surrogate material and suitable glass-forming additives that were chosen to maximize the glass waste loading. Since these crucible tests suggested that the glass solubility of waste constituent sulfur would limit maximum achievable waste loadings, a highly durable glass with the highest possible sulfur capacity was formulated for melter testing. The target composition of this glass at 20% waste loading, designated SBW-22, is summarized in Table 4.5, where only major constituents are identified.

4.3 Glass Former and Chemical Additives

To produce the target glass composition for a specified waste-loading value (20% for RSM-2), calculated quantities of glass-forming chemicals were blended with a prespecified batch volume of SBW surrogate solution. The actual glass-forming chemicals employed during this feed-batching operation are summarized in Table 4.6 along with their corresponding oxide-equivalent form. The proportions of each of these equivalent oxide forms, which have been normalized to 100%, define a frit composition that

could be directly blended, in appropriate proportions, with the SBW to create the same target glass. However, individual chemical additives were used during all RSM batching operations. The batching sheets used to prepare the melter feeds appear in Appendix C along with the other test-data logging sheets.

Table 4.3. SBW and PNNL Surrogate Compositions

	RSM-01-2 SBW Composition				
	Target	**Batch #1**		**Batch #2**	
Analyte	**mg/L**	**mg/L**	**%Dif**	**mg/L**	**%Dif**
Aluminum	17,900.00	14,900.00	-17.0	16,400.00	-8.4
Barium	7.66	10.00	30.6	7.90	3.2
Boron	133.00	147.00	10.6	120.00	-9.7
Cadmium	84.80	96.00	13.2	79.00	-6.8
Calcium	1,890.00	1,850.00	-2.2	1,650.00	-13.0
Cerium	47.10	55.00	16.8	50.00	6.2
Cesium	295.00	280.00	-5.1	300.00	1.7
Chromium	174.00	193.00	10.7	186.00	6.7
Cobalt	1.14	ND	ND	ND	ND
Copper	44.30	55.00	24.2	51.00	15.1
Iron	1,210.00	1,200.00	-1.1	1,280.00	5.5
Lead	271.00	383.00	41.4	257.00	-5.1
Lithium	2.36	3.00	27.4	2.70	14.6
Magnesium	292.00	318.00	8.8	310.00	6.0
Manganese	775.00	365.00	-53.0	810.00	4.6
Mercury	404.00	464.00	14.8	455.00	12.6
Molybdenum	18.50	15.00	-19.0	16.00	-13.0
Nickel	86.30	96.00	11.2	96.00	11.2
Phosphorous	424.00	438.00	3.2	400.00	-5.7
Potassium	7,670.00	9,020.00	17.6	8,380.00	9.3
Ruthenium	12.60	25.00	98.4	17.00	34.9
Sodium	47,300.00	47,000.00	-0.7	44,700.00	-5.5
Strontium	592.00	620.00	4.7	569.00	-3.9
Sulfur	2,240.00	2,200.00	-1.7	2,330.00	4.1
Titanium	2.77	1.30	-53.0	0.80	-71.0
Zinc	68.60	76.00	10.8	71.00	3.5
Zirconium	6.04	4.00	-34.0	ND	ND
Chloride	1,040.00	880.00	-15.0	840.00	-19.0
Fluoride	900.00	ND	ND	ND	ND
Iodide	16.50	ND	ND	ND	ND
Nitrate	327,000.00	319,000.00	-2.4	330,000.00	1.0

Table 4.4. SBW Surrogate Physical Characteristics

Batch	Sp. Grav.	LOI (%)	Oxides (g/L)
SBW -1	1.22	91.5	104
SBW-2	1.24	90.8	114
Avg	1.23	91.1	109
		SBW Target = 123	

Table 4.5. SBW-22 Target Glass Composition at 20% Waste Loading

Oxide	Wt%
SiO_2	54.40
Na_2O	13.70
Al_2O_3	5.45
B_2O_3	4.89
Li_2O	4.89
CaO	4.44
V_2O_5	3.90
ZrO_2	1.95
Fe_2O_3	1.50
Balance	4.88

Table 4.6. Melter Feed Glass-Former Additives and Equivalent Frit Composition

Frit	Oxide		Glass Former		Mass Ratio
Oxide	Wt%	MW	Chemical	MW	(g-Chem/g-frit)
B_2O_3	6.03	69.6	H_3BO_3	61.8	0.107
Fe_2O_3	1.52	160	Fe_2O_3	160	0.015
Li_2O	6.11	29.9	$LiOH*H_2O$	42.0	0.171
SiO_2	68.0	60.1	SiO_2	60.1	0.68
CaO	5.02	56.1	$Ca(OH)_2$	74.1	0.066
MgO	1.75	40.3	$Mg(OH)_2$	58.3	0.025
Na_2O	4.29	62.0	NaOH	40.0	0.055
V_2O_5	4.88	182	V_2O_5	182	0.048
ZrO_2	2.44	123	ZrO_2	123	0.024

4.3.1 Glass-Former Rheological Effects

The glass-forming additives identified in Table 4.6 and used during RSM-2 are all chemical bases that can and do affect the pH of aqueous media in which they are suspended. At 20% SBW waste

loading, the required concentration of basic oxides was more than sufficient to increase the pH of the resultant melter feed slurry above 6. This caused unacceptable thickening of the melter slurry feed, making it rheologically unsuitable for tank storage and melter-feed delivery systems. To overcome the basicity of the glass formers, laboratory tests indicated that 60 cc of concentrated HNO_3 acid had to be added for each liter of SBW used in a feed batch. However, when large-scale batching operations were conducted, it was found that an additional 40 cc or a total of 100 cc of nitric had to be used for each liter of SBW used in the batching process. To compensate for the added nitrate oxidant, feed reductant concentrations had to be significantly increased as discussed below.

4.3.2 Reductant Additives

As mentioned earlier, the organic reductants, sucrose or glycolic acid, were added to feed batches to control the oxidation states of multivalent elements in the product glass. The final selection of a reducing agent for SBW vitrification will be based on the combined results from multiples tests at different scales, including laboratory-scale crucible testing, melt-rate furnace testing, research-scale melter testing, and pilot-scale melter testing. The major criteria for selecting a reducing agent for SBW vitrification are that the reductant

1. will not detrimentally affect the ultimate chemical composition or physical properties of the glass product
2. is adequately reactive, providing the capability to chemically control glass oxidation state without producing reductive inclusion in the glass
3. is soluble or easily mixed in the SBW solution
4. can or has been demonstrated to be safe in melter applications
5. does not adversely affect melt rate or waste throughput or the rheology of the melter feed
6. is cost effective.

For the initial selection of reductant candidates, only organic compounds and inorganic carbon were considered. Organic reductants have an extensive record of usage in nuclear processes. Additionally, organic reductants are generally oxidized completely in the melter and exit with the off-gas without affecting the final composition of the glass (Criterion #1). Among the hundreds of potential organic reductants, the selection was limited to those that are relatively non-volatile, so that some reductant would remain following denitration in the cold cap (Criterion #2).

Sugar was an obvious candidate because of its historical use (Jain and Pan 2000) as a reductant for vitrification and other treatment processes (Criteria #1 through #6). Because of the extensive record of successful uses of sugar as a radioactive waste reductant, it was established at the onset of this development effort as the baseline reductant for SBW vitrification. Other organic reductants included for comparative selection testing are cornstarch, glycolic acid, and activated carbon. Cornstarch was selected to explore the potential differences resulting from using an alternative carbohydrate. Although glycolic acid is 25% less efficient in reducing capability (by calculation), and considerably more expensive than sugar, it was chosen because it had been identified as less likely to form potentially dangerous, unstable

carbon-nitrate compounds. Activated carbon was chosen because it had been used as an effective reducing agent in a past SBW vitrification test performed in the pilot-scale EV-16 melter at Clemson (Perry et al. 2001). Additionally, activated carbon, being less reactive than sugar or other carbohydrates, was considered to have a better potential to reduce sulfate to gaseous SO_2. Therefore, activated carbon can be an asset if molten salt accumulation is problematic but a liability if volatilized sulfur affects secondary waste stream composition. Although activated carbon is not soluble in SBW, it was found to mix adequately with the slurry feed, provided it is first ground to a powder, and sufficient agitation is used (Criterion #3).

Reductant comparison testing for the RSM-2 test run, however, was limited to the use of sugar, glycolic acid, or no reductant at all. The test plan called for operating under both oxidizing and reducing melter conditions using both reductants. Activated carbon was not included in this testing due to its demonstrated low reactivity with nitrate, its insolubility (Criterion 3), and its chemical persistence as inclusions in the molten glass melt. Cornstarch was not included in this testing because previous laboratory testing of melter feeds revealed that this reductant could cause significant feed gelling problems.

4.3.3 Reductant Loading

Although reductant is also useful for denitrating the feed during melter processing, the maximum reductant concentration that can be effectively used is limited by the oxidation state of the product glass. Excess reductant will tend to reduce not only nitrates, but also glass oxides. The glass oxidation state is usually characterized by the fraction of iron in its +II valence state, which by glass-quality criteria, is constrained to be maintained at or under 0.3. Based on initial crucible testing, the recommended baseline sucrose concentration range for achieving minimal measurable and maximum tolerable reduced conditions were estimated to be 135 g/L-SBW and 170 g/L-SBW, respectively.

However, to overcome the basicity of the glass formers that has been previously discussed, 100 cc of concentrated HNO_3 acid had to be added for each liter of SBW used in feed batching. Initially, when it appeared that only 60 cc of concentrated HNO_3 were needed, laboratory tests suggested that the bounding sugar concentrations be increased by 45 g to compensate for the added oxidant. When large-scale batching operations indicated a need for an additional 40 cc of concentrated nitric (100 cc total) for each liter of SBW used, the nitrate ratio was used to scale the corresponding min/max sugar range to 200 g/L-SBW and 235 g/L-SBW, respectively. While this baseline sugar concentration was used during initial phases of testing, reductant loadings were parametrically varied throughout the test to assess the impact of reductant type (sucrose/glycolic acid) and concentration upon processing rates, nitrogen oxide emissions, and glass-oxidation state. Concentration ranges for glycolic acid were derived from corresponding sugar values using the relative reducing power of these reductants.

4.4 Melter Feed Characteristics

Melter-feed samples were collected from each feed batch prepared during the RSM-2 test. These feed samples were subsequently analyzed to determine their physical properties and chemical

composition. Table 4.7 presents the physical properties associated with all the feeds of differing reductant composition. Also presented in this table, for comparison purposes, are the corresponding total-oxide target values derived from SBW-surrogate and target-glass composition data previously discussed. Feed total-oxide values derived from laboratory loss-on-drying (LOD, @ 110 °C) and loss-on-ignition (LOI of dried SBW @ 1150°C) tests are found to satisfactorily meet target value expectations well within a 10% uncertainty, with an average that is nominally equivalent to the target value itself.

Table 4.7. Physical Properties of Surrogate SBW Melter Feeds

		Weight Loss %		g-Oxide/L-SBW	
Sample # RSM-2	**Sp. Grav.**	**LOD**	**LOI**	**Found**	**Target**
005	1.44	53.5	37.8	584	615
017	> 1.36	52.7	35.7	582	615
048	1.42	54.2	30.6	636	615
072	1.47	44.4	42.7	656	615
LOD = loss on drying; LOI = loss on ignition					

The oxide-equivalent compositions of each of the feed batches prepared during RSM testing are summarized in Table 4.8 and compared to target values based upon the previously defined waste and glass-former composition values. All of the major feed-component concentrations were reasonably consistent throughout the nominal 5 days of testing, although -048 and -045 appear somewhat atypical for unknown reasons. With the exception of vanadium, there were no trends or large variations of importance, and most major components were close to their target values. However, due to detection-sensitivity limitations, the weight fractions of several trace constituents could not be reported, and those that could appear to be overestimated. Since the targeted concentrations of these trace constituents are so low, the bulk chemicals used cannot be overlooked as unintended trace-element contributors; consequently, these greater-than-expected as-found values will be adopted as baseline data.

The variability in the vanadium data is a little mystifying in that it is not a trace constituent. Since detection sensitivity is not an issue for this element and other major constituents are not similarly affected, species-specific analytical processing difficulties and/or inhomogeneity in the feed tank or in the analytical sample must be responsible for this observed variability. As will be discussed in a later section, much less dispersion in the vanadium glass composition is observed, suggesting that settling and resultant loss in the feed tank is not an issue.

Apart from the trace constituents and the vanadium variability just discussed, the overall melter-feed composition data agreed quite well with feed-formulation expectation values. In general, it appears there was good control over feed composition throughout the entire test period, which will be corroborated when the glass data are subsequently discussed.

Table 4.8. Melter Feed Composition for Batch Samples RSM-01-2-XYZ

	Feed Composition as Oxides (Wt%)									
Oxide	**-005**	**-017**	**-038**	**-048**	**-059**	**-072**	**-084**	**Ave**	**Target**	**%Dev**
Al_2O_3	4.240	4.540	4.780	3.320	3.430	4.160	5.100	4.220	5.450	-22.0
B_2O_3	4.730	4.590	4.910	3.790	4.150	5.030	4.680	4.560	4.890	-6.9
BaO	ND	ND	ND	ND	ND	ND	ND	ND	0.001	ND
CaO	3.650	3.830	3.890	2.850	3.100	3.700	3.620	3.520	4.440	-21.0
CdO	ND	ND	ND	ND	ND	ND	ND	ND	0.016	ND
Cr_2O_3	ND	0.055	ND	ND	ND	ND	0.058	0.057	0.041	37.6
Cs_2O	0.043	0.051	0.050	0.035	0.039	0.045	0.046	0.044	0.050	-12.0
CuO	ND	ND	ND	ND	ND	ND	ND	ND	0.009	ND
Fe_2O_3	1.570	1.630	1.680	1.480	1.500	1.640	1.600	1.590	1.500	6.1
K_2O	1.690	1.810	1.770	1.320	1.390	1.600	1.580	1.590	1.490	7.0
Li_2O	5.280	5.280	5.380	3.900	4.160	4.950	5.030	4.850	4.890	-0.7
MgO	1.520	1.580	1.650	1.160	1.300	1.600	1.600	1.490	1.480	0.6
MnO	0.072	0.081	0.082	0.057	0.061	0.074	0.152	0.083	0.161	-49.0
MoO_3	ND	ND	ND	ND	ND	ND	ND	ND	0.004	ND
Na_2O	12.100	12.800	11.200	11.000	10.400	12.700	13.100	11.900	13.700	-13.0
NiO	0.091	0.089	0.111	0.095	0.089	0.088	0.092	0.093	0.018	427.0
P_2O_5	ND	ND	ND	ND	ND	ND	ND	ND	0.157	ND
PbO	ND	ND	ND	ND	ND	ND	ND	ND	0.047	ND
RuO_2	ND	ND	ND	ND	ND	ND	ND	ND	0.003	ND
SO_3	0.882	0.965	0.986	0.678	0.816	0.941	0.884	0.879	0.900	-2.3
SiO_2	57.600	56.300	59.100	54.900	57.900	58.400	56.400	57.200	54.400	5.3
SrO	0.093	0.107	0.111	0.073	0.080	0.097	0.096	0.094	0.113	-17.0
V_2O_5	4.910	5.030	3.190	11.200	8.710	3.750	4.850	5.950	3.900	52.5
ZnO	0.014	0.016	0.014	0.010	0.010	0.013	0.016	0.013	0.014	-3.9
ZrO_2	1.470	1.320	1.140	4.090	2.880	1.220	1.150	1.890	1.950	-3.0

4.5 Product Glass Characteristics

Glass grab samples were collected from RSM pours, which nominally occurred every 2 h. Fe^{+2}:Fe_{tot} ratios were measured throughout the entire melter-testing period to quickly assess the impact of changing reductant levels upon the chemical state of the glass product. In addition, representative glass samples produced from each of the major RSM-2 processing campaigns were compositionally analyzed and subjected to PCT and TCLP testing. The results of these measurements, as well as processing observations, will now be discussed.

4.5.1 Oxidation-State Evaluations

As previously discussed (see Section 4.3), the SBW reductant and reductant concentration were experimental parameters during RSM testing. The purpose of varying the reductant concentration was to establish optimum conditions that would maximize nitrate destruction while maintaining acceptable glass-

oxidation-state conditions and melter processing conditions for each reductant type tested. To accomplish this, oxidation states were promptly measured for glass samples taken before and after melter-feed reductant levels were altered.

A chemical method for measuring an Fe^{+2} complex colorimetrically at a wavelength of 515 ηm was used to establish the vitreous iron fraction in the +II valance state. This is accomplished by dissolving a powdered glass sample in H_2SO_4 and HF, buffering the resultant solution with sodium acetate/boric acid solution containing an o-phenanthroline complexing agent, and conducting an Fe^{+2}-specific absorbance measurement at 515 ηm. Ascorbic acid is subsequently used to reduce all remaining iron in the dissolved sample to the Fe^{+2} state, which allows the total Fe to be measured by a subsequent absorption measurement. These results normally allow the fraction of iron in the +II valence state in the glass sample to be directly determined. A detailed procedure similar to that used for making these oxidation-state measurements is reproduced in Appendix D.

4.5.1.1 Contemporaneous Glass-Oxidation Results

A significant amount of important process-development information resulted from the RSM-2 test run. Reductant-concentration estimates during RSM-2 were determined by applying laboratory-generated redox curves to the increases in nitric acid content of the melter feed previously described. The uncertainties associated with these semi-empirical reductant loading estimates resulted in changing the order of planned test iterations detailed in the RSM-2 Test Plan (see Appendix A), as is shown in Table 4.9 and graphically illustrated in Figure 4.1. The initial 200 g/L-SBW sugar concentration was intended to be sufficient to reduce all feed nitrate while still producing an oxidized glass. Instead, however, the resulting glass was quite reduced. The laboratory redox curves, generated using the SBW-9 glass formulation in preparation for the RSM-1 test (Goles 2001) are seemingly not accurately applicable for determining the required reductant concentrations when using a different glass formulation (SBW-22). The required addition of concentrated nitric acid likely played some role in the unexpected results, as the required sugar concentration was changed proportionally with nitrate concentration.

The results of comparing RSM-2 processing-campaign test results using different reductants are also somewhat surprising. Although iron in the glass product was generally reduced to the extent expected using sugar as a reductant, this was not the case when using glycolic acid as a reductant (samples RSM-2-049 through -067). As shown in Table 4.9 and Figure 4.1, not only was the predicted quantity of glycolic acid insufficient to produce a reduced glass (Fe^{+2}/Fe_{tot} ratio of 0.2 to 0.3), a 50% excess failed to reduce iron to any significant extent. This does not agree with previous laboratory crucible tests and will have to be verified in the laboratory or during subsequent testing (a 70% glycolic acid solution was used in the RSM-2 test run, while glycolic acid crystals were used during crucible testing). The volatility of glycolic acid may be responsible for its inability to interact and effectively reduce oxidized feed constituents under thermal processing conditions. Additionally, the use of glycolic acid as a reductant results in a thicker, dryer looking cold cap, as compared with the use of sugar. The glycolic acid cold cap was constantly accumulating, requiring lower feed rates than when sugar was used as the reductant.

Table 4.9. SBW Reductant Type and Loadings vs. Glass Oxidation State

		Reductant		
Sample#	**Date/Time**	**Cmpd**	**g/L-SBW**	**Fe^{+2}/Fe_{tot}**
RSM-2-007	8/6/01 23:00	Sucrose	200	0.18
RSM-2-009	8/7/01 1:10	Sucrose	200	0.23
RSM-2-010	8/7/01 3:25	Sucrose	200	0.38
RSM-2-012	8/7/01 5:20	Sucrose	200	0.35
RSM-2-014	8/7/01 9:20	Sucrose	200	0.41
RSM-2-018	8/7/01 14:50	Sucrose	200	0.34
RSM-2-020	8/7/01 16:45	Sucrose	180	0.40
RSM-2-021	8/7/01 19:10	Sucrose	180	0.89
RSM-2-023	8/7/01 21:36	Sucrose	46	0.72
RSM-2-025	8/8/01 0:05	Sucrose	46	0.41
RSM-2-026	8/8/01 2:39	Sucrose	46	0.26
RSM-2-027	8/8/01 4:35	Sucrose	170	0.30
RSM-2-029	8/8/01 5:50	Sucrose	170	0.25
RSM-2-031	8/8/01 7:30	Sucrose	170	0.23
RSM-2-032	8/8/01 8:45	Sucrose	170	0.19
RSM-2-035	8/8/01 9:45	Sucrose	170	0.17
RSM-2-036	8/8/01 10:22	Sucrose	170	0.19
RSM-2-037	8/8/01 11:13	Sucrose	170	0.15
RSM-2-039	8/8/01 13:10	Sucrose	175	0.18
RSM-2-040	8/8/01 14:50	Sucrose	175	0.19
RSM-2-043	8/8/01 16:18	Sucrose	175	0.09
RSM-2-044	8/8/01 18:00	Sucrose	175	0.13
RSM-2-049	8/8/01 21:20	Glycolic	280	0.06
RSM-2-051	8/8/01 22:52	Glycolic	280	0.04
RSM-2-053	8/9/01 3:26	Glycolic	280	0.02
RSM-2-055	8/9/01 5:25	Glycolic	280	0.01
RSM-2-060	8/9/01 9:50	Glycolic	340	0.01
RSM-2-062	8/9/01 10:30	Glycolic	340	0.02
RSM-2-063	8/9/01 11:35	Glycolic	340	0.01
RSM-2-065	8/9/01 12:45	Glycolic	364	0.01
RSM-2-067	8/9/01 15:55	Glycolic	420	0.01
RSM-2-074	8/9/01 21:00	Sucrose	160	0.14
RSM-2-076	8/9/01 22:30	Sucrose	160	0.09
RSM-2-078	8/10/01 1:10	Sucrose	160	0.05
RSM-2-081	8/10/01 3:15	Sucrose	160	0.03
RSM-2-100	8/10/01 22:30	Sucrose	250	0.15

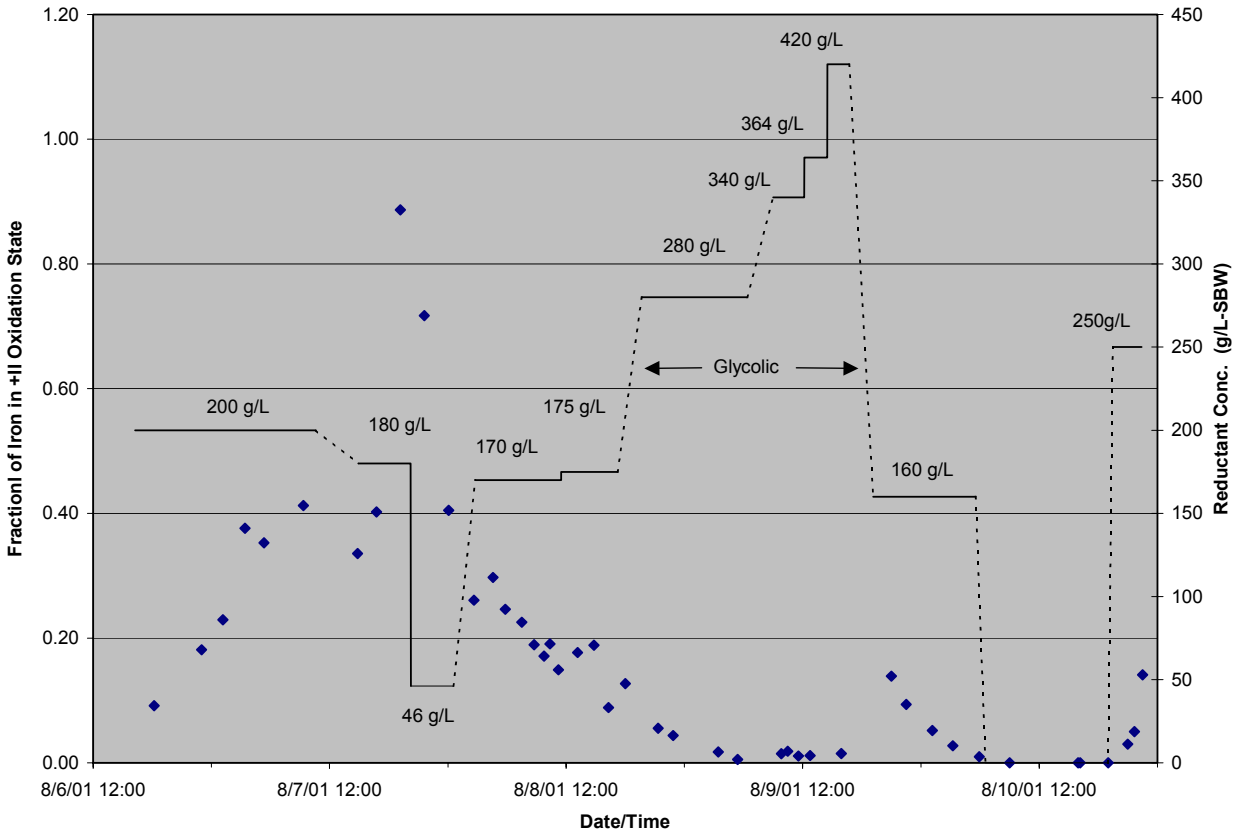

Figure 4.1. Historical Sugar/Glycolic Feed Concentrations and Glass Oxidation-State Values

The glass oxidation-state results presented in Table 4.9 and Figure 4.1 clearly show over-reduced conditions for samples RSM-2-021 and -023. In this upset case, the glass Fe^{+2}/Fe_{tot} ratio was 0.9 (extremely reduced). Significant foaming in the melt also occurred during that period. No error was identified in the makeup sheets pertaining to the 20-L batch that was adjusted to a target sugar concentration of 180 g/L-SBW. It now appears that inadequate tank agitation created nonuniform delivery of sugar to the melter during this processing campaign (see Section 6.1.1). To recover from this highly reduced condition, feed with very little reductant (46 g/L-SBW) was processed for 3-h after which the sugar content was adjusted to 175 g sugar/L SBW, just slightly lower than the previous target.

The overall glass oxidation-state results suggest that SBW sugar concentrations as high as 175 g/L are adequate for maintaining acceptable glass-oxidation conditions. This is somewhat surprising given that feed containing up to 160 g-sugar/L-SBW produced acceptable reduced glass during the previous test RSM-1, despite the fact that the SBW-9 formulation did not require the additional acid (100 cc of HNO_3/L-SBW) to maintain the pH of the melter feed stream ≤ 2. The steep slope of the redox curve at moderate Fe^{+2}/Fe_{tot} ratios is apparently responsible for this very nonlinear result.

4.5.1.2 Off-Line Oxidation State Determination and Vanadium

As discussed earlier, the redox state of a glass is usually described in terms of the fraction of vitreous Fe in the (+II) oxidation state. That ratio is generally determined via sequential spectrophotometric measurements of, first, ferrous and, then, total iron in solutions prepared by dissolving a sample of the glass in HF plus H_2SO_4. Since a primary goal of the second RSM melter run was to compare reductants (sugar and glycolic acid), many such redox-state determinations were performed during the course of the test. However, several problems were encountered, including 1) the overall precision of the method was much worse than usual, 2) the color-forming reaction was much slower than usual, 3) the results did not correlate well with changes in the amount/type of reductant used, and 4) accurate repeatability was extremely difficult to attain.

Not until several weeks after the RSM-2 testing were these problems eventually traced to the fact that the glass formulation employed during that run contained more than twice as much vanadium as it did iron. Vanadium interferes with the chemical determination of a glass's redox state for the following reasons. A glass melt equilibrated at any given oxygen fugacity [$p(O_2)$] will exhibit a vanadyl ion (V^{+4}) to vanadate ion (V^{+5}) ratio close to that of its ratio of ferrous iron (Fe^{+2}) to ferric iron (Fe^{+3}). In other words, in the melter, the redox potentials of the V^{+4}/V^{+5} and Fe^{+2}/Fe^{+3} couples are almost coincident. Conversely, in acidic aqueous solutions, the V^{+4}/V^{+5} redox couple is situated at a much more oxidizing potential than is the Fe^{+2}/Fe^{+3} couple, ~ 1.00 V vs 0.77 V, respectively. This means that vanadate oxidizes ferrous iron during acid-dissolution of any glass that contains both species. This oxidation reaction may then be reversed if a sufficient quantity of a ferrous-ion-selective complexing agent, such as o-phenanthroline (or Ferrozine), is added to the solution; i.e., vanadyl ion will now serve as a reductant for ferric ion. If that back-reaction goes to completion and if the glass originally contained vanadyl ion as well as vanadate, more of the ferrous ion complex that is measured by the spectrophotometer will form than would have been the case if the glass had not contained vanadium. The degree of interference that this causes depends upon the relative amount of vanadium to iron, the glass's "true" redox state in terms of its equivalent $p(O_2)$ level, and the degree to which the reactions go to completion.

Once the root-cause of the problem was identified, research commenced on developing alternative ways of characterizing these glasses' redox state via the analyses of solutions prepared from them. That study entailed the development of ways to

- speciate vanadium in the solutions
- perform a "cold" (in solution) potentiometric determination of redox state
- determine the $\sum$Red/$\sum$Ox ratio (the relative concentration of all reduced species [Fe^{+2}+V^{+4}] to all oxidized species [Fe^{+3} + V^{+5}]).

All of these efforts were successful. Table 4.10 compares the results of various ways of characterizing several RSM-2 glasses.

Of the various "redox ratios" listed in this Table, only the $\sum$Red/$\sum$Ox figures accurately reflect the redox state of the glass while it is in the melter. The amount of analytical bias caused by vanadium in all

of the other approaches increases with the glass's "true" Fe^{+2}/Fe^{+3} (or Fe^{+2}/Fe_{tot}) ratio. Because the degree of bias/error is relatively small for strongly oxidized glasses ($Fe^{+2}/Fe_{tot} < 0.05$), provided that the melter is operated on the oxidizing side of its redox "spectrum," it really does not make much difference which measure of redox state is used.

For completeness, the following paragraphs briefly describe the different redox methods developed during this evaluation.

Table 4.10. Results of Various Ways of Characterizing the Redox States of RSM-2 Glasses

Glass	Fe^{+2}/Fe_{tot} (original)	Fe^{+2}/Fe_{tot} (reworked)	Fe^{+2}/Fe^{+3}	V^{+4}/V^{+5}	∑Red/(∑Red+∑Ox)	∑Red/∑Ox
21	0.90	0.883	7.542	0.417	0.408	0.689
26	0.21	0.387	0.633	0.248	0.272	0.373
36	0.20	0.189	0.232	0.055	0.084	0.092
55	0	0.015	0.015	0.038	0.008	0.008
67	0.01	0.037	0.038	0.035	0.017	0.018
81	0.03	0.069	0.074	0.043	0.031	0.032
94	0	0.006	0.006	0.055	0.005	0.005
100	0.15	0.165	0.197	0.038	0.063	0.067
EA[(a)]		0.162	0.194	NA	0.165	0.198

(a) EA = Environmental Assessment (Jantzen et al. 1993)

Vanadium Speciation. Vanadate ion forms a strongly colored (yellow) complex with tungstate in acetate-buffered aqueous solutions. A few mg of glass are dissolved with HF/H_2SO_4 in the usual fashion. An aliquot of this solution is added to an acetate/borate buffer-complexer solution, and tungstate is added. The resulting solution is diluted to a suitable volume, and its absorbance—a measure of the vanadate concentration—is measured at 400 nm. Vanadyl is determined by difference. Another aliquot of the sample solution is oxidized with permanganate, the excess permanganate is destroyed with sulfite, and "total vanadium" is determined as described previously. The difference in the two absorbances is proportional to the concentration of vanadyl ion.

Cold Potentiometry. A few mg of glass are dissolved in the usual fashion. Enough boric acid is added to saturate the solution, a drop of conc. HCl is added, and then most of it is transferred to a platinum crucible. A reference electrode is inserted into the solution, and the potential between it and the crucible is measured with a voltmeter. (The crucible serves double-duty as the container and high-surface-area sensor surface.)

∑Red/∑OX Ratio Determination. To determine ∑Red species, about 20 mg of glass are dissolved in about 10 drops each of HF & H_2SO_4, and a half gram or so of boric acid is added plus enough water to bring the total volume up to ~ 10 cc. An aliquot of that solution is transferred to a calibrated test tube. A drop of a strong solution of ferric sulfate and then an excess of the same acetate-buffered o-phenanthroline solution used for the usual reduction oxidation (REDOX) determination are also added.

After dilution to a suitable volume, the absorbance of the resulting orange color is measured at 510 ηm. That response is a measure of the solution's total reduced species relative to ferric iron.

To determine ∑OX species, an excess of ferrous sulfate is added to another aliquot of the same solution, and then its total Fe^{+3} ion concentration is determined by adding KSCN and measuring the absorbance of the resulting colored (red) Fe^{+3}-SCN_x complex. That response is a measure of all oxidizing species relative to ferrous ion.

4.5.1.3 Real-Time Glass Oxidation State Monitoring

An electrochemical method for determining the melter-glass oxidation state in real time was evaluated during RSM-2 testing. Two platinum wires housed in open-ended alumina tubes were used as electrochemical sensors in this application. A purge gas was used to exclude molten glass from the interior of each alumina tube that housed and sheathed a platinum electrode wire that ran to the tip of each alumina tube.

The construction of each electrode was identical, and operationally they differed only by the purge gas used. The reference electrode used an oxygen purge that kept the glass at the tip of the electrode fully oxidized. The sensing electrode used an inert argon purge that had no influence upon the oxidation state of the glass in the vicinity of the electrode's tip. Under these conditions, the direct current (DC), electrochemical potential developed between the reference and sensing electrodes would, therefore, be a measure of how much more reduced the bulk glass was relative to the fully oxidized condition at the reference electrode.

The results of this evaluation were inconclusive due to the glass dissolution of electrode sheathing material. Proper operation of the reference electrode required controlling localized conditions at the electrode tip. Deterioration of the electrode sheathing material prevented the required control conditions to be sustained over a meaningful evaluation period.

4.5.2 Processing and Feed Oxidizing Condition

Optimal processing conditions for the SBW-22 feed formulation were obtained using sugar over a range of concentrations that spans the acceptable oxidizing conditions of the glass product, i.e., Fe^{+2}: Fe_{tot} at detection limits to 0.3. However, under over-reduced conditions, sugar produced very active glass foaming conditions that upset processing conditions and thereby reduced average processing rates. Melter processing rates using glycolic acid at all concentrations were found to be inferior to that achievable by sugar. Additionally, the use of glycolic acid as a reductant results in a thicker, dryer looking cold cap than that produced by sugar. Moreover, the glycolic acid cold cap was constantly accumulating, resulting in lower feed rates than achieved during operations using sugar reductant. At the highest concentration used, glycolic acid created a thick glass foam layer that was extremely difficult to dissipate. Processing SBW-22 without a reductant modifier also created foaming conditions that significantly impacted processing rates. The very stable foam layer that resulted was extremely viscous

and impacted glass pouring, making the melt rate based upon glass canister accumulations practically impossible to measure.

4.5.3 Compositional Data

Glass-product compositional data associated with representative melter feeds processed during the RSM-2 tests are summarized in Table 4.11 along with the corresponding target value. All major oxide constituents compared quite favorably with their respective target values. However, like the feed-composition data discussed earlier, several trace constituents were not reported due to detection-sensitivity limitations, but others that were reported, were found to be greater than expectations, presumably because of analytical limitations or unintended contributions from the bulk chemicals used. These glass data suggest that the classic semi-volatiles feed components (e.g., B, S, alkalis, etc.) did not partition significantly to the process exhaust. Partitioning values or melter decontamination factors (DFs) will be discussed in a later section dealing with off-gas emission characterization. The average specific gravity of the nonfoaming SBW-22 glass samples was determined to be 2.56 while aerated foam glasses, produced while processing SBW-22 without reductant additive, averaged 2.14.

The Table 4.11 glass samples were also analyzed at two additional analytical laboratories: Savannah River Technology Center (SRTC) and KLM (KLM Analytical, Richland, WA). The glass data from all three laboratories are compared in Table 4.12. Unlike PNNL and SRTC[(a)] laboratories that used wet-chemical analytical techniques (inductively coupled plasma-atomic emission spectroscopy [ICP-AES] and atomic adsorption [AA]), KLM directly measured the composition of the glass matrix using energy and wavelength dispersive x-ray fluorescence techniques. Although the x-ray data have limited accuracy for low Z elements such as B and F, this technique provides a wealth of information for the remaining elements of the periodic table. The overall agreement between these independent data sets is quite good, recognizing the analytical limitations associated with the analytical methods used.

4.5.4 Glass Durability

Representative glass samples generated throughout the duration RSM-2 testing were subjected to standard durability tests. Specifically, glass samples were subjected to both product consistency test (PCT) and TCLP leach testing protocols. The results obtained are summarized below.

4.5.4.1 PCT Test Results

PCT testing was performed according to methods outlined and procedures referenced in a report by Jantzen et al. (1993). This report also gives round-robin leach results for environmental assessment (EA) glass that can be used to check and validate analytical results. The glasses were crushed using a stainless steel mortar and pestle and sized using brass (100 and 200 mesh size) screens. To avoid the problems associated with CO_2 permeation through the Teflon (which significantly lowers the leach rate if/when it

(a) DK Peeler of Westinghouse Savannah River Company, Aiken, SC.

Table 4.11. Oxide Composition of Vitrified SBW-22 Feed Formulation

	Glass Wt% for RSM-2-XYZ Samples								
Oxide	**-004**	**-014**	**-040**	**-069**	**-094**	**-100**	**Avg***	**Target**	**%Dev**
Al_2O_3	7.480	4.080	4.160	3.940	4.580	4.810	4.370	5.450	-20.0
B_2O_3	8.180	4.870	4.830	5.000	4.880	4.820	4.880	4.890	-0.2
BaO	0.151	0.018	0.010	0.007	0.008	0.007	0.008	0.001	480.0
CaO	3.030	3.050	3.410	4.270	3.580	3.590	3.710	4.440	-20.0
CdO	ND	ND	ND	ND	ND	ND	ND	0.016	ND
Ce_2O_3	ND	ND	ND	ND	ND	ND	ND	0.009	ND
Co_2O_3	ND	ND	ND	ND	ND	ND	ND	0.000	ND
Cr_2O_3	0.223	0.105	0.107	0.094	0.115	0.123	0.110	0.041	170.0
Cs_2O	0.027	0.035	0.040	0.048	0.042	0.041	0.043	0.050	-20.0
CuO	ND	ND	ND	ND	ND	ND	ND	0.009	ND
Fe_2O_3	6.030	2.040	1.790	1.790	1.760	1.830	1.790	1.500	19.0
K_2O	2.170	1.530	1.630	1.280	1.770	1.670	1.590	1.490	6.7
Li_2O	3.580	4.440	4.660	4.870	4.940	4.970	4.860	4.890	-0.6
MgO	0.647	1.390	1.540	1.600	1.570	1.580	1.570	1.480	6.1
MnO	0.286	0.096	0.085	0.083	0.145	0.149	0.115	0.161	-30.0
MoO_3	ND	ND	ND	ND	ND	ND	ND	0.004	ND
Na_2O	14.100	9.730	11.100	15.200	13.300	13.000	13.100	13.700	-4.0
NiO	0.233	0.146	0.138	0.081	0.138	0.134	0.123	0.018	590.0
P_2O_5	ND	ND	ND	ND	ND	ND	ND	0.157	ND
PbO	ND	ND	ND	ND	ND	ND	ND	0.047	ND
RuO_2	0.026	0.021	0.035	0.000	0.000	0.039	0.037	0.003	1300.0
SO_3	0.739	0.693	0.904	0.900	0.895	0.934	0.908	0.900	0.9
SiO_2	45.400	52.200	54.300	53.400	55.200	56.000	54.800	54.400	0.7
SrO	0.074	0.099	0.115	0.132	0.114	0.113	0.119	0.113	5.3
TiO_2	0.212	0.036	ND	ND	0.034	0.035	0.034	0.001	4500.0
V_2O_5	1.450	3.770	3.750	5.140	4.640	4.750	4.570	3.900	17.0
ZnO	0.019	0.017	0.012	0.014	0.013	0.013	0.013	0.014	-5.0
ZrO_2	1.680	2.600	1.650	3.130	3.640	0.397	2.210	1.950	13.0
Sum	95.80	91.00	94.30	101.00	101.00	99.10	99.00	99.60	

*Exclusive of -004 and -014.

Table 4.12. Comparative Oxide Composition of SBW-22 Glass

	RSM-01-2-004			RSM-01-2-014			RSM-01-2-040			RSM-01-2-069			RSM-01-2-094			RSM-01-2-100		
Oxide	**PNNL**	**SRTC**	**KLM**	**PNNL**	**SRTC**	**KLM**	**PNNL**	**SRTC**	**KLM**	**PNNL**	**SRTC**	**KLM**	**PNNL**	**SRTC**	**KLM**	**PNNL**	**SRTC**	**KLM**
Ag_2O	ND	ND	0.121	ND	ND	< 0.002	ND	ND	< 0.001	ND	ND	< 0.001	ND	ND	< 0.001	ND	ND	< 0.001
Al_2O_3	7.480	7.920	8.460	4.080	4.380	4.780	4.160	4.630	5.030	3.940	4.270	4.420	4.580	4.670	4.310	4.810	4.840	5.500
As_2O_3	ND	ND	0.002	ND	ND	0.001	ND	ND	0.002	ND	ND	0.001	ND	ND	0.001	ND	ND	0.001
B_2O_3	8.180	8.820	5.310	4.870	5.440	3.060	4.830	5.150	2.000	5.000	5.220	2.670	4.880	4.890	1.770	4.820	4.800	1.580
BaO	0.151	ND	0.008	0.018	ND	< 0.005	0.010	ND	< 0.004	0.007	ND	< 0.004	0.008	ND	< 0.004	0.007	ND	< 0.004
CaO	3.030	3.010	3.360	3.050	3.570	3.760	3.410	3.630	3.820	4.270	3.770	3.930	3.580	3.580	3.670	3.590	3.580	3.680
CdO	ND	ND	0.013	ND	ND	0.013	ND	ND	0.019	ND	ND	0.017	ND	ND	0.016	ND	ND	0.015
CeO_2	ND	ND	< 0.007	ND	ND	0.009	ND	ND	< 0.007	ND	ND	0.010	ND	ND	0.009	ND	ND	0.007
Cl	ND	ND	0.070	ND	ND	0.115	ND	ND	0.139	ND	ND	0.134	ND	ND	0.090	ND	ND	0.088
Cr_2O_3	0.223	0.267	0.322	0.105	0.124	0.157	0.107	0.107	0.140	0.094	0.091	0.134	0.115	0.112	0.158	0.123	0.153	0.199
Cs_2O	0.027	ND	0.032	0.035	ND	0.038	0.040	ND	0.041	0.048	ND	0.048	0.042	ND	0.045	0.041	ND	0.043
CuO	ND	0.014	0.012	ND	0.009	0.006	ND	0.009	0.009	ND	0.009	0.007	ND	0.010	0.006	ND	0.009	0.007
Fe_2O_3	6.030	6.390	5.950	2.040	2.130	1.900	1.790	1.820	1.610	1.790	1.740	1.620	1.760	1.720	1.580	1.830	1.720	1.570
Ga_2O_3	ND	ND	< 0.001	ND	ND	< 0.001	ND	ND	< 0.001	ND	ND	< 0.001	ND	ND	< 0.001	ND	ND	< 0.001
HgO	ND	ND	< 0.001	ND	ND	< 0.001	ND	ND	< 0.001	ND	ND	< 0.001	ND	ND	< 0.001	ND	ND	< 0.001
I	ND	ND	< 0.002	ND	ND	< 0.003	ND	ND	< 0.003	ND	ND	< 0.002	ND	ND	< 0.002	ND	ND	< 0.002
In_2O_3	ND	ND	< 0.002	ND	ND	< 0.002	ND	ND	< 0.002	ND	ND	< 0.002	ND	ND	< 0.002	ND	ND	< 0.002
K_2O	2.170	2.700	2.460	1.530	1.600	1.290	1.630	1.660	1.380	1.280	1.750	1.420	1.770	1.820	1.390	1.670	1.640	1.330
La_2O_3	ND	ND	< 0.004	ND	ND	< 0.004	ND	ND	< 0.004	ND	ND	< 0.004	ND	ND	< 0.004	ND	ND	< 0.004
Li_2O	3.580	3.660	ND	4.440	4.580	ND	4.660	4.690	ND	4.870	4.820	ND	4.940	4.600	ND	4.970	4.470	ND
MgO	0.647	0.662	-0.066	1.390	1.490	1.810	1.540	1.590	1.920	1.600	1.660	1.840	1.570	1.530	1.670	1.580	1.570	1.740
MnO	0.286	0.275	0.253	0.096	0.078	0.076	0.085	0.065	0.064	0.083	0.061	0.059	0.145	0.110	0.107	0.149	0.121	0.117
MoO_3	ND	ND	0.047	ND	ND	0.015	ND	ND	0.012	ND	ND	0.013	ND	ND	0.014	ND	ND	0.012
Na_2O	14.100	14.000	12.000	9.730	12.300	12.400	11.100	13.000	13.100	15.200	13.400	13.200	13.300	13.600	12.800	13.000	12.800	13.100
Nb_2O_3	ND	ND	0.003	ND	ND	4E-4	ND	ND	< 4E-4	ND	ND	< 4E-4	ND	ND	< 4E-4	ND	ND	< 4E-4
NiO	0.233	0.185	0.183	0.146	0.061	0.058	0.138	0.064	0.061	0.081	0.024	0.028	0.138	0.058	0.062	0.134	0.057	0.059
P_2O_5	ND	0.508	0.868	ND	0.183	< 0.367	ND	0.179	< 0.367	ND	0.172	< 0.344	ND	0.160	< 0.367	ND	0.160	< 0.367
PbO	ND	0.052	0.051	ND	0.045	0.045	ND	0.049	0.047	ND	0.049	0.049	ND	0.043	0.046	ND	0.043	0.044
PdO	ND	ND	< 0.001	ND	ND	< 0.002	ND	ND	< 0.001	ND	ND	< 0.001	ND	ND	< 0.001	ND	ND	< 0.001
Rb_2O_3	ND	ND	0.003	ND	ND	6E-4	ND	ND	< 3E-4	ND	ND	4E-4	ND	ND	< 3E-4	ND	ND	< 3E-4
Rh_2O_3	ND	ND	< 0.001	ND	ND	0.001	ND	ND	< 0.001	ND	ND	< 0.001	ND	ND	< 0.001	ND	ND	< 0.001
RuO_2	0.026	ND	0.004	0.021	ND	0.003	0.035	ND	0.005	ND	ND	0.005	ND	ND	0.003	0.039	ND	0.003
SO_3	0.739	0.698	0.799	0.693	0.763	0.949	0.904	0.853	1.050	0.900	0.885	1.050	0.895	0.843	0.949	0.934	0.810	0.949
Sb_2O_3	ND	ND	< 0.002	ND	ND	< 0.002	ND	ND	< 0.002	ND	ND	< 0.002	ND	ND	< 0.002	ND	ND	< 0.002
SeO_2	ND	ND	< 0.001	ND	ND	< 0.001	ND	ND	< 0.001	ND	ND	< 0.001	ND	ND	< 0.001	ND	ND	< 0.001

Table 4.12 (Contd)

	RSM-01-2-004			RSM-01-2-014			RSM-01-2-040			RSM-01-2-069			RSM-01-2-094			RSM-01-2-100		
Oxide	**PNNL**	**SRTC**	**KLM**	**PNNL**	**SRTC**	**KLM**	**PNNL**	**SRTC**	**KLM**	**PNNL**	**SRTC**	**KLM**	**PNNL**	**SRTC**	**KLM**	**PNNL**	**SRTC**	**KLM**
SiO_2	45.400	48.800	46.400	52.200	56.900	52.000	54.300	58.000	53.100	53.400	57.400	51.800	55.200	56.500	51.100	56.000	57.800	52.400
SnO_2	ND	ND	< 0.002	ND	ND	< 0.002	ND	ND	< 0.002	ND	ND	< 0.002	ND	ND	< 0.002	ND	ND	< 0.002
SrO	0.074	ND	0.068	0.099	ND	0.096	0.115	ND	0.110	0.132	ND	0.105	0.114	ND	0.103	0.113	ND	0.098
Te_2O_3	ND	ND	< 0.002	ND	ND	< 0.002	ND	ND	< 0.002	ND	ND	< 0.002	ND	ND	< 0.002	ND	ND	< 0.002
ThO_2	ND	ND	< 0.001	ND	ND	< 0.001	ND	ND	< 0.001	ND	ND	< 0.001	ND	ND	< 0.001	ND	ND	< 0.001
TiO_2	0.212	ND	0.222	0.036	ND	0.045	ND	ND	0.040	ND	ND	0.035	0.034	ND	0.045	0.035	ND	0.060
UO_2	ND	ND	< 0.001	ND	ND	< 0.001	ND	ND	< 0.001	ND	ND	< 0.001	ND	ND	< 0.001	ND	ND	< 0.001
V_2O_5	1.450	1.120	1.320	3.770	3.630	3.700	3.750	3.280	3.320	5.140	3.740	3.820	4.640	3.670	3.750	4.750	3.810	3.780
Y_2O_3	ND	ND	0.006	ND	ND	0.006	ND	ND	0.007	ND	ND	0.006	ND	ND	0.007	ND	ND	0.007
ZnO	0.019	0.007	0.019	0.017	0.002	0.012	0.012	ND	0.013	0.014	ND	0.013	0.013	ND	0.013	0.013	ND	0.013
ZrO_2	1.680	0.921	1.040	2.600	1.730	1.870	1.650	1.890	1.920	3.130	1.660	1.650	3.640	1.940	2.020	0.397	1.960	1.970

occurs), the leach vessels were contained within a glass desiccator, the bottom of which was covered with a limewater solution while in the 90°C convection oven.

The PCT results obtained are presented in Table 4.13. All of the RSM-2 (SBW-22) glasses turned out to be of much better quality than EA glass, and the EA leach results were well within the range of the round-robin results presented in the Jantzen report.

Table 4.13. PCT Leachate Results from SBW-22 Glasses

	Leachate	ppm/(g/m^2/day)			
Sample	**pH**	**B**	**Na**	**Li**	**Si**
EA Glass Obs.	11.7	562/1.18	1540/0.91	180/0.67	882/0.28
EA glass Ref.	11.85	587/1.24	1662/0.97	190/0.71	893/0.28
RSM-023	10.51	28/0.14	131/0.091	29/0.093	155/0.044
RSM-040	10.96	30/0.15	194/0.13	45/0.14	256/0.074
RSM-053-1	11.06	35/0.17	236/0.16	55/0.18	290/0.083
RSM-067	11.04	40/0.20	248/0.17	53/0.17	324/0.093
RSM-081	10.91	46/0.22	278/0.19	59/0.19	382/0.11
RSM-094	10.94	22/0.11	242/0.17	48/0.15	281/0.081
RSM-100	10.9	32/0.16	227/0.16	46/0.15	308/0.089

It should be noted that the normalized leach-rate calculation (g/m^2/day) assumes the same surface area/g quoted in Jantzen et al. (1993) (196 cm^2/g) for EA glass. Thus, normalized leach rate = fraction leached/area per gram/7days: where fraction leached = ppm in leachate/1000/wt% of that element in the formulation. For example, for EA glass, its wt% Na is 12.46, the mean ppm of Na leached during a PCT is 1662, and the area/g of the particles = 0.0196 m^2/g. Therefore, its normalized leach rate is represented by the expression (1662/1000/12.46)/0.0196/7 = (ppm in leachate/wt% in glass)/137=0.97 g/m^2/day.

4.5.4.2 TCLP Test Results

Samples of 100- to 200-mesh glass particles were leached for 18 h with 20× more (by mass) TCLP Leachant #1 (standard EPA Method 1311 conditions) (TCLP 1992). The leachates were then filtered and analyzed via ICP-AES for those RCRA elements known to have been in the glass formulations. The glass samples all met Universal Treatment Standards (UTS) for all elements as demonstrated in Table 4.14. However, the analytical results for two leachate samples (#81 and 94) did have borderline nickel values. In view of both the relatively low concentration of nickel in the waste, and the leach characteristics of the other RCRA metals, it was suspected that the “high” nickel values were due to contamination introduced by the stainless steel mortar and pestle used to crush and mill the glasses.

Table 4.14. TCLP Leachate Concentrations from SBW-22 Glasses

	Leachate Concentration (ppm)					
Sample	**Ni**	**Ba**	**Pb**	**Cr**	**Cd**	**Hg**
RSM-023	3.3	< 0.05	< 0.1	< 0.05	< 0.02	< 0.02
RSM-040	2.8	< 0.05	< 0.1	< 0.05	< 0.02	< 0.02
RSM-053-1	1.8	< 0.05	< 0.1	< 0.05	< 0.02	< 0.02
RSM-067	0.9	< 0.05	< 0.1	< 0.05	< 0.02	< 0.02
RSM-081	7.6	< 0.05	< 0.1	< 0.05	< 0.02	< 0.02
RSM-094	7.8	< 0.05	< 0.1	< 0.05	< 0.02	< 0.02
RSM-100	1.9	< 0.05	< 0.1	< 0.05	< 0.02	< 0.02

A second TCLP test was run on fresh samples of both of these two glasses, taking care not contaminate the new samples. Results showed, as expected, that the TCLP leachates from both glasses 81 and 94 contained much less nickel, 0.12 ppm and 0.78 ppm, respectively. Thus all SBW-22 glasses, even the highly reduced ones, conform to all existing RCRA land-disposal limits (40 CFR 268).

5.0 Process Characterization

RSM testing of INEEL's SBW flowsheet was initiated on August 6, 2001, and concluded on August 10, 2001. During this 120-h period, the SBW-22 glass formulation at 20% waste loading was successfully processed over a range of oxidizing conditions produced by varied concentrations of two separate reductants—sugar and glycolic acid. The operational impacts of processing with and without reductant modifiers and the concentration-dependent effects of each of the reductants upon process stability, glass-oxidation control, molten-salt-phase accumulation, sulfur partitioning, mercury speciation, off-gas properties, and secondary waste-stream composition were all evaluated during the August melter test. The process observations and melter-related test results derived from all phases of RSM-2 testing will now be discussed. Off-gas characterization, secondary waste stream composition, and process byproducts and residuals will be discussed separately in subsequent sections of this report.

5.1 Processing Observations and Parameters

The melting kinetics of the SBW-22 feed formulation using a sugar reductant was found to be similar to that of the previously evaluated SBW-9 formulation (Goles 2001), but its cold-cap characteristics were somewhat different. Specifically, a much more developed structure was produced by the SBW-22 formulation, which, upon feed interruption, required significantly more time to burn off than was observed for SBW-9. Foaming, caused by an inadvertent over-reducing condition, was the only factor that adversely affected processing conditions involving sugar reductant.

The use of glycolic acid reductant with SBW-22 produced a much more developed cold-cap structure than sugar, although, as discussed earlier, it failed as an effective oxidation-state modifier. Due to its ineffective redox control, relatively high loadings of this reductant were used that further increased cold-cap accumulation and concomitant process-rate reductions. At the higher glycolic acid loadings, coning of the cold cap began interfering with the melter feed nozzle and had to be physically knocked down. In addition, excessive gas generation at the bottom of the cold cap created glass-foaming conditions that reduced heat-transfer rates and further increased cold-cap accumulations.

Feed was also processed without any reductant modifier during RSM-2 testing. In all cases, inferior processing conditions resulted. Under these strong oxidizing conditions, glass foaming created very significant processing difficulties. In particular, a thick and very persistent insulating foam/cold-cap layer was created at the conclusion of an extended processing campaign that had to be physically broken up and stirred into the bulk glass before subsequent testing conditions could commence.

These qualitative descriptions of the impact of reductant type and concentration upon SBW-22 processing rates are quantitatively characterized in Table 5.1 and graphically compared in Figure 5.1. These data clearly demonstrate the superior processing rates associated with the entire (intended) range of sugar concentrations used during RSM-2 testing. Excluding the over-reduced processing condition, the overall average glass production rate of sugar-reduced SBW-22 exceeded that achieved for SBW-9 by ~ 20%. However, the peak sustained SBW-9 glass processing rate during the RSM-1 test was 20% greater than that achieved during the current test with SBW-22. Recognizing the conservatism associated with the initial SBW flowsheet testing during RSM-1, the processability of the SBW-9 formulation is probably slightly better than that of SBW-22.

Table 5.1. RSM-2 Slurry Feeding Rates and Calculated Glass Production Rates*

							Reductant	
Start	**Stop**	**kg/d**	**kg/h**	**l/h**	**g-ox/h**	**lbs/h/Ft^2**	**Compnd**	**g/l-SBW**
8/6/01 16:00	8/7/01 8:00	65.5	2.73	1.89	839	9.42	sugar	200
8/7/01 15:07	8/7/01 20:00	53.3	2.22	1.54	683	7.67	sugar	180
8/7/01 20:45	8/8/01 1:30	46.3	1.93	1.34	593	6.65	sugar	46
8/8/01 3:00	8/8/01 12:00	65.6	2.73	1.90	840	9.43	sugar	170
8/8/01 14:52	8/8/01 16:20	55.8	2.32	1.61	715	8.03	sugar	175
8/8/01 21:00	8/9/01 3:30	61.0	2.54	1.76	781	8.77	glycolic	280
8/9/01 4:10	8/9/01 6:18	61.3	2.55	1.77	785	8.82	glycolic	280
8/9/01 10:17	8/9/01 11:36	50.6	2.11	1.46	648	7.28	glycolic	340
8/9/01 12:14	8/9/01 16:00	48.3	2.01	1.40	619	6.95	glycolic	364&420
8/9/01 19:15	8/10/01 0:20	63.8	2.66	1.85	817	9.18	sugar	160
8/10/01 2:00	8/10/01 5:30	63.1	2.63	1.83	808	9.08	sugar	160
8/10/01 7:00	8/10/01 9:00	46.8	1.95	1.35	599	6.73	none	ND
8/10/01 11:09	8/10/01 12:11	47.9	2.00	1.39	614	6.89	none	ND
8/10/01 12:51	8/10/01 14:30	50.1	2.09	1.45	642	7.20	none	ND
8/10/01 17:25	8/10/01 19:10	42.8	1.78	1.24	549	6.16	none	ND
8/10/01 20:30	8/10/01 21:30	61.5	2.56	1.78	788	8.85	sugar	250
Overall Average		55.2	2.30	1.60	708	7.94		

* Feed density: 1.44 g/cc, SBW/Fd volume expansion factor: 1.4, Waste loading: 0.2, SBW g-Ox/L: 124

As is graphically shown in Figure 5.1, the processability of SBW-22 using glycolic acid at any concentration is inferior to that of sugar at all intended concentrations. Although the average rate differs by only ~ 15%, the issue of choice is moot given the ineffectiveness of glycolic acid to control the glass oxidation state at any of the tested concentrations (see Figure 4.1).

The lowest processing rates observed during RSM-2 testing were recorded with an unreduced SBW-22 feed stream. The average feeding rate achieved under these maximum, oxidizing process conditions was 75% of that recorded for sugar. Since operating conditions continually deteriorated over time under these strongly oxidizing conditions, it is questionable, based on RSM-2 results, whether processing of unreduced SBW-22 can be sustained for any reasonably long period of time.

By combining all feed-batch dropout data, an average melter feeding rate for all SBW feeds over the entire processing campaign (feeding and nonfeeding) can be graphically derived as shown in Figure 5.2. The fact that the average overall feeding rate is significantly less than the batch rates previously described is due almost entirely to nonfeeding periods associated with feed-batch preparations. Much higher process efficiency (total operating efficiency [TOE]) would result if batch preparation down times were excluded for these data. As previously discussed, Table 5.1 summarizes feeding-rate data for all individual periods of sustained processing that occurred during RSM-2 testing.

Also included in Table 5.1 are area-normalized, glass-production rates derived from the nominal total oxide loading of the feed (620 g/L), the glass pool surface area, and the corresponding melter feeding rates. These values comfortably exceed the reference (cold-lid) LFCM design production rate of

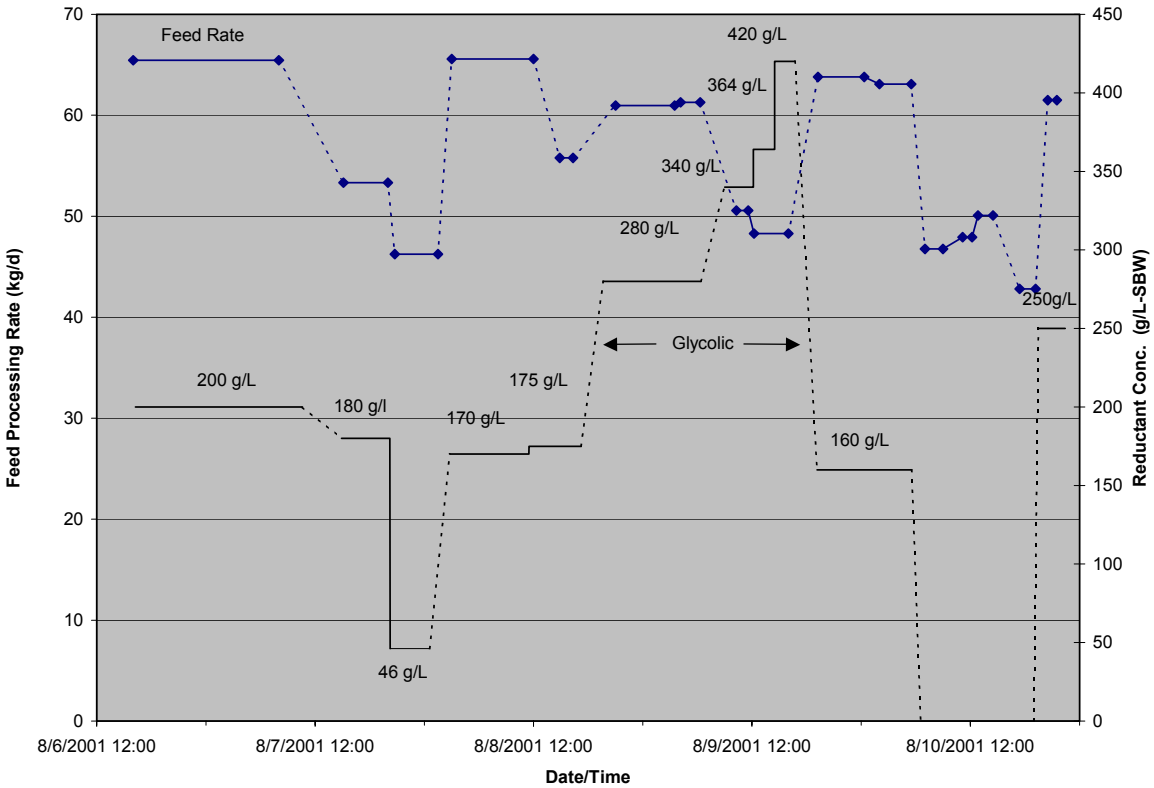

Figure 5.1. SBW-22 Sugar/Glycolic Concentrations and Feed Processing Rates

4 lbs/h/ft^2 that is often quoted and used for flowsheet and equipment-sizing estimates (Perez et al. 2001). Indeed, this reference-normalized production rate is even exceeded when projections are based upon the overall average feeding rate that is inclusive of all melter idling and feed-batching periods. This derived overall average-production-rate value is about 40% higher than that suggested by actual glass-accumulation information that was manually recorded throughout the duration of the test and summarized in Figure 5.3. The lower-than-expected glass accumulation rate is due to the effects of averaging over non-feeding periods and the negative bias introduced by significant glass-sampling activities.

5.2 Operating Parameters

The primary functional indicators of the RSM processing system are temperatures (glass melt, melter plenum space, and post-film cooler off-gas stream), pressure (plenum, off-gas), and melter electrical values (electrode current and voltage). The process data relating to these operating parameters will now be discussed.

5.2.1 Process Temperatures

During RSM testing, the temperatures of the following process items were routinely logged:

- glass melt
- melter plenum

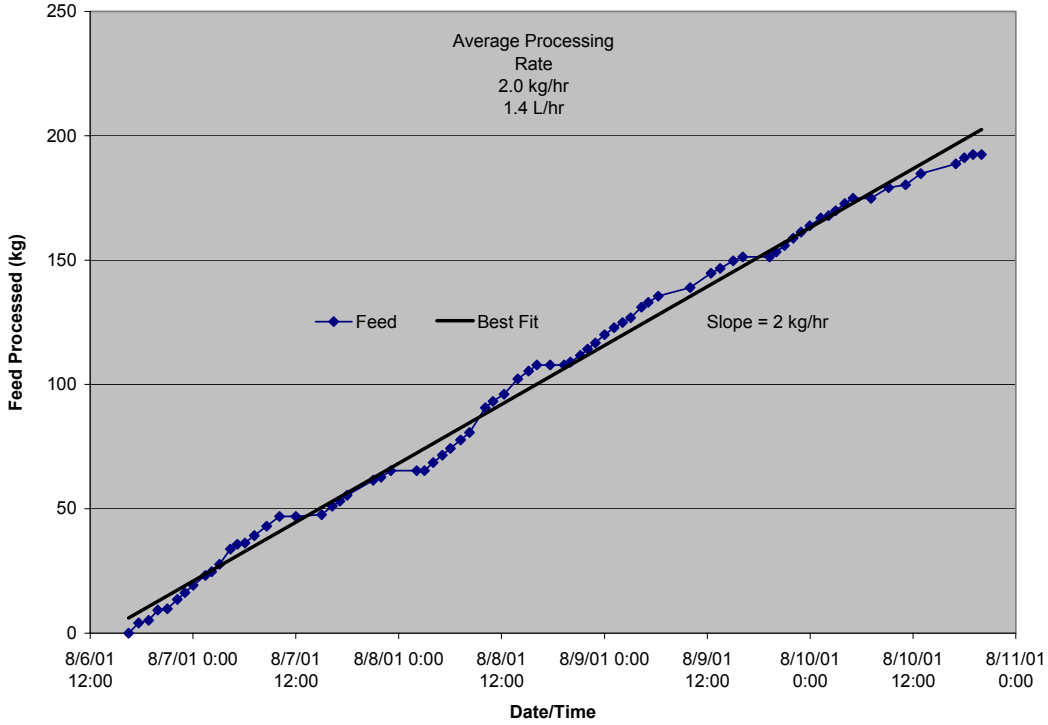

Figure 5.2. RSM-2 Feed Processing History

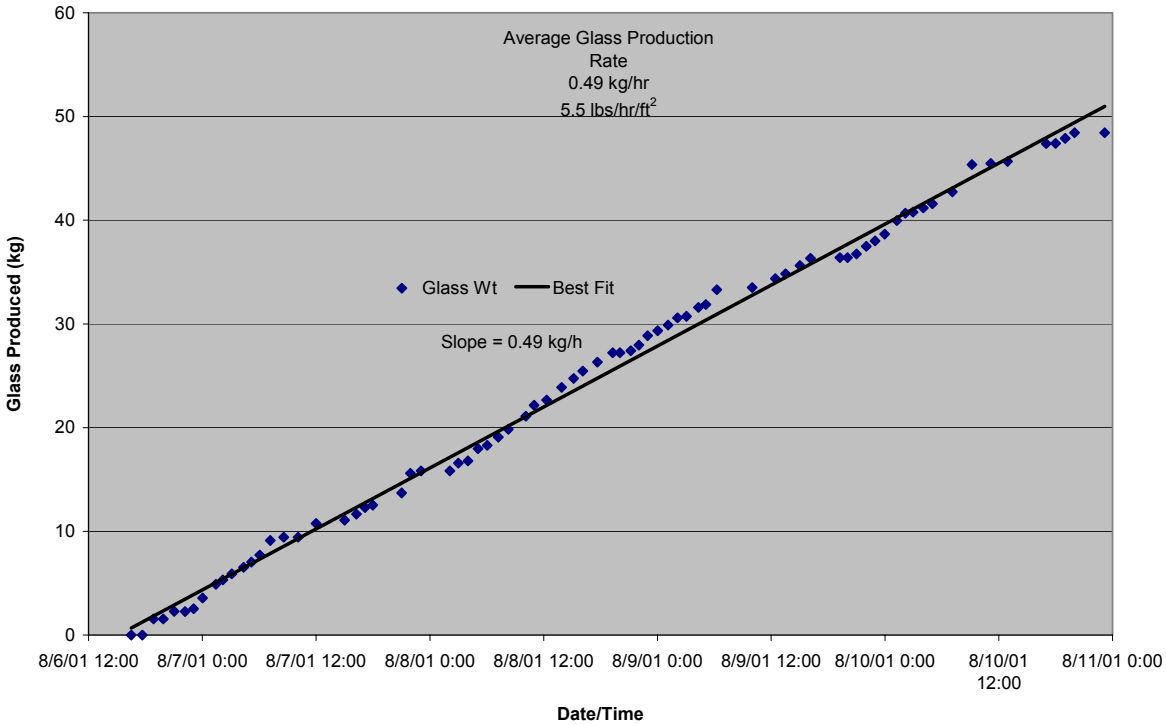

Figure 5.3. RSM-2 Glass Production History

- post film-cooler off-gas flow
- EVS scrub solution
- EVS off-gas exhaust.

Table 5.2 summarizes the maximum, minimum, and average temperatures of the melter's glass, plenum, and off-gas stream for each day of active processing during the RSM-2 test. Also presented in this table are the standard deviations associated with the average temperatures listed. The magnitude of temperature variations about the mean should be indicative of overall processing stability during each day of testing. Overall, these data are consistent with both expectations and earlier measurements recorded during an earlier RSM SBW flowsheet evaluation (Goles 2001). However, melter off-gas temperatures during the current test were successfully maintained at ~ 150°C, 50°C greater that that achieved during the previous (RSM-1) test.

Although the RSM-2 plenum temperatures appear higher than observed during RSM-1 testing, the averages recorded during RSM-1 were exclusive of non-feeding periods, and would, therefore, be biased low relative to the nonselective averaging results presented in Table 5.2. It should also be pointed out that the rather small dispersion in operational plenum temperature is an artifact of RSM design. The plenum temperature is determined by radiative heating from the glass pool and thermal energy provided by a kiln heater surrounding the plenum (see Section 3.1). This auxiliary heat source would be expected, therefore, to modify and damp cold-cap shielding effects to some extent. The process temperature data to be presented in the next section suggests that, for the RSM, the largest possible plenum-temperature swing resulting from 0% to 100% cold-cap transition is ~ 200°C. A 600°C plenum temperature for a 100% melter cold-cap condition is not typical for a cold-lid LFCM of standard design (Perez et al. 2001).

The operating characteristics of auxiliary heat sources supporting RSM operations are summarized in Table 5.3. These data are rather unremarkable in that they reflect operational expectations and previous melter experiences.

All variable process-temperature data collected during the SBW flowsheet evaluations are graphically presented in Appendix E using various logical groupings. The most prominent process-related features associated with the historical displays of glass and plenum temperatures are

- glass-temperature spikes corresponding to power interruption associated with salt-probing operations, or power adjustments required to change glass-temperature
- plenum temperature swings related to melter cold-cap coverage.

The nearly identical responses of both glass-monitoring thermocouples are displayed in these daily graphical presentations. The effects of misalignment (reduced effective cross-sectional area) resulting in temperature differences around the electrode thermowells are apparent during the day shift on 8/7. Realignment of the skewed electrode at 16:30 re-established temperature uniformity for the remainder of the test.

The time-related, plenum temperature responses associated with cold-cap burn off appear to depend on whether reductant is used and also upon the type reductant used. Figure 5.4 displays these time-dependent responses for SBW-22 containing sugar, glycolic acid, and no reductant. Although these

situations depicted are not identical, they nevertheless demonstrate qualitative differences in cold-cap melting characteristics.

Unlike the plenum and melter glass temperatures, the temperature variations of film-cooler, EVS, and HEME exhaust temperatures are, as expected, nominally correlated. The large and somewhat unique post film-cooler temperature excursions observed and displayed in these graphical data (Appendix E) result from higher-than-normal plenum inleakage rates produced primarily by melt-cavity probing activities. Although the temperature history of the EVS scrubbing liquor is correlated with melter off-gas temperature, it provides very little direct process information since its responses are severely damped by its large volumetric mass and the influences of its water-cooled heat exchanger.

Table 5.2. Melter and Off-Gas System Operating-Temperature Characteristics

		Temperature (°C)					
Date/Item		**Glass**	**Plenum**	**MOG***	**EVS Exh****	**HEME Exh**	**Scrub Liq**
8/6/01	Average	1146	657	174	36	ND	32
	StdDev	13	54	31	2	ND	1
	Min	1091	498	127	32	34	29
	Max	1162	771	284	43	34	34
8/7/01	Average	1128	672	134	34	36	31
	StdDev	37	73	14	1	1	1
	Min	1007	548	98	30	34	28
	Max	1181	862	171	36	38	32
8/8/01	Average	1145	627	156	34	36	31
	StdDev	27	76	22	1	2	1
	Min	993	550	123	29	32	27
	Max	1165	825	274	37	39	34
8/9/01	Average	1145	627	156	34	36	31
	StdDev	27	76	22	1	2	1
	Min	993	550	123	29	32	27
	Max	1165	825	274	37	39	34
8/10/01	Average	1145	627	156	34	36	31
	StdDev	27	76	22	1	2	1
	Min	993	550	123	29	32	27
	Max	1165	825	274	37	39	34
8/6–8/10	Average	1145	630	134	36	35	31
	StdDev	12	51	25	1	2	1
	Min	1012	533	97	32	31	28
	Max	1161	768	199	39	39	34

*MOG = melter off-Gas (post-film cooler); ** Exh = exhaust

Table 5.3. Melter Kiln, Pour Spout, and Canister Oven Temperatures

Date/Item		Temperature (°C)			
		Average	Std Dev	Min	Max
8/6/01	Kiln	823	14.80	767	837
	Over Flow	1050	3.54	1030	1070
	Canister	801	2.00	799	805
8/7/01	Kiln	834	5.52	824	849
	Over Flow	1050	5.48	1020	1060
	Canister	795	8.77	773	801
8/8/01	Kiln	833	3.38	824	845
	Over Flow	1050	5.89	1010	1060
	Canister	785	23.50	724	801
8/9/01	Kiln	832	4.88	819	852
	Over Flow	1050	6.95	954	1060
	Canister	787	13.70	744	800
8/10/01	Kiln	832	2.42	825	838
	Over Flow	1050	1.74	1040	1060
	Canister	784	23.90	723	801
8/6 – 8/10	Kiln	832	6.50	767	852
	Over Flow	1050	5.35	954	1070
	Canister	789	18.40	723	805

5.2.2 Process Pressures

Melter and differential off-gas system pressures were recorded throughout the duration of the RSM test. Specifically, the process pressures recorded were

- plenum gauge pressure
- film cooler pressure drop
- EVS pressure drop
- HEME pressure drop.

A graphical summary of these operating parameters extracted from the hourly process log is presented in Figure 5.5. Unlike the previous RSM-1 test, the automatic off-gas control based on plenum pressure was fully operational during RSM-2, and it successfully maintained the melter plenum set point (nominally -2 in. wc), within the capabilities of the blower, throughout all phases of testing. The off-gas film cooler, which accumulated friable surface deposits that restricted flow and ultimately taxed the vacuum capability of the RSM blower system, presented the only challenge to the automated control system. The two sharp increases in film-cooler and total off-gas system pressure drops displayed in Figure 5.5 are illustrative and symptomatic of the growth of film-cooler obstructions. The difference between manual and automatic plenum vacuum control is also illustrated by the 1st and 2nd of these two sequential conductance limiting events, respectively. The 2nd sharp decrease in plenum vacuum illustrated in Figure 5.5 corresponds to probing operations that opened an atmospheric air source to the plenum.

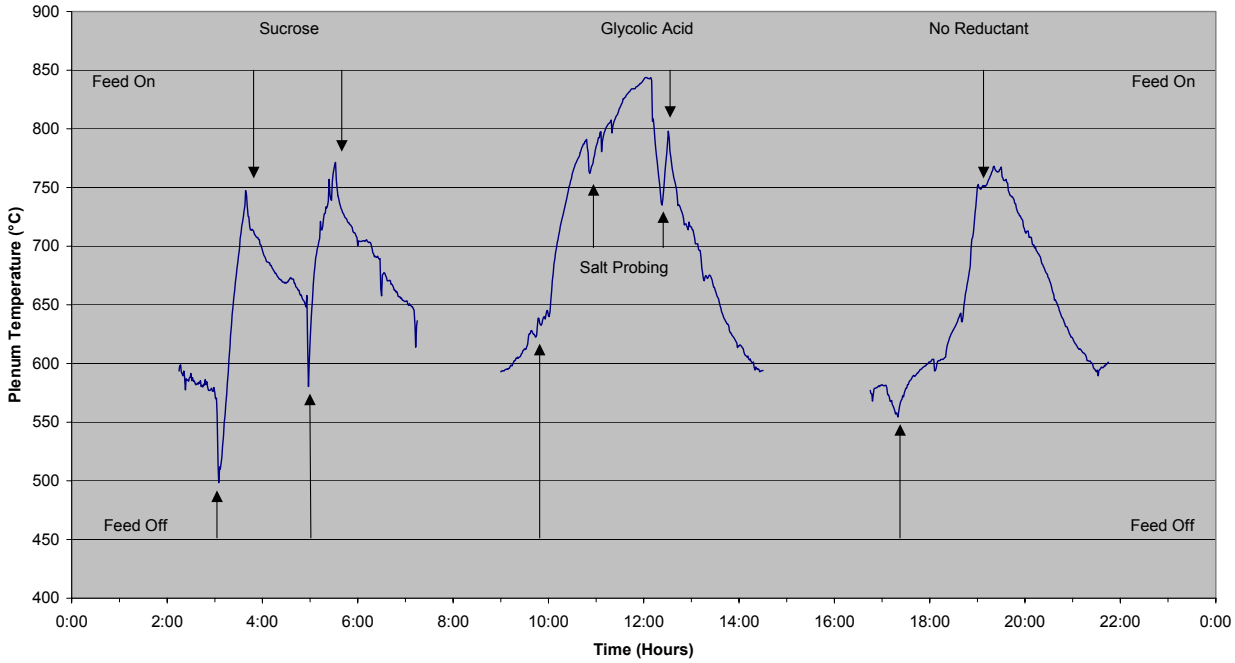

Figure 5.4. Reductant-Dependent Cold-Cap Burn-Off Characteristics
(arrows indicate feed on/off conditions)

Although the manual data in Figure 5.5 only illustrate two film-cooler accumulation events, many more actually occurred throughout the duration of the RSM-2 test. As mentioned above, film-cooler deposits were, in general, friable and easily cleared by striking the film-cooler's exterior with a hammer. However, on two to three separate occasions, a rod had to be used to knock out flow-restricting deposits (see Figure 3.14). It should be noted that the buildup of off-gas debris in the melter's off-gas film cooler is not an expected or natural outcome. In fact, its function is to eliminate or at least minimize this problem! Post-test inspections suggest that a cracked weld between the gas distribution jacket and the sintered metal frit forming the cylindrical inner wall of this device may have significantly impacted the surface area uniformity of the film-cooler's sweep gas.

The pressure-drop history of the remaining off-gas constituents, the EVS and HEME, were remarkably stable throughout the entire duration of the RSM-2 test. Physically, they performed as expected. The secondary waste streams generated by these devices will be discussed in more detail in the next section.

5.2.3 Melter Electrical Data

The RSM's electrodes, kiln, discharge, and pour-spout heating loads are all controlled by phase angle, silicon-controlled rectifiers (SCRs). The SCRs control the voltage going to the load and are capable of adjustments anywhere from zero to the full line voltage (120 V and 208 V). An SCR interface module provides the RSM's PLC with analog signals directly related to the voltage, current and power outputs of each SCR. Although these values were logged at 1-min intervals, data taken at a lower frequency (1-h) are displayed in Figure 5.6 to allow correlations to be easily recognized.

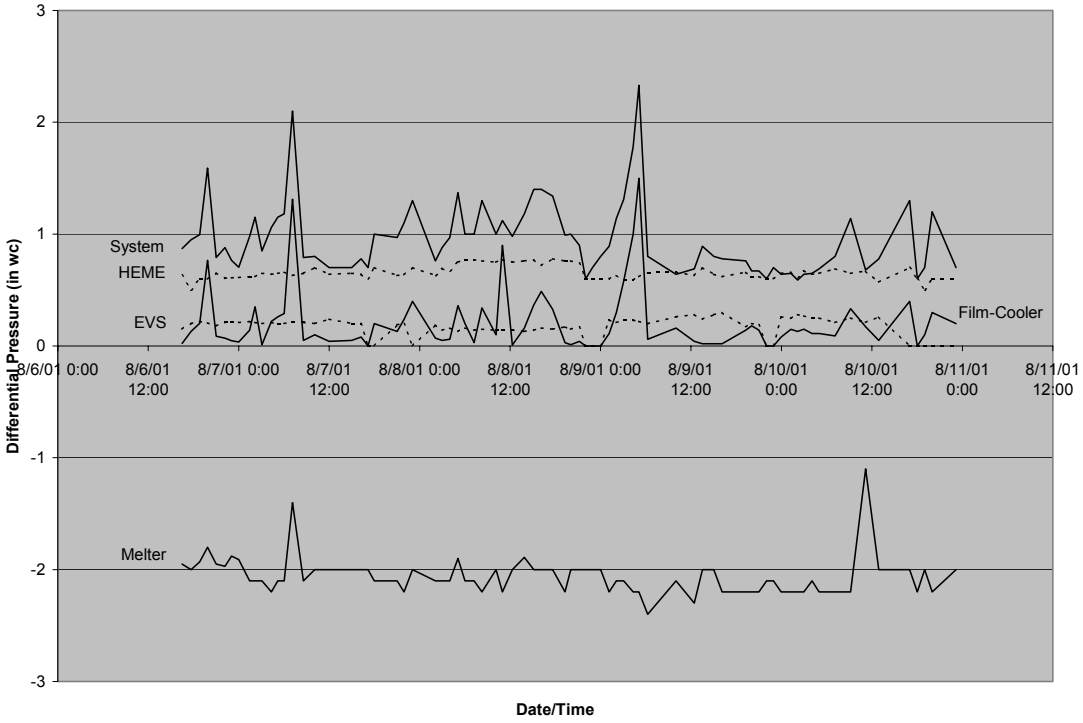

Figure 5.5. Melter Operating and Off-Gas Differential Pressures

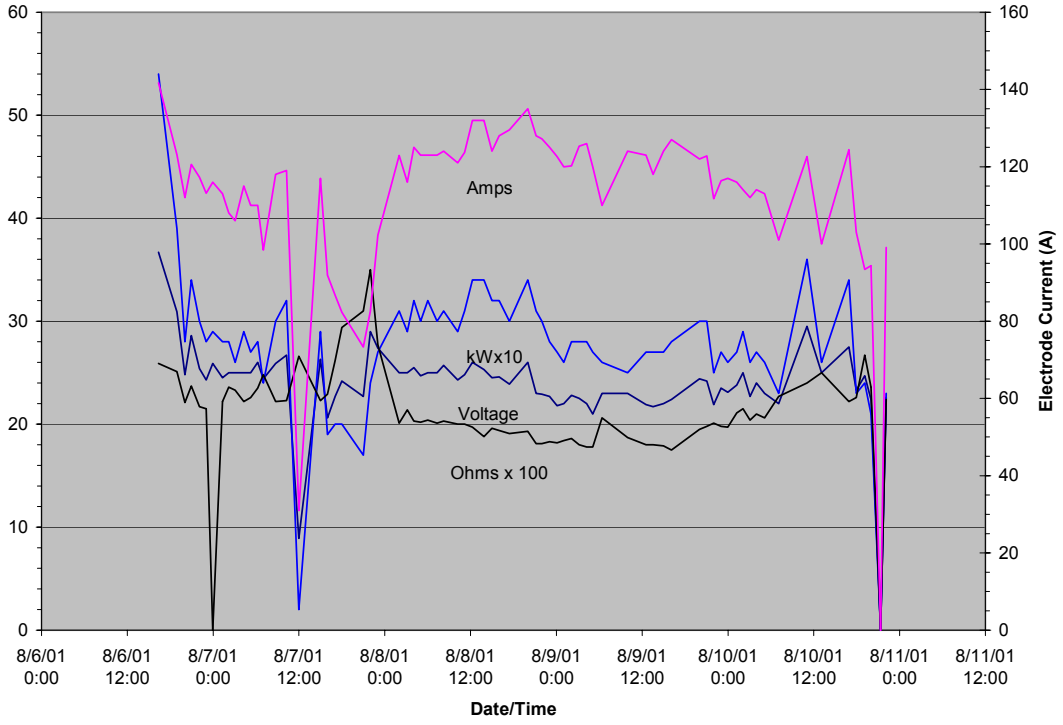

Figure 5.6. RSM Electrode Voltage/Current/Resistance/Power Operating Characteristics

Except for the very noisy period during the last half of 8/7/01 that resulted from power-setpoint alterations necessitated by Redox probe replacements in the early afternoon and glass-foaming conditions toward the end of the evening, the Joule heating electrical properties are quite similar to those recorded during the previous RSM-1 test. As would be expected from the constant power control imposed upon the RSMs' Joule heating electrical circuit, the electrode power fluctuated about a value that remained fairly constant throughout melter testing. Also, the correlated behavior between the electrical parameters, voltage, power, current, and resistance fully reflect their expected functional interrelationships.

Like the instabilities associated in the late evening of 8/7, the somewhat erratic electrical behavior of the melter during the last day of melter testing (8/10) was associated with the processing of SBW-22 without reductant additive. The variability of these melter electrical parameters is a physical manifestation of the processing difficulties qualitatively discussed in the previous section. On the basis of the two campaigns conducted, it appears that long-term continuous processing of this glass formulation without a reductant modifier is not a reasonable option.

Table 5.4 summarizes the maximum, minimum, and average electrode operating parameters and their standard deviations during each day of active processing. The average Joule heating requirements during periods of sustained processing were used with corresponding average campaign-feeding rates (see Table 5.1) to derive specific Joule-energy processing requirements for SBW-22 feeds. These data are summarized in Table 5.5.

Recognizing that a significant fraction of the power required in processing slurry feeds is consumed by boiling away water, the specific energy requirements to process SBW-22 are found to be measurably less than those recorded previously for SBW-9, as expected. Typical energy requirements for slurry-feeding Joule-heated ceramic melters range from 2 to 4 kW-h/kg of glass produced (Perez et al. 2001). The specific energy requirements for vitrifying SBW-22 are at the upper end of this range, which may in part be due to a slightly higher-than-normal weight fraction of water in this melter feed stream. It should also be noted that in addition to the electrical power delivered to the melt pool, significant combustion energy is also being provided by the reductant feed-stream component. By combining this combustion energy source to the Joule heating component for each of the RSM's batch-processing campaigns, slightly higher but much more consistent vitrification specific-energy values are obtained. Although the RSM construction and design is hardly representative of ceramic-lined production melters, the energy expended to vitrify the SBW feeds is, nevertheless, consistent with generalized LFCM operating expectations.

Like the melter electrodes, the RSM's kiln and overflow heater circuits were similarly characterized. Because of the relatively invariant nature of the electrical loads involved, these data do not contain much structure or embedded information and are therefore summarized on a daily basis in Table 5.6.

Table 5.4. RSM Electrode Circuit Operating Characteristics

Date/Item		Temperature (°C)			
		Average	**StdDev**	**Min**	**Max**
8/6/01	Volts	26.600	2.180	17.600	37.300
	Amps	116.000	6.730	68.000	145.000
	kWatts	3.100	0.385	1.200	5.400
	Ohms	0.229	0.018	0.134	0.288
8/7/01	Volts	25.600	3.520	0.378	44.300
	Amps	102.000	19.300	1.550	159.000
	kWatts	2.660	0.707	0.001	6.170
	Ohms	0.258	0.077	0.110	2.070
8/8/01	Volts	24.800	1.410	17.200	36.100
	Amps	123.000	11.300	38.200	157.000
	kWatts	3.040	0.359	0.655	5.550
	Ohms	0.205	0.029	0.181	0.450
8/9/01	Volts	23.200	2.050	1.260	38.800
	Amps	121.000	9.510	5.600	158.000
	kWatts	2.820	0.371	0.007	6.120
	Ohms	0.191	0.019	0.100	0.405
8/10/01	Volts	25.200	3.040	8.550	55.200
	Amps	106.000	13.700	26.700	153.000
	kWatts	2.680	0.547	0.228	7.860
	Ohms	0.241	0.039	0.140	0.388
8/6–8/10	Volts	24.800	2.780	0.378	55.200
	Amps	113.000	16.100	1.550	159.000
	kWatts	2.830	0.532	0.001	7.860
	Ohms	0.224	0.052	0.100	2.070

5.2.4 EVS Condensate Tank, Film-Cooler Injection Air

As described earlier, the EVS acts to both quench the melter exhaust stream and remove entrained debris generated by the melter source. As seen in Table 5.2, the EVS's scrubbing-liquor temperature remained fairly constant (~ 30°C) throughout RSM testing. Consequently, the EVS also would be expected, as in the RSM-1 test, to remove condensable melter-generated gases of which steam is a primary component. Figure 5.7 provides a historical summary of the EVS's scrubbing liquor/condensate tank volume throughout the duration of the RSM test.

These volume data suggest that, except at the very beginning of the test, no significant quantities of steam accumulated during effluent collection campaigns that were always begun with a 60-L charge of 1 M HNO_3. Also presented in Figure 5.7 are the manually recorded Film Cooler, injection-air rotameter readings that have not been corrected for the nominally constant delivery-pressure conditions used. These rotameter readings do not support a flowrate-related explanation for the initial turn down in the steam-collection rate. Indeed, these readings suggest a slight lowering of off-gas flowrates during the last half of the test. However, the EVS exhaust temperatures summarized in Table 5.2 are consistent with low

condensate accumulation conditions, as will be further discussed below after actual flowrate conditions are clarified.

Apart from the film-cooler rotameter readings discussed above, the total melter off-gas noncondensible flow, including the film-cooler injection air, was independently measured, quasi-continuously, throughout the test using He-dilution techniques. In this method, helium gas was injected into the melter's plenum at a fixed rate of 2 L/m. Its concentration in the sampling stream, after being diluted by melter steam and inleakage, film-cooler injection air, and a fixed Ar dilution stream (see Section 3.4), was used to measure and monitor total unquenched melter flowrates. These process flowrates are discussed and characterized in Section 6.1 and graphically summarized in Appendix F.

Table 5.5. Specific Process Energy Requirements for SBW-22 Feeds

		Reductant		Feed	Glass	Power (kW)		SpEnergy (kW-h/kg)	
Start	**Stop**	**Type**	**g/L-SBW**	**l/h**	**lbs/h/Ft^2**	**Joule**	**Combust**	**Joule**	**Total**
08/06 16:00	08/07 08:00	sugar	200	1.89	9.42	2.9	1.1	3.5	4.8
08/07 15:07	08/07 20:00	sugar	180	1.54	7.67	2.4	0.8	3.5	4.7
08/07 20:45	08/08 01:30	sugar	46	1.34	6.65	2.7	0.2	4.5	4.8
08/08 03:00	08/08 12:00	sugar	170	1.90	9.43	3.0	1.0	3.6	4.7
08/08 14:52	08/08 16:20	sugar	175	1.61	8.03	3.2	0.8	4.4	5.6
08/08 21:00	08/09 03:30	glycolic	280	1.76	8.77	2.8	0.8	3.6	4.6
08/09 04:10	08/09 06:18	glycolic	280	1.77	8.82	2.7	0.8	3.4	4.4
08/09 10:17	08/09 11:36	glycolic	340	1.46	7.28	2.8	0.8	4.3	5.5
08/09 12:14	08/09 16:00	glycolic	392	1.40	6.95	2.7	1.2	4.3	6.3
08/09 19:15	08/10 00:20	sugar	160	1.85	9.18	2.8	0.9	3.4	4.5
08/10 02:00	08/10 05:30	sugar	160	1.83	9.08	2.7	0.9	3.4	4.4
08/10 07:00	08/10 09:00	none	----	1.35	6.73	2.6	0.0	4.4	4.4
08/10 11:09	08/10 12:11	none	----	1.39	6.89	2.9	0.0	4.7	4.7
08/10 12:51	08/10 14:30	none	----	1.45	7.20	2.7	0.0	4.2	4.2
08/10 17:25	08/10 19:10	none	----	1.24	6.16	2.5	0.0	4.6	4.6
08/10 20:30	08/10 21:30	sugar	250	1.78	8.85	2.8	1.3	3.5	5.2
								Average =	4.9
								Std Dev =	11%

Using an average noncondensible flowrate of 13 standard cubic feet per minute (scfm) (see Section 6.1), and a 1.4-L/h average feeding rate discussed earlier and documented in Figure 5.2, the average EVS exhaust temperature required to produce evaporative loss rates equivalent to process steam rates is ~ 34°C. This is, indeed what is observed in the summary data presented in Table 5.2. The high initial average EVS exhaust temperature indicated in this table is apparently misleading, as it includes the influences of the data taken during the nonprocessing period at the start of the test.

Table 5.6. Operational Characteristics of Melter Kiln and Overflow Heaters

		Kiln			OverFlow Heater		
Date/Item		**Volts**	**Amps**	**kWatts**	**Volts**	**Amps**	**kWatts**
8/6/01	Average	34.200	7.900	0.289	128.000	13.300	1.710
	StdDev	8.970	2.060	0.185	5.290	0.544	0.138
	Min	17.000	4.130	0.070	108.000	11.600	1.260
	Max	74.800	17.100	1.280	140.000	14.500	2.030
8/7/01	Average	22.000	5.120	0.167	133.000	13.500	1.790
	StdDev	15.400	3.550	0.143	6.250	0.537	0.152
	Min	1.500	0.360	0.001	116.000	12.100	1.400
	Max	45.800	10.400	0.476	140.000	14.000	1.970
8/8/01	Average	21.200	4.890	0.122	131.000	13.500	1.770
	StdDev	8.890	2.120	0.072	4.420	0.442	0.114
	Min	1.250	0.000	0.000	112.000	11.800	1.320
	Max	39.500	8.930	0.353	140.000	14.100	1.970
8/9/01	Average	15.100	3.420	0.070	133.000	13.600	1.800
	StdDev	8.810	2.100	0.058	5.780	0.537	0.129
	Min	1.000	0.000	0.000	18.500	2.070	0.038
	Max	41.500	9.170	0.380	151.000	15.000	2.260
8/10/01	Average	22.000	5.090	0.134	135.000	13.800	1.850
	StdDev	9.940	2.250	0.100	6.470	0.596	0.168
	Min	1.750	0.465	0.001	111.000	12.000	1.330
	Max	43.000	9.450	0.406	152.000	15.000	2.280
8/6 - 8/10	Average	21.100	4.880	0.136	132.000	13.500	1.800
	StdDev	11.900	2.780	0.121	5.960	0.546	0.147
	Min	1.000	0.000	0.000	18.500	2.070	0.038
	Max	74.800	17.100	1.280	152.000	15.000	2.280

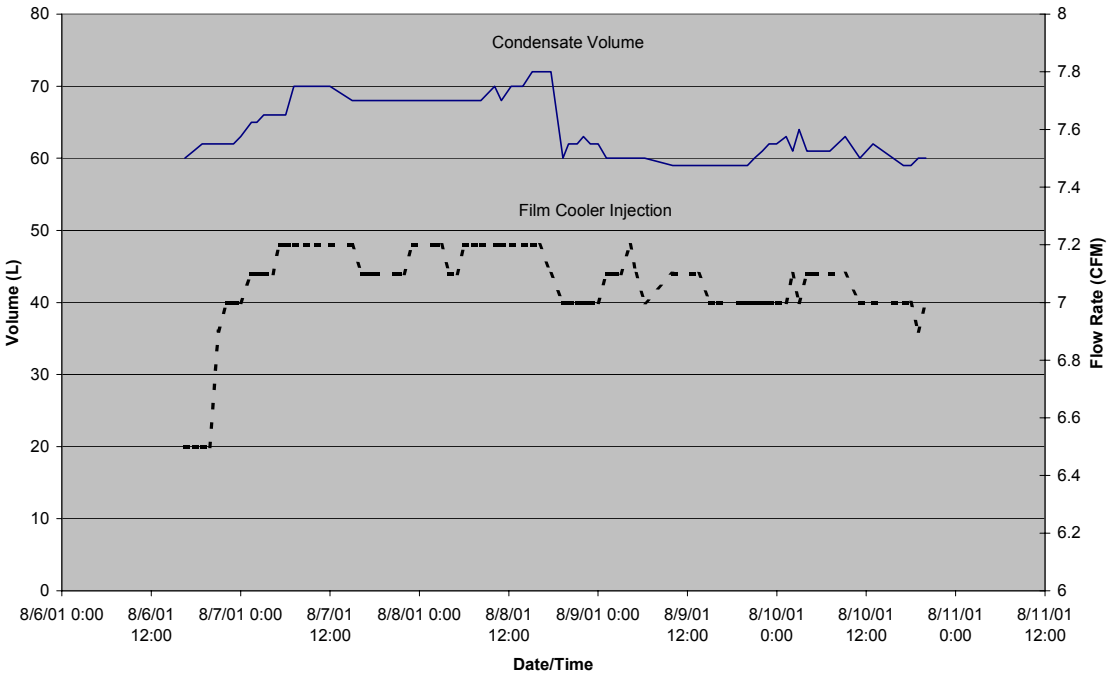

Figure 5.7. RSM-2 Condensate Tank Volume and Film-Cooler Flowrate History

6.0 Melter Off-Gas Emission Characterization

Off-gas emission studies were conducted during SBW-22 flowsheet testing to characterize the melter-effluent source. As described in Section 3.4, the off-gas sampling network assembled in support of this objective was designed to determine the composition of the melter exhaust with regard to non-condensable as well as condensable effluents.

The composition of melter-generated, non-condensable (20ºC) effluent emissions was established using a variety of gas analyzers that are described in Table 3.2. The instruments used were designed to continuously (or quasi-continuously) monitor and record process-exhaust concentrations of H_2, He, N_2, CO, O_2, CO_2, N_2O, NO, NO_2, SO_2 and total hydrocarbons (THC). In addition to these conventionally applied analytical instruments, a continuous emission monitor for volatile forms of mercury was also integrated into the RSM-2 off-gas monitoring network. Discrete sampling for gaseous emissions, including the halogens, hydrogen halides, and volatile forms of sulfur and mercury as well as condensed-phase effluents, was also conducted as described below.

To characterize the melter source of gaseous and condensed-phase effluents, manual sampling trains composed of an absolute filter, condenser, and a series of chemically specific gas scrubbers were employed (see Section 3.4). The manner in which any given element is distributed across the various discrete sampling stages of this device allows the physical/chemical state or states assumed by this effluent species to be inferred.

The operational data and experimental results obtained from the melter off-gas studies conducted in support of the SBW flowsheet evaluations are discussed below.

6.1 Gaseous Effluent

For the surrogate SBW-22 melter feed used during the August 2001 test, CO_2, and NO_x (NO and NO_2) were the major non-condensable (~ 25°C) gases produced by the vitrification process. Table 6.1 summarizes the maximum, minimum, and average concentrations of melter-generated gaseous effluents measured during sustained, continuous processing periods throughout the duration of RSM-2 testing. Since a fixed waste loading of 20% was used throughout the melter test, each of these continuous processing periods is distinguished by date of occurrence, reductant modifier used (if any) and reductant concentration in units of g/L-SBW. The actual time intervals associated with each of these processing periods are uniquely identified in Table 5.1 that has been previously discussed (see Figure 5.1). Also presented in Table 6.1 are the standard deviations associated with the average concentrations listed, and the total off-gas flowrate data. All data in this table represent the actual condition in the melter off-gas jumper between the film-cooler and EVS, including steam, reduced to standard temperature and pressure (STP) conditions.

Table 6.1. Unquenched, Post Film-Cooler, Melter Off-Gas Composition, and Flow Rate (Wet Basis)

	Post Film Cooler Concentration (ppm)										Flow (scfm)
Date/ Red / Conc*	**He**	**H_2**	**O_2**	**N_2**	**CO**	**CO_2**	**N_2O**	**NO**	**NO_2**	**THC**	
8/6-8/7 / S / 200											
Average	5,620	222	180,000	645,000	854	9,280	416	4,560	2,170	62	13
Std. Dev.	493	91	61,700	44,100	700	2,940	202	1,650	1,090	21	1.2
Minimum	3,930	ND	134,000	502,000	ND	ND	ND	ND	ND	4	10
Maximum	6,820	526	604,000	780,000	2,540	15,400	1,580	11,400	6,930	114	18
8/7-8/7 / S / 180											
Average	5,930	253	185,000	692,000	1,620	9,480	208	5,050	457	55	12
Std. Dev.	1,040	170	24,800	93,000	1,160	4,650	151	2,700	664	38	1.5
Minimum	4,450	ND	163,000	615,000	ND	491	ND	28	ND	9	7
Maximum	9,820	1,470	279,000	1,050,000	9,710	38,700	1,500	22,700	5,980	360	16
8/7-8/8 / S / 46											
Average	5,550	ND	195,000	717,000	ND	2,510	110	4,010	1,140	26	13
Std. Dev.	301	ND	8,700	32,200	ND	1,250	93	2,170	664	4	0.7
Minimum	4,800	ND	175,000	648,000	ND	347	ND	9	10	25	12
Maximum	6,080	ND	211,000	781,000	ND	5,820	612	8,570	2,590	55	15
8/8-8/8 / S / 170											
Average	4,740	198	193,000	721,000	957	8,250	296	6,310	750	55	15
Std. Dev.	303	63	7,510	25,700	459	1,890	109	1,860	825	14	1.0
Minimum	3,430	ND	176,000	661,000	ND	612	ND	35	ND	19	13
Maximum	5,610	443	211,000	783,000	2,580	13,900	772	15,800	3,930	99	21
8/8-8/8 / S / 175											
Average	4,840	151	184,000	694,000	759	7,660	308	6,400	1,210	30	15
Std. Dev.	89	26	2,100	7,100	290	1,320	79	1,770	1,180	13	0.3
Minimum	4,700	83	180,000	680,000	ND	4,910	101	4,210	ND	13	14
Maximum	5,120	203	189,000	712,000	1,450	11,100	463	14,300	7,480	83	15
8/8-8/9 / G / 280											
Average	5,370	312	183,000	687,000	58	8,560	325	5,750	925	48	13
Std. Dev.	370	60	4,730	17,800	129	773	73	786	470	6	1.2
Minimum	3,380	118	171,000	641,000	ND	6,350	125	6	ND	33	12
Maximum	5,940	498	190,000	713,000	562	11,000	491	9,040	3,660	64	21
8/9-8/9 / G / 280											
Average	5,670	279	178,000	665,000	283	7,780	355	5,620	711	42	12
Std. Dev.	297	43	1,490	5,450	162	789	70	717	414	3	0.6
Minimum	4,860	194	175,000	654,000	ND	5,730	194	4,080	ND	41	11
Maximum	6,670	406	184,000	685,000	703	10,800	597	7,390	2,050	54	14
8/9-8/9 / G / 392											
Average	5,530	430	172,000	643,000	219	10,100	490	4,030	216	58	13
Std. Dev.	162	105	2,250	7,850	192	1,430	116	916	324	3	0.4
Minimum	5,060	248	166,000	624,000	ND	6,790	223	1,770	ND	50	12
Maximum	5,970	784	177,000	659,000	859	14,500	768	6,720	1,520	75	14
8/9-8/10 / S / 160											
Average	5,250	140	177,000	660,000	387	6,120	199	992	186	89	13
Std. Dev.	80	45	2,620	8,970	329	1,820	104	1,690	517	18	0.2
Minimum	4,990	47	168,000	632,000	ND	2,250	ND	ND	ND	68	13
Maximum	5,440	249	182,000	677,000	1,560	13,000	490	8,320	3,190	162	14

Table 6.1 (Contd)

	Post Film Cooler Concentration (ppm)										Flow (scfm)
Date/ Red / Conc*	**He**	**H_2**	**O_2**	**N_2**	**CO**	**CO_2**	**N_2O**	**NO**	**NO_2**	**THC**	
8/10-8/10 / S / 160											
Average	5,170	102	180,000	672,000	391	5,990	204	ND	0	84	14
Std. Dev.	689	51	12,400	46,100	347	2,180	117	ND	0	13	3.4
Minimum	2,360	ND	128,000	476,000	ND	ND	ND	ND	ND	68	11
Maximum	6,150	268	191,000	713,000	1,710	12,600	582	ND	0	144	30
8/10-8/10 /None / ---											
Average	5,600	ND	191,000	702,000	ND	770	ND	ND	0	64	13
Std. Dev.	182	ND	9,610	34,900	ND	126	ND	ND	2	4	0.4
Minimum	5,280	ND	163,000	598,000	ND	649	ND	ND	ND	55	12
Maximum	6,050	ND	204,000	744,000	ND	1,140	ND	ND	20	69	13
8/10-8/10 / None / ---											
Average	5,400	ND	191,000	702,000	ND	545	ND	4,820	2,430	ND	13
Std. Dev.	412	ND	9,030	32,800	ND	58	ND	1,610	741	ND	1.2
Minimum	4,030	ND	162,000	596,000	ND	432	ND	ND	0	ND	12
Maximum	5,880	ND	199,000	734,000	ND	638	ND	6,940	3,190	ND	17
8/10-8/10 / None / ---											
Average	6,260	ND	195,000	715,000	ND	1,240	ND	5,660	4,010	ND	11
Std. Dev.	737	ND	11,700	42,100	ND	208	ND	1,730	767	ND	1.2
Minimum	4,860	ND	187,000	684,000	ND	799	ND	2,870	2,050	ND	8
Maximum	9,060	ND	246,000	898,000	ND	1,890	ND	12,800	6,150	ND	15
8/10-8/10 / None / ---											
Average	5,900	ND	185,000	677,000	ND	687	ND	3,970	2,840	ND	12
Std. Dev.	476	ND	8,250	29,900	ND	99	ND	990	585	ND	1.5
Minimum	3,200	ND	136,000	499,000	ND	536	ND	ND	46	ND	11
Maximum	6,250	ND	206,000	757,000	ND	867	ND	5,590	4,370	ND	22
8/10-8/10 / S / 250											
Average	5,180	363	172,000	648,000	2,010	11,000	341	5,670	1,460	37	14
Std. Dev.	168	83	2,120	7,410	153	1,300	54	702	362	16	0.4
Minimum	5,000	243	168,000	635,000	1,760	8,090	225	4,320	571	8	13
Maximum	5,620	499	176,000	661,000	2,350	12,700	471	7,170	2,150	62	14
8/6 - 8/10 / All / All											
Average	5,470	163	184,000	683,000	502	6,000	217	4,190	1,230	43	13
Std. Dev.	387	49	11,300	29,000	262	1,390	78	1,290	574	10	1.0
Minimum	4,290	62	163,000	610,000	117	2,530	58	1,160	179	26	11
Maximum	6,430	356	230,000	756,000	1,600	10,900	555	9,110	3,550	89	17

Date/Red/Conc*: Process start & stop date/reductant used - **S**ugar or **G**lycolic acid/reductant concentration - g/L-SBW.

For a nominal, total, post film-cooler melter off-gas flowrate of ~ 13 scfm and an average steady-state feeding rate of 1.5 L/h, the MOG concentrations of the major effluent gases (CO_x and NO_x) were found to be, nominally, 0.68% and 0.61%, respectively. These values are consistent with the concentrations measured during the previous RSM-1 test, given that, in the earlier test, NO_2 data were not available. The combustible gases CO and H_2 were also detected, but at much lower concentrations: 0.076% and 0.025%, respectively. As in the previous test, these combustible-gas concentrations are found to be well below their respective lower flammability limits of 4.65% for H_2 and 15.5% for CO. Even without the benefit of film-cooler dilution, off-gas concentrations of these gases were found to be well below lower flammability as is shown in Table 6.2, which uses the processing feeding rates in Table 5.1, previously

discussed water, nitrate, and reductant feed-stream composition, and the post film-cooler effluent concentrations (Table 6.1) data to project the melter-generated exhaust composition, assuming no melter inleakage, before film-cooler dilution. These data show that even if melter air inleakage is not taken into consideration, no flammability issues in the melter plenum are raised. To generalize these LFCM steady-state processing findings, in the plenum and before the film cooler, steam is the primary factor that eliminates the flammability hazards of H_2 and CO, and the dilution caused by air inleakage and the film-cooler air injection precludes any possibility for auto-ignition downstream of the film cooler and EVS.

Table 6.2. Projected Melter Effluent Concentrations and Flow Before Film Cooler Dilution

	Projected Melter Exhaust Concentration (ppm)								Flow
Date/ Red / Conc*	**He**	**H_2**	**CO**	**CO_2**	**N_2O**	**NO**	**NO_2**	**THC**	**(scfm)**
8/6-8/7 / S / 200									
Average	53,700	2,120	8,170	88,700	3,980	43,600	20,800	595	1.32
Std. Dev.	4,710	871	6,700	28,100	1,930	15,700	10,400	200	ND
Minimum	37,600	ND	ND	ND	ND	ND	ND	43	ND
Maximum	65,200	5,030	24,300	147,000	15,100	109,000	66,300	1,090	ND
8/7-8/7 / S / 180									
Average	67,900	2,900	18,500	109,000	2,380	57,800	5,230	626	1.06
Std. Dev.	12,000	1,950	13,300	53,300	1,730	31,000	7,600	438	ND
Minimum	51,000	ND	ND	5,620	ND	325	ND	103	ND
Maximum	113,000	16,800	111,000	443,000	17,200	260,000	68,500	4,130	ND
8/7-8/8 / S / 46									
Average	86,700	ND	ND	39,300	1,720	62,700	17,800	402	0.81
Std. Dev.	4,700	ND	ND	19,500	1,450	33,900	10,400	62	ND
Minimum	74,900	ND	ND	5,420	ND	145	163	385	ND
Maximum	95,000	ND	ND	90,900	9,560	134,000	40,500	857	ND
8/8-8/8 / S / 170									
Average	54,800	2,290	11,100	95,500	3,420	73,000	8,680	637	1.29
Std. Dev.	3,510	723	5,310	21,900	1,260	21,500	9,550	166	ND
Minimum	39,700	ND	ND	7,080	ND	403	ND	220	ND
Maximum	64,900	5,130	29,800	160,000	8,940	183,000	45,500	1,140	ND
8/8-8/8 / S / 175									
Average	63,900	1,990	10,000	101,000	4,070	84,600	16,000	393	1.10
Std. Dev.	1,170	347	3,840	17,500	1,050	23,400	15,600	170	ND
Minimum	62,100	1,100	ND	64,900	1,340	55,600	ND	178	ND
Maximum	67,700	2,690	19,200	147,000	6,110	188,000	98,900	1,100	ND
8/8-8/9 / G / 280									
Average	56,700	3,290	611	90,300	3,430	60,700	9,770	504	1.25
Std. Dev.	3,910	632	1,360	8,160	770	8,290	4,960	62	ND
Minimum	35,700	1,250	ND	67,100	1,320	60	ND	343	ND
Maximum	62,700	5,260	5,930	116,000	5,180	95,400	38,600	673	ND
8/9-8/9 / G / 280									
Average	56,200	2,770	2,800	77,000	3,510	55,600	7,040	416	1.25
Std. Dev.	2,940	427	1,610	7,820	690	7,100	4,100	26	ND
Minimum	48,100	1,920	ND	56,700	1,930	40,400	ND	410	ND
Maximum	66,100	4,020	6,970	107,000	5,910	73,200	20,300	532	ND

Table 6.2 (Contd)

	Projected Melter Exhaust Concentration (ppm)								Flow
Date/ Red / Conc*	**He**	**H_2**	**CO**	**CO_2**	**N_2O**	**NO**	**NO_2**	**THC**	**(scfm)**
8/9-8/9 / G / 392									
Average	66,400	5,170	2,630	122,000	5,880	48,400	2,590	694	1.06
Std. Dev.	1,940	1,270	2,300	17,200	1,390	11,000	3,900	40	ND
Minimum	60,700	2,970	ND	81,500	2,680	21,300	ND	605	ND
Maximum	71,700	9,420	10,300	174,000	9,220	80,700	18,300	901	ND
8/9-8/10 / S / 160									
Average	56,600	1,510	4,170	66,000	2,140	10,700	2,010	956	1.24
Std. Dev.	867	484	3,550	19,600	1,120	18,200	5,580	196	ND
Minimum	53,700	512	ND	24,300	ND	ND	ND	736	ND
Maximum	58,600	2,680	16,800	140,000	5,290	89,700	34,400	1,750	ND
8/10-8/10 / S / 160									
Average	59,100	1,160	4,470	68,300	2,330	ND	1	958	1.23
Std. Dev.	7,870	585	3,970	24,900	1,330	ND	1	143	ND
Minimum	26,900	ND	ND	ND	ND	ND	ND	779	ND
Maximum	70,200	3,050	19,500	144,000	6,640	ND	5	1,650	ND
8/10-8/10 / --- / ---									
Average	89,500	ND	ND	12,300	ND	ND	5	1,030	0.79
Std. Dev.	2,910	ND	ND	2,010	ND	ND	36	57	ND
Minimum	84,500	ND	ND	10,400	ND	ND	ND	877	ND
Maximum	96,700	ND	ND	18,300	ND	ND	315	1,110	ND
8/10-8/10 / --- / ---									
Average	87,800	ND	ND	8,880	ND	78,500	39,500	ND	0.80
Std. Dev.	6,710	ND	ND	938	ND	26,200	12,100	ND	ND
Minimum	65,700	ND	ND	7,030	ND	ND	0	ND	ND
Maximum	95,700	ND	ND	10,400	ND	113,000	51,800	ND	ND
8/10-8/10 / --- / ---									
Average	84,500	ND	ND	16,700	ND	76,400	54,100	ND	0.84
Std. Dev.	9,950	ND	ND	2,800	ND	23,300	10,400	ND	ND
Minimum	65,600	ND	ND	10,800	ND	38,700	27,700	ND	ND
Maximum	122,000	ND	ND	25,500	ND	173,000	83,000	ND	ND
8/10-8/10 / --- / ---									
Average	98,600	ND	ND	11,500	ND	66,500	47,500	ND	0.72
Std. Dev.	7,960	ND	ND	1,660	ND	16,600	9,780	ND	ND
Minimum	53,500	ND	ND	8,970	ND	ND	769	ND	ND
Maximum	105,000	ND	ND	14,500	ND	93,400	73,100	ND	ND
8/10-8/10 / S / 250									
Average	54,600	3,820	21,200	116,000	3,590	59,700	15,400	388	1.29
Std. Dev.	1,760	869	1,610	13,600	567	7,390	3,810	173	ND
Minimum	52,600	2,560	18,500	85,200	2,370	45,400	6,010	83	ND
Maximum	59,200	5,250	24,700	133,000	4,950	75,500	22,700	648	ND
8/6 - 8/10 / All / All									
Average	69,100	1,800	5,580	68,100	2,430	51,900	16,400	506	1.07
Std. Dev.	4,860	544	2,900	15,900	886	16,200	7,210	115	ND
Minimum	54,200	687	1,230	29,000	642	13,500	2,310	317	ND
Maximum	80,900	3,960	17,900	125,000	6,280	111,000	44,200	1,040	ND

Date/Red/Conc* : Process start & stop date/reductant used - **S**ugar or **G**lycolic acid/reductant concentration - g/L-SBW.

Because column configuration of the online gas chromatograph was found to be unresponsive to the effluent-gas species SO_2, a dedicated, SO_2-specific, ultraviolet (UV)-fluorescence analyzer was incorporated into the RSM's continuous emission monitoring system (see Figure 3.10). Although this instrument's lower limit of detection (as specified by the manufacturer) is < 0.1 ppm, an NO interference response (~ 1%) limited the sensitivity of this device, on average, to SO_2 concentrations > 50 ppm.

Although almost 2 orders of magnitude below the lower detection limit of the quadrupole mass spectrometer used during RSM-1, this instrument's sensitivity was still insufficient to detect melter-generated off-gas concentrations of SO_2. The lack of instrumental SO_2 data does not necessarily mean that volatility losses for sulfur are small; rather, it is a reflection of the fact that SO_2 production is not thermodynamically favored when plenum temperatures are ≤ 600°C. Unfortunately, sulfuric acid and its anhydride (SO_3), being reactive condensable gases, are not detectable by this, or any other, ambient-temperature gas analyzer; however, the magnitude of these potential losses will be evaluated later in this section when sampling and secondary-waste-stream composition are discussed.

The time-dependent behavior of gaseous process effluent emissions was recorded at nominally 1-min intervals throughout the melter-processing campaign. Because steady-state feeding conditions were maintained throughout most phases of testing, the average process exhaust concentrations of these off-gas components remained nominally invariant, although standard deviations about the norm can be quite large. These concentrations were, however, predictably perturbed by scheduled feed-batch preparations, changes in film-cooler injection rates, and during feed sampling and/or feed-system repair. Appendix G graphically presents, by analyzer type, the temporal behavior of melter off-gas effluents on a daily basis.

6.1.1 Total Hydrocarbon Emissions

The responses of the total hydrocarbon analyzer (reported as total hydrocarbons, THCs, in units ppm of CH_4), illustrated in Figure 6.1, indicate that incompletely oxidized hydrocarbons with significant room-temperature vapor pressures were present in the melter exhaust except, as expected, during the processing period when feed reductant was not being used. Furthermore, the highest and lowest sustained THC off-gas concentrations occurred at the highest and lowest organic feed loadings (> 350 g-glycolic/L-SBW and no reductant), respectively. The high and noisy spiked emissions of THC beginning on 8/7 at 16:30 resulted from reduced film-cooler dilution coupled with increased plenum inleakage that were a direct consequence of operational maintenance to reposition the melter's electrodes. The resultant reduction in the off-gas dilution rate significantly increased the concentration of THCs (as well as all other melter-generated effluents) over a 30-min. period as is more clearly shown in Figure 6.2. However, soon after the film-cooler injection rate was re-established to its nominal operational value and all effluent rates decreased, unstable dynamic foaming conditions commenced. This again increased THC emission rates as the incoming feed stream was enveloped by hot-glass foam. These elevated THC emissions continued until 8/7 at 19:00 when the foaming subsided.

It is interesting to speculate what caused the foaming instability during the processing of this feed batch containing 180 g-sugar/L-SBW. During the first 2 h of processing, THC emissions were as one might expect—lower than during the previous batch campaign that contained sugar at 200 g/L-SBW. A glass sample taken just before organic emissions increased showed a slight decrease in glass-oxidation state relative to the condition established by the previous batch. However, the subsequent glass sample taken well after foaming was initiated was highly reduced (see Table 4.9 and Figure 4.1). A common explanation for the foaming, the increased THC effluent concentrations, and the production of reduced glass is a sharp increase in the reductant fed to the melter after the first 2-h of processing. A lab-book

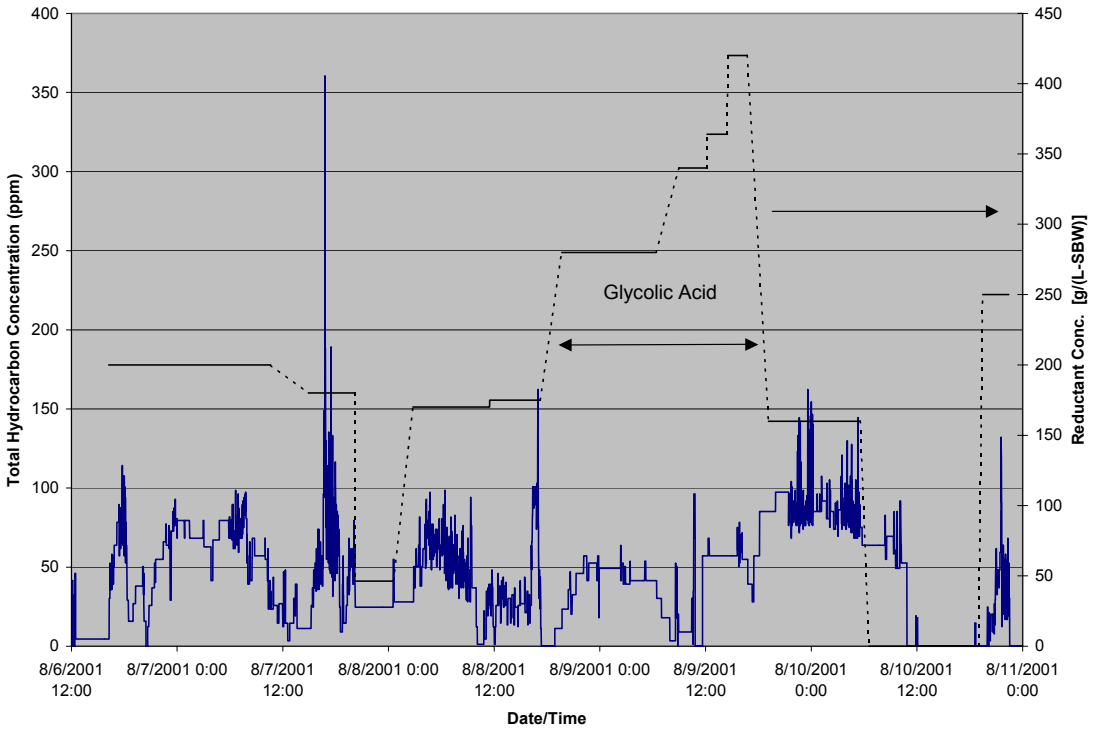

Figure 6.1. Post Film-Cooler THC and SBW-22 Sugar/Glycolic Concentrations

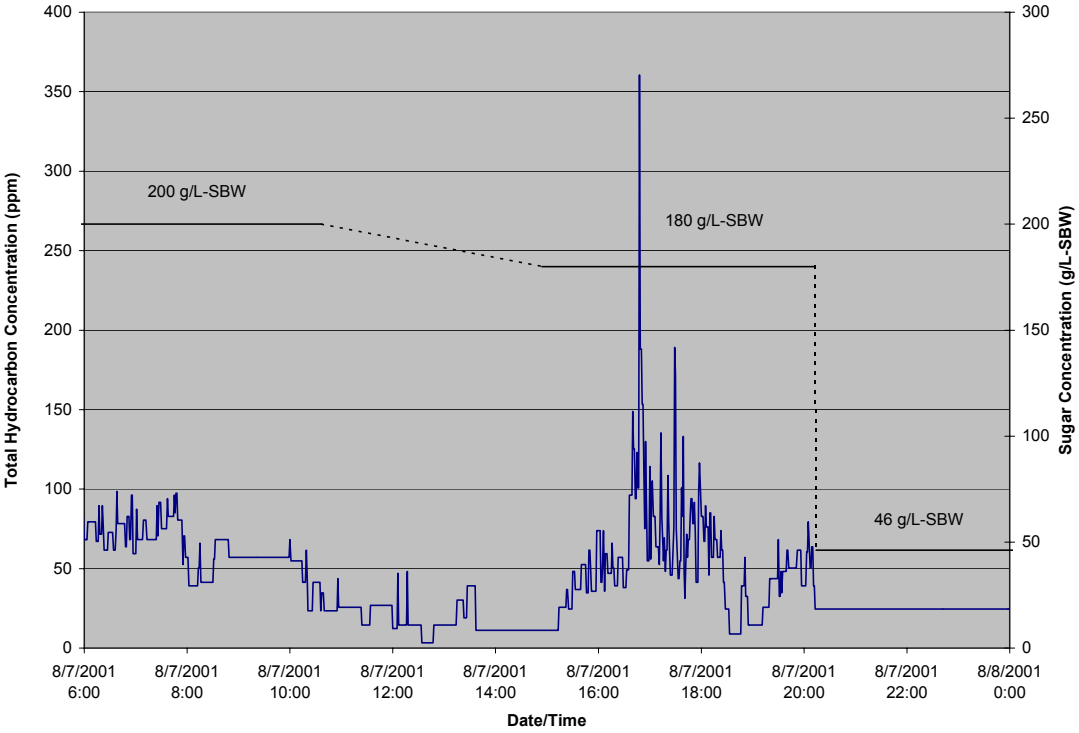

Figure 6.2. Time-Resolved, Post Film-Cooler THC and SBW-22 Sugar Concentrations

entry during this foaming period noted that the melter feed tank was not being properly mixed, which further supports the notion that the sugar added to the batch was not necessarily uniformly fed to the melter.

Overall, the behavior of the melter organic volatile source term was found to be quite similar to that experienced during the previous RSM-1 test. Specifically, although off-gas concentrations of these thermal, volatile byproducts of incomplete oxidation were relatively low, they were nominally found to be functionally related to SBW sugar loadings. It also appears that overfeeding and abrupt introduction of feed material into the hot melter are responsible for many of the volatile organic compound (VOC) concentration spikes that were observed during both RSM SBW flowsheet tests.

6.1.2 Melter Nitrogen Oxide Source Term

The nitrogen oxide concentration data in Table 6.1 were extracted and compared with maximum possible concentrations based on feed nitrate composition and previously characterized processing rates. These normalized data, summarized in Table 6.3, show that, on the average for reduced feeds, ~ 80% of nitrogen fed to melter is reduced to nitric oxide, as opposed to 60% for feed containing no reductant. Furthermore, during the processing of reductant-containing feeds, the cumulative molar concentrations of the nitrogen oxides evolved from the melter were found to be less, on the average, than those predicted from processing conditions (reductant type, concentration, and feeding rates). The fact that these quantities differ suggests the possibility of nitrate reduction to N_2. If one excludes the 8/8 14:52 result, because of its short duration and atypical value, the data in Table 6.3 suggest that 13% of the processed nitrates were reduced to N_2.

As noted in the comments of Table 6.3, the nitrogen oxide analyzer failed on 8/9 and had to be replaced. Although calibrated, the new analyzer appeared to be overestimating NO_x concentrations for the remainder of the test (the last four processing periods). Since the first three of these periods dealt with unreduced feed, and 100% mass closure utilizing NO, NO_2, and N_2O is a logical expectation, an average over-response factor derived from their totals was used to renormalize the totals for the last four processing periods. The effect of renormalization not only improves the mass balance conditions involving processing periods without reductant, but also improves the consistency of the result associated with the final processing campaign that utilized sugar reductant.

If the atypical 8/8 14:52 data are excluded and the renormalized result from the final processing campaign is combined with early test results of reductant loaded feeds, an average of ~ 15% of the nitrates fed to the melter appear to have been reduced to N_2. In addition, the reduction percentage to N_2 appears to be related to reductant concentration.

Table 6.3. Campaign-Dependent Nitrogen Oxide Mass Balance Data

		Reductant		% Maximum Theoretical				% Reduced to N_2		
Start	**Stop**	**Compnd**	**g/l-SBW**	**NO**	**NO_2**	**N_2O**	**Total**	**Data**	**Norm***	**Comments**
8/6/2001 16:00	8/7/2001 8:00	sugar	200	53	25	5	83	17	17	ND
8/7/2001 15:07	8/7/2001 20:00	sugar	180	69	6	3	78	22	22	ND
8/7/2001 20:45	8/8/2001 1:30	sugar	46	67	19	2	87	13	13	ND
8/8/2001 3:00	8/8/2001 12:00	sugar	170	87	10	4	101	-1	-1	ND
8/8/2001 14:52	8/8/2001 16:20	sugar	175	101	19	5	125	-25	ND	Short 1.5-h Period
8/8/2001 21:00	8/9/2001 3:30	glycolic	280	75	12	4	91	9	9	ND
8/9/2001 4:10	8/9/2001 6:18	glycolic	280	69	9	4	82	18	18	ND
8/9/2001 10:17	8/9/2001 11:36	glycolic	340	ND	ND	ND	ND	ND	ND	ND
8/9/2001 12:14	8/9/2001 16:00	glycolic	364&420	64	3	8	75	25	25	ND
8/9/2001 19:15	8/10/2001 0:20	sugar	160	ND	ND	3	ND	ND	ND	Analyzer Problem
8/10/2001 2:00	8/10/2001 5:30	sugar	160	ND	ND	3	ND	ND	ND	Analyzer Problem
8/10/2001 7:00	8/10/2001 9:00	none	ND	ND	ND	0	ND	ND	ND	Analyzer Problem
8/10/2001 11:09	8/10/2001 12:11	none	ND	80	40	0	120	-20	3	New Analyzer & Cal
8/10/2001 12:51	8/10/2001 14:30	none	ND	78	55	0	133	-33	-8	New Analyzer & Cal
8/10/2001 17:25	8/10/2001 19:10	none	ND	67	48	0	116	-16	6	New Analyzer & Cal
8/10/2001 20:30	8/10/2001 21:30	sugar	250	76	19	5	100	0	19	New Analyzer & Cal
* Renormalization only applied to 8/10 data.								Red Ave =	15	Exclusive 1.5-h Test

Table 6.3 also reveals another interesting process-related trend. It appears that nitrous oxide off-gas concentration is the only reliable indicator of the reducing power (reductant type and concentration) of the feed reductant being processed. This may be due to its chemical stability relative to NO and NO_2.

6.1.3 Melter Carbon Oxide Source Term

As previously conducted for nitrogen oxides, the off-gas concentrations of the carbon oxides, CO and CO_2, or CO_x, were compared to maximum possible conditions based on the reductant used, its concentration and measured processing rates for the campaigns previously discussed. THCs were not included in this analysis because of their negligible relative concentrations. The resultant normalized carbon oxide data, summarized in Table 6.4, illustrate that CO_2 represents over 90% of the melter's CO_x source term irrespective of reductant identity or feed concentration. Although the relative proportions of carbon oxides produced are not apparently related to reductant type, their cumulative yield apparently is. Except in two instances the total CO_x yields during the processing of sugar containing feeds are significantly greater than when glycolic feeds are used.

Table 6.4. Campaign-Dependent Carbon Oxide Mass-Balance Data

		Reductant		% Maximum Theoretical				
Start	**Stop**	**Compnd**	**g/L-SBW**	**CO_2**	**CO**	**Total**	**Norm**	**% Unoxidized**
8/6/2001 16:00	8/7/2001 8:00	sugar	200	99	9	109	109	----
8/7/2001 15:07	8/7/2001 20:00	sugar	180	132	22	154	110	----
8/7/2001 20:45	8/8/2001 1:30	sugar	46	143	0	143	102	----
8/8/2001 3:00	8/8/2001 12:00	sugar	170	121	14	135	97	3
8/8/2001 14:52	8/8/2001 16:20	sugar	175	125	12	137	98	2
8/8/2001 21:00	8/9/2001 3:30	glycolic	280	98	1	98	70	30
8/9/2001 4:10	8/9/2001 6:18	glycolic	280	83	3	86	62	38
8/9/2001 10:17	8/9/2001 11:36	glycolic	340	ND	ND	ND	ND	----
8/9/2001 12:14	8/9/2001 16:00	glycolic	364&420	101	2	103	74	26
8/9/2001 19:15	8/10/2001 0:20	sugar	160	88	6	94	67	33
8/10/2001 2:00	8/10/2001 5:30	sugar	160	91	6	97	69	31
8/10/2001 7:00	8/10/2001 9:00	none	----	----	----	----	----	----
8/10/2001 11:09	8/10/2001 12:11	none	----	----	----	----	----	----
8/10/2001 12:51	8/10/2001 14:30	none	----	----	----	----	----	----
8/10/2001 17:25	8/10/2001 19:10	none	----	----	----	----	----	----
8/10/2001 20:30	8/10/2001 21:30	sugar	250	108	20	128	92	8

Indeed the CO_x process yields from sugar-containing feeds exceed expectations by an average of 26%, while glycolic containing feeds are deficient by 4%. Clearly there is a problem with the CO_x calibration for all but, possibly, the initial processing period, as higher than expected yields make no physical sense. Conceptually these data can be clarified by deriving a correction factor that minimizes variations from expectation and apply the factor to normalize the biased data. Since feeds containing sugar do not accumulate a significant cold-cap structure and their yields are overestimated, they formed the basis of the correction factor used.

The resultant corrected data highlight the fact that steady-state processing conditions involving feeds containing glycolic acid were difficult to maintain. These data do not suggest that glycolic acid volatility alone is responsible for the mass balance deficiency, but rather cold-cap growth was also responsible for lower than expected emissions during the monitored portions of these processing campaigns. The extent to which organic carbon was lost to the RSM's off-gas system will be discussed later in this section, when secondary waste stream compositional data are presented.

Although the correction applied to the CO_x off-gas data improves the overall mass balance condition involving the processing of sugar-containing feed, it is not perfect. In 2 of the 8 campaigns involving sugar reductant, poorer mass balance agreement resulted. The reason why these two tests differed from the other six is unknown. The source of this inconsistency and/or the over estimation condition noted above, is not apparent from instrument calibration results logged before, during, and after RSM-2 testing. Off-normal mass-flow responses to varying process gases composition may be responsible for unobservable gas-dilution errors.

6.1.4 Mercury Species Concentrations

Mercury speciation in a complex off-gas is not well understood, and reliable methods for online identification of mercury species have not been developed. Speciation is restricted to differentiating between elemental and oxidized mercury by using an EPA-metals partitioning train or by using chemical pretreatment methods that allow measurement of total mercury and elemental mercury by a continuous emissions monitor (CEM). The oxidized mercury is obtained by the difference between the two measurements. Both the EPA metals train and a CEM were used to measure and speciate mercury in RSM-2 off-gas. Obtaining reliable measurements of elemental mercury and its various chemical forms can often be difficult. Elemental mercury (Hg^o) and oxidized forms of mercury, such as $HgCO_3$, HgO, $HgCl_2$, Hg_2Cl_2 and organo-mercury compounds, adsorb strongly on metal surfaces, filter materials, and particulate matter. At glass-melter temperatures (1000 to 1200°C), mercury is expected to be present in the elemental state. As the off-gas cools, mercury can oxidize.

6.1.4.1 Thermodynamic Model of Mercury Speciation

In an effort to shed light on the mercury speciation in the melter exhaust, the HSC® (a thermodynamic-equilibrium chemistry computer program marketed by Outokumpu Research, Oy, Pori, Finland) thermodynamic equilibrium model predictions shown in Figure 6.3 were developed. Since the model presumes an equilibrium condition, it serves only to indicate what mercury speciation is possible. Several models were developed using off-gas components (CO, CO_2, NO_2, HCl, etc.), measured while the melter was operating under test conditions involving glycolic acid (Conditions 3 and 4), sugar (Condition 5), and no-reductant (Condition 6). These models were very similar to each other. Hence, Figure 6.3 is a reliable representation of predicted mercury speciation under the various test conditions. The model indicates that at the film-cooler outlet, where mercury was measured, the predominant mercury species could have been $HgCl_2$ under favorable kinetic conditions. The formation of $HgCl_2$ and other compounds at the average measured film-cooler outlet temperature of 131°C can occur via the following reactions:

$$2Hg^{o}_{(g)} + 4HCl_{(g)} + O_{2(g)} = 2HgCl_{2(g)} + 2H_2O_{(g)} \qquad \Delta G = -94 \text{ Kcal} \qquad (1)$$

$$2Hg^{o}_{(g)} + O_2 = 2HgO \qquad \Delta G = -33\text{Kcal} \qquad (2)$$

$$4Hg^{o}(g) + 2NO_2 = 4HgO + N_2 \qquad \Delta G = -93\text{Kcal} \qquad (3)$$

$$2HgO + 2CO_2 = 2HgCO_3 \qquad \Delta G = -7\text{Kcal} \qquad (4)$$

Sample lines to the mercury CEM of 30 ft or better may have provided sufficient residence time for reaction. As particles, $HgCO_3$ and HgO can adsorb onto surfaces and other particles, making measurement difficult. As expected, at temperatures greater than about 500°C, gaseous Hg^o is the predominant species.

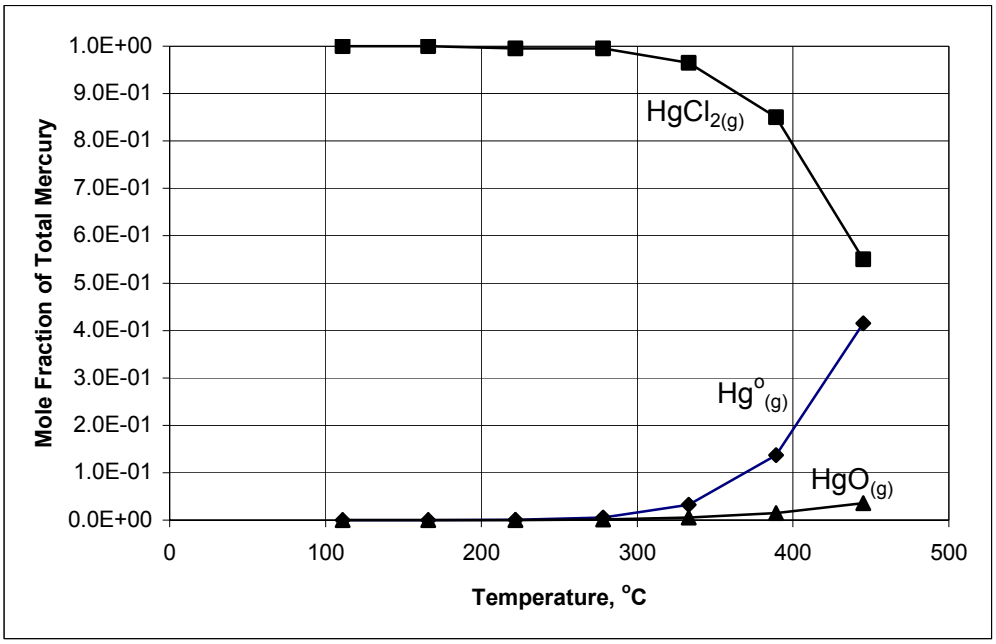

Figure 6.3. Thermodynamic Prediction of Speciation of Mercury Compounds

6.1.5 Mercury Measurements by the CEM

Sampling points for mercury measurements by the CEM were located downstream of the film cooler, EVS, and the HEME. Since there were just two sample lines, one sample line had to be switched between the EVS exhaust and the HEME exhaust. The CEM normally operates with two channels, enabling the near-simultaneous measurement of both Hg^o and oxidized forms of mercury. One channel was inoperable, requiring switching from one channel to the other in order to measure both forms of mercury. This situation, combined with changing conditions in the melter and long times required to achieve stabilization of the mercury measurements, resulted in the inability to obtain a complete set of measurements for all melter test conditions.

Table 6.5 lists mercury measurements for combined test Conditions 3, 4, 5, and 6. Test Conditions 3 and 4 used glycolic acid, while Tests 5 and 6 were conducted with sucrose and with no reductant, respectively. Mercury measurements for combined Tests 3 and 4 indicated that the mercury in the melter exhaust was largely in the elemental form and that 82% was removed by the EVS, and a large portion of the remainder was removed by the HEME. The fact that a large percentage of the mercury was in the elemental form indicates that conditions may not have been sufficiently oxidizing and/or residence times were inadequate for complete oxidation. Overall removal was greater than 97%. This was an unexpected result in that high Hg^o removal efficiencies are not characteristic of wet scrubbers. It is possible that the Hg^o adsorbed onto particulate matter, which was efficiently removed by the scrubber. It is possible that in time, mercury desorbed and became oxidized in the more acidic HEME solutions. Hg^o concentrations in the vapor space above EVS and HEME solutions measured 0.2 to 0.4 and 0.7 to 0.8 mg/m^3, respectively.

The results for test Condition 5 (sucrose) were much the same as the glycolic acid tests. The majority of the mercury in both the melter and the EVS exhaust was in the elemental form, and EVS removal efficiencies were nearly identical to those determined for the glycolic acid tests. The only mercury measurement taken during test Condition 6 (no reductant) was in the melter exhaust. The mercury concentration measured was much lower than for Tests 3, 4, and 5. This is discussed below.

Data comparing CEM and EPA metals-train mercury measurements (see Section 6.2) are listed in Table 6.6. Listed in the table are the estimated total mercury concentrations (E) at the indicated sampling times, the measured concentration (M), and the M:E ratio. This ratio declined dramatically from the glycolic acid tests to the test with no reductant for both the CEM and the EPA metal-train measurements. This indicates that the off-gas may have been sufficiently oxidizing in Tests 5 and 6, resulting in conditions favorable for oxidation of mercury according to the reactions discussed above. This may have been particularly the case when no reductant was used. The NO_2 concentrations were highest (2500 to 3500 ppm) in this case. The oxidation of Hg^o in the presence of 1% NO_2 at 80°C, and the corresponding inability of the CEM to measure the oxidation product, was observed at tests at the INEEL. The speculation was that the oxidation product was HgO in a particulate form that either became adsorbed onto surfaces and/or did not dissolve in the alkaline $SnCl_2$ solutions used to reduce the mercury to Hg^o. The NO_x analyzer malfunctioned during the sucrose test, resulting in no NO_2 measurements.

The metals train and CEM mercury-speciation results for the glycolic acid and sucrose tests listed in Table 6.6 are in good agreement, as is the total measured mercury and the M:E ratio for the glycolic acid tests. For the test with no reductant, very low M:E ratios were obtained for both methods. If oxidized-mercury species were present as particulates, they should have been collected on the metals-train filter unless they became trapped upstream of the sampling point. To resolve some of the uncertainties concerning online mercury behavior, it is recommended that all sampling points be continually monitored.

Table 6.5. CEM Mercury Measurements

				Melter Exhaust					EVS Exhaust						HEME Exhaust	
Date	**Time**	**Test Cond #**	**Reductant**	**Meas. Total Mercury Conc µg/dscm**	**Meas. Hg^o Conc. µg/dscm**	**% Hg^o**	**HgO_X[a] Conc µg/dscm**	**% HgO_X**	**Meas. Total Mercury Conc. µg/dscm**	**Meas. Hg^o Conc. µg/dscm**	**% Hg**	**HgO_X Conc µg/dscm**	**% HgO_X**	**% Total Mercury Rem[c]**	**Meas. Total Mercury Conc. µg/dscm**	**Est. % Total Mercury Rem.[e]**
8/9	0122-1759	3&4	Glycolic Acid	1.60E4	1.51E4	94.2	9.00 E3	5.63	2.84E3	ND	ND	ND	ND	82	BC[d]	> 97
8/9-8/10	2325-0446	5	Sucrose	1.31E4	1.14E4	87.0	1.70E3	1.30	2.53E3	3.09E3	100	-5.60E2[b]	0	81	ND	ND
8/10	1748-1915	6	None	2.13E3	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND

a. HgO_X = oxidized mercury obtained by difference between total mercury and elemental mercury (Hg^o).
b. Negative value due to Hg^o measurement being higher than the total mercury measurement. Measurements were sequential.
c. % Mercury removal by EVS based on mercury measured in melter exhaust and EVS exhaust.
d. BC – Signal obtained for this measurement was well below calibration curve.
e. Estimate of total mercury removed based on total mercury measured in melter exhaust and lowest mercury concentration measurable by calibration curve.

NOTES: 1. All mercury concentrations are reported as micrograms/dry standard cubic meter (µg/dscm) at 20ºC.
2. ND = no data
3. dscm = dry standard cubic meters

Table 6.6. Comparison of CEM and EPA Metals Train Mercury Measurements in Melter Exhaust

Test Cond.	Date	Sampling Time		Reductant	(E) Estimated Total Mercury Conc. µg/dscm	CEM				Metals Train				
		CEM	Met. Train			(M Meas.) Total Mercury Conc. µg/dscm	Ratio M:E	%Hg^o	%HgO_X[a]	(M) Meas. Total Mercury Conc. µg/dscm	Ratio M:E	%Hg^o	%HgO_X	%PM[c]
3&4	8/9	0122-1759	0210-0310	Glycolic Acid	2.33E4	1.60E4	0.69	95	5	1.81E4	0.75	97.7	2.0	0.3
5	8/9-10	2325-0446	2310-0220	Sucrose	2.30E4	1.31E4	0.57	87	13	5.33E3	0.22	90.0	9.9	0.1
6	8/10	1748-1915	1740-1905	None	1.73E4	2.13E3	0.12	ND[b]	ND	1.25E3	0.067	63.8	31.1	5.1

a. HgO_X= oxidized mercury
b. ND = no data
c. PM= particulate matter.

6.2 Sampling and Total Melter Emission Characterization

The previous discussions concerning melter effluents focused upon gas-phase emissions. However with very few exceptions, the effluents that enter the MOG system that require long-term environmental isolation are primarily condensed-phase matter (i.e., aerosols). Many feed components, classified as semivolatiles, are volatilized to some extent within the melter; however, rapid condensation in the melter plenum transforms most of these effluent vapors to airborne aerosols before they can be carried into the off-gas system. Feed and/or glass matter can also be physically ejected into the melter plenum volume by cold-cap and/or glass-surface turbulence. Once in the plenum, this debris can become entrained in gas currents and exhausted from the melter as entrained particulate matter. Both of these loss mechanisms produce off-gas system aerosols; however, the physical characteristics, chemical composition, and escape efficiencies of these two types of airborne matter are markedly different.

Entrained aerosols typically have a mass median diameter of » 1 µm and are compositionally similar to the feed. Consequently, entrainment losses, to first approximation, will influence all feed components in the same way. Feed constituents that fume, e.g., alkali halides, quickly form condensation aerosols, which are predominantly submicron and are chemically dissimilar to the bulk feed. The importance of the volatilization/condensation loss mechanism is totally dependent upon the physical and chemical properties of the feed components and the range of compounds they can form. Consequently, melter aerosol loss rates will be exacerbated by the presence of semi-volatile feed components, and effluent emission rates of elements capable of forming semi-volatile compounds will always be greater than those elements forming refractory non-volatile compounds.

The continuous mercury-emission monitor study previously discussed addressed persistent forms of volatile mercury that are exhausted from the melter and transported to the melter off-gas system's quench-scrubber. However, the melter source of mercury can also include condensed-phase mercury compounds (e.g., HgO) that do not exhibit significant vapor pressures at off-gas stream temperatures (~ 150 °C). In addition, several other melter-generated feed-constituent effluents, such as boron, sulfur, ruthenium, etc., may also challenge the off-gas system in more than one physical state. Consequently, the total-source-term sampling system discussed in Section 3.4 was employed during RSM-2 to characterize both the volatile and condensed-phase source terms of the melter.

6.2.1 Manual Melter Off-Gas Sampling Description

Manual sample-train measurements of the melter effluent source were performed at the film-cooler outlet (see Section 3.3) using the multistage equipment described in Section 3.4. At this location, the total process off-gas flowrate was determined by mass balance calculations using helium dilution techniques, melter feeding rates, and feed-composition data. The film-cooler outlet flowrate ranged around 12 to 15 wet scfm (at 0ºC, 1 atmosphere). The off-gas temperature at the film-cooler outlet ranged from about 120 to 160ºC. Manual train sampling was performed when both sucrose and glycolic acid were added to the feed as reductants, and also when the feed possessed no reductant additives. To the extent possible, operating conditions such as feedrate, melter temperatures, and off-gas temperatures were stabilized and maintained in stable ranges during the manual train-sampling periods. If conditions became unstable during a manual train-sampling period, then the sampling was stopped until the unstable condition was resolved.

Three separate sample-train configurations were used to satisfy off-gas characterization objectives previously discussed. These configurations were designed to satisfy conditions set forth by the following EPA sampling protocols (40 CFR 60):

- EPA Method 0050 for HCl, Cl_2, total F, HI, and I_2
- EPA Method 0060 for multiple metals, including Hg
- EPA Method 8 for SO_2 and SO_3.

Typically the Method 0060 protocol is utilized to characterize 12 to 16 metal analytes. This was increased to 27 metals by calibrating the ICP-AES for all of the metal analytes. Hg was included as one of these target analytes by selecting appropriate sequentially arranged chemical scrubbers: HNO_3/H_2O_2 and acidified $KMnO_4$. Under these conditions, the 0060 sample train was able to differentiate not only between condensed and volatile forms of mercury, but also between chemically-combined, gas-phase mercury species like $HgCl_2$, which are preferentially captured in the HNO_3/H_2O_2 solution, and elemental Hg, which is not readily captured in the HNO_3/H_2O_2 solution but is subsequently oxidized and captured by the second chemical scrubber containing acidified $KMnO_4$ absorbing solution. In a similar way, the EPA Method 0050 and 8 trains allow differentiation between the gaseous oxides of sulfur and the elemental and binary acid forms of the halogens, respectively.

A concentrated tabular summary of off-gas sampling times, conditions, and results are summarized in Appendix I. These data form the basis for the formal discussion of the sampling results that follow.

6.2.2 Aerosol Mass DFs

Effluent loss rates are traditionally expressed in terms of equipment DFs. A device DF value for a particular feed component is derived by taking the ratio of the rate at which the component enters the device to the rate at which it exits. Aerosol DFs are partial DFs that relate to only one off-gas effluent form: aerosols. The melter's aerosol mass DFs, as measured by the filter catches of the differential sampler previously described, are tabulated in Table 6.7 for each of the processing campaigns using sugar and glycolic acid additives and SBW-22 without reductant. These melter DFs are all reasonable in that LFCM gross aerosol DFs normally range from 50 to 100. Moreover, their magnitudes are consistent with previous small-scale melter flowsheet tests that proved to be representative when tested on a larger scale.

However, the much lower result obtained during the processing of sugar-containing feed is somewhat surprising in that processing stability for this feed formulation, in general, exceeded that of all other formulations tested. The fact that much more extensive cold-cap development occurred when processing feeds containing glycolic acid or no reductant may be responsible for the differences observed. Non-steady-state conditions may have also have artificially elevated the DFs associated with the glycolic processing campaign, as processing difficulties involving feed-pile coning and possible cold-cap growth may have occurred during these sampling campaigns.

Melter processing of feed with no reductant also resulted in low overall off-gas particulate partitioning. This no doubt resulted from the development of the stable glass-foam condition that created a rather cold, insulating surface of taffy-like consistency (see Section 5.1) that significantly reduced heat transfer and resultant processing rates (see Figure 5.1) and minimized thermal activities within the melter.

As discussed earlier, processing SBW-22 without a reductant modifier does not appear, on the basis of RSM-2 experiences, to be a realistic option.

Table 6.7. Gross, Post Film-Cooler Melter Aerosol Emission Characteristics

Sample Period		Reductant		Feed	MOG Flw	Aerosol	Melter	
Start	Stop	Type	g/L-SBW	Rate (l/h)	(dscfm)	(mg/dscm)	DF	Loss%
8/8/01 9:55	8/8/01 10:55	Sugar	170	1.9	13.8	896	ND	ND
8/8/01 13:00	8/8/01 13:50	Sugar	170	1.8	13.7	ND	34	2.9
8/8/01 15:22	8/8/01 16:50	Sugar	170	1.6	13.8	1040	ND	ND
8/9/01 0:40	8/9/01 1:40	Glycolic	280	1.8	11.8	204	ND	ND
8/9/01 2:10	8/9/01 3:10	Glycolic	280	1.8	11.7	ND	202	0.5
8/9/01 3:35	8/9/01 4:35	Glycolic	280	1.8	11.9	183	ND	ND
8/10/01 12:15	8/10/01 13:15	None	----	1.5	11.0	313	ND	ND
8/10/01 13:50	8/10/01 14:00	None	----	1.5	11.0	291	ND	ND
8/10/01 17:40	8/10/01 19:05	None	----	1.2	10.8	ND	109	0.9

Also listed in Table 6.7 are off-gas aerosol-loading measurements that were obtained during separate sampling campaigns. These data are consistent with the DFs previously discussed. The highest melter loss rates of particulate matter occurred when sugar-containing feed was processed.

6.2.3 Aerosol Elemental DFs

Individual melter aerosol DFs have been calculated for all melter-feed components using the compositional data derived from off-gas filter samples. A comparison of filter compositional data associated with all previously described processing conditions with the target feed formulation is summarized in Table 6.8. All these data have been normalized by the sample's iron (a classic nonvolatile feed constituent) weight fraction to illustrate the importance of the contributions of the previously discussed aerosol loss mechanisms upon the overall melter-effluent source. These data clearly illustrate the unmistakable influence and importance of thermal fuming (condensation aerosols) upon the melter-particulate source term. For all reductant-modified feeds processed, the concentration of all the classic semivolatiles (B, Cd, Cs, Hg, Pb, Ru, S and all alkali earths, etc.) are seen to be enriched by up to 2-orders of magnitude over their nominal feed-composition values. These results are in total conformity with generalized LFCM effluent-emission expectations developed from past melter-source-term characterization studies (Goles and Schmidt 1992). However, the extent to which semivolatiles are selectively lost to the off-gas system varies, depending on process conditions such as the degree and stability of cold-cap coverage.

Since volatility is a precursor for the generation semi-volatile condensation aerosols that, because of their sub-micron size, are efficiently transported out of the melter plenum into the off-gas system, exposure of the feed stream to hot glass surfaces is the primary factor that influences the magnitude of semi-volatile losses. Consequently, all semivolatiles should be affected in a similar way by changing processing/cold-cap conditions. Table 6.8 clearly shows that the contributions of the semivolatiles in the particulate effluent matter collected in all RSM-2 processing campaigns that used reductant are, indeed, similar.

Table 6.8. Normalized Composition of Melter-Generated Aerosols and Melter Feed

	Norm Particulate Effluent and Feed Composition				
	Sugar		Glycolic	None	SBW-22
Element	170 g/L	160 g/L	280 g/L	----	Feed
Al	8.52	5.47	4.86	2.26	2.76
B	66.80	35.70	9.01	2.58	1.45
Ba	0.02	0.06	0.06	0.00	0.00
Ca	21.00	11.50	4.45	2.71	3.04
Cd	0.25	0.20	0.10	0.01	0.01
Cr	1.01	0.30	0.17	0.05	0.03
Cs	5.84	1.49	0.80	0.06	0.05
Cu	0.12	0.04	0.03	0.00	0.01
Fe	1.00	1.00	1.00	1.00	1.00
Gd	0.01	0.01	0.00	0.00	0.00
Hg	0.21	0.01	0.11	0.01	0.06
K	121.00	27.90	14.50	1.18	1.18
Li	62.10	21.30	6.88	1.89	2.17
Mg	0.00	1.58	0.63	0.70	0.85
Mn	0.26	0.19	0.04	0.11	0.20
Mo	0.14	0.03	0.02	0.00	0.00
Na	666.00	187.00	82.40	9.81	9.72
Ni	0.11	0.05	0.03	0.01	0.01
P	0.85	0.35	0.11	0.08	0.07
Pb	0.31	0.18	0.06	0.06	0.04
Ru	1.03	0.56	0.44	0.02	0.00
S	30.10	10.80	6.72	0.60	0.35
Ti	0.00	0.00	0.00	0.00	0.00
V	4.72	2.91	2.03	0.53	2.09
Zn	0.13	0.06	0.05	0.01	0.01
Zr	0.25	0.04	0.07	0.50	1.38

Having explained the common source for semivolatile loss, the reason for the very different aerosol effluent compositional data associated with the processing of SBW-22 without a reductant modifier can now be described. As discussed in the previous section (see also Section 5.1), a stable, taffy-like foam layer developed over the melter glass pool that effectively insulated the incoming feed from the hot bulk glass. Consequently, no thermally hot surfaces were available to produce the fuming condition that enhances the loss rate of feed constituents capable of forming semi-volatile compounds. Since entrainment was the only effective melter aerosol loss mechanism under these cold-processing conditions, the aerosol effluent collected was almost totally representative of the feed. Indeed, the melter's glass surface was not sufficiently hot enough to even enhance boron losses by a significant factor.

The evaluation of the aerosol-composition data discussed above can also now explain why the high gross aerosol DFs recorded during RSM-2 occurred during the processing of feed with no reductant. Specifically, the cold-melt surface significantly reduced the vigor of feed-pile thermal activities that are responsible for physically ejecting feed and glass material into the melter's plenum. Since the size distribution and the velocities of ejected material would also be influenced by the dynamism of the feed-drying process, a lower off-gas carryover efficiency of plenum airborne matter would also be expected from insulating melt-surface conditions. The combination of low-entrainment efficiency and the loss of

the volatilization/condensation process are, therefore, clearly responsible for the high melter aerosol DF recorded for this feed-processing campaign.

The presence of an extensive insulating cold-cap structure during the processing of feed containing glycolic acid had a similar effect as the insulating foam previously discussed. This in combination with possible cold-cap accumulations previously discussed resulted in a very high apparent DF for the processing campaign using this reductant.

Using the aerosol compositional information previously described, melter DFs associated with aerosol loss for individual elements were calculated, the results of which are summarized in Table 6.9. These tabular results reinforce conclusions of previous discussions that the highest semivolatile-enriched aerosol loss rates occurred during the processing of sugar-containing feed. Although an overall mass DF associated with the processing of melter feed containing 160 g-sugar/L-SBW was not measured, it can be inferred from the data in Tables 6.7 through 6.9. Given that the semivolatile composition of aerosols generated by the 160 g-sugar/L-SBW feed was greater than produced by the glycolic formulation, the similarity of their corresponding DF data suggests that the overall melter particulate DF was significantly better for the 160 g-sugar/L-SBW feed than that recorded for the earlier 170 g-sugar/L-SBW processing campaign. Specifically, the overall melter particulate mass DF achieved during the processing of the 160 g-sugar/L-SBW feed is estimated to be ~ 100, based on relative composition data and the measured mass DF for the glycolic campaign.

Overall, the element-specific particulate DFs recorded during RSM-2 are reasonably close to general expectations and are with few exceptions consistent with previous SBW melter-testing results. Of the four test conditions evaluated, the processing campaign involving the 170 g-sugar/L-SBW feed formulation was most similar to results obtained during RSM-1, although slightly higher on the average. However, significantly lower particulate emission rates for sulfur and cesium were recorded during the current test. Specifically, RSM-2 sulfur partitioning was at worst a factor of 2 less than occurred during RSM-1, and on average was a factor of 5 less. This is a desirable result as SBW-22 was formulated specifically to retain as much feed-component sulfur in the process glass as possible. It must be remembered that the above results do not address volatile losses of this or other elements. To address volatility partitioning, the composition of gas-scrubber sampling components will have to addressed. This as well as the related matter of total melter DFs are the subjects to be discussed in the next section.

6.2.4 Volatile Partitioning and Total Elemental DFs

Since only a very few feed components are lost to the off-gas processing system in the gaseous state, essentially all the aerosol performance values listed in Table 6.9 also represent total melter DFs for these nonvolatile elements. However, notable exceptions to this statement can include elements such as B, Cl, F, Hg, P, and S whose gaseous forms can, under certain processing conditions, dominate melter off-gas system losses and determine their melter DFs (C and N were discussed earlier). Of these effluent elements, mercury, sulfur, and the halogens were specifically targeted for characterization as previously described. Since EPA 0050 and 8 sampling protocols only address volatile forms of sulfur and the halogens, RSM-2 sampling campaigns using these protocols only characterized the volatile component of process losses associated with these elements.

Table 6.9. Elemental Melter DFs Associated with Aerosol Emissions

	Particulate Effluent DFs: SBW-22 & Additives				
	Sugar		Glycolic	None	Overall Ave*
Element	170 g/L	160 g/L	280 g/L	----	
Al	329	461	430	60	165
B	27	45	147	33	41
Ba	79	18	15	13	19
Ca	232	381	817	87	203
Cd	53	61	104	56	64
Cr	40	120	178	40	62
Cs	9	32	49	44	22
Cu	65	183	203	80	105
Fe	1,560	1,410	1,160	75	257
Hg	146	2,150	201	126	198
K	14	54	85	67	34
Li	46	120	306	72	84
Mg	ND	668	1,390	80	272
Mn	307	376	1,460	36	117
Mo	29	130	172	107	70
Na	17	53	100	54	37
Ni	869	1,520	1,980	361	787
P	216	468	1,220	104	233
Pb	143	220	576	38	102
Ru	20	34	35	59	32
S	15	38	50	36	28
V	622	909	1,080	267	542
Zn	73	143	159	39	76
Zr	7,840	42,700	22,600	187	723
Mass	34	ND	202	109	69
* Based on average partitioning values.					

Since the aerosol matter collected by the -8 and –0050 sampling trains was not analyzed for sulfur or the halogens, respectively, volatile partitioning can be simply and directly accessed by the analytical data generated by these sampling trains. As was done for particulate matter, volatile melter DFs, which are partial DFs relating only to volatile losses, can be derived for sulfur and halogen feed components during the RSM-2 processing conditions for which sampling was conducted. These volatile DF data are summarized in Table 6.10.

Unlike sulfur that shows no detectable volatility, the halogen feed components as a group all exhibit high melter volatility losses. The relationships of these losses to process-reducing conditions are somewhat surprising, however. As an example, Table 6.10 indicates that the highest melter DF recorded for fluorine occurred during nonreductant feed-processing conditions. However, one would expect that strong oxidizing conditions would promote higher halogen volatility and concomitant melter losses, not less. Although the chlorine and iodine data are not perfectly correlated, their emission behavior during nonreductant feed processing was not at all similar to that of fluorine. While the off-gas behavior of fluorine characterized during RSM testing may, indeed, be real, it is difficult to rationalize on the basis of systematics and chemistry.

Table 6.10. Volatile Melter DFs for Halogen and Sulfur Feed Constituents

	Volatile DFs: SBW-22 and Additives			
	Sugar	**Glycolic**	**None**	**Overall**
Element	**170 g/L**	**280 g/L**	**----**	**Ave***
Cl	9	14	1	3
F	3	1	23	2
I	1	1	2	2
S	> 18	> 18	> 18	**NA**

* Based on partitioning values.

Although halogen volatile losses were expected, the lack of detectable sulfur volatility (< 2 ppmv[(a)]) throughout all phases of testing was not necessarily a predictable outcome. It is nevertheless a very important validation of the SBW-22 formulation that was specifically designed to maximize the vitreous retention of feed-stream sulfur. This, indeed, has been successfully accomplished because, without significant volatility, the higher-than-expected particulate DF for sulfur now is equivalent to its total DF, which, as previously discussed, is a factor 2 higher than recorded using the previous SBW-9 formulation during RSM-1 testing.

Beyond the halogen and sulfur-specific sampling, the -0060 sampling train also addressed volatility with a focus and emphasis on mercury. Table 6.11 summarizes the percentage of the total mass of each effluent species that was collected by the heated filter of the multicomponent sampling system previously described in Section 3.4. These values represent the percentage of each detected effluent species that partitioned to the off-gas system as condensed phase matter, i.e., aerosols, while the correspondingly volatile partitioning percentages are derived by subtracting these values from 100.

With the exception of mercury, boron and the halogens, that were previously discussed, these data show little evidence for the presence of significant quantities of gas-phase effluents. Moreover, the mercury-volatility systematics are consistent with the corresponding processing conditions. Under reducing conditions, volatile partitioning, presumably in the form of elemental mercury, is essentially 100%. Under oxidizing conditions, about 5% of effluent mercury was collected on the sampler's heated filter, presumably as HgO. This is in complete agreement with the continuous-emission monitoring results previously discussed.

Apart from mercury volatility, the gas scrubbing solutions of the -0060 sampling train, as previously described, were chosen to provide a degree of mercury speciation information as well. Table 6.12 summarizes the distribution of mercury effluent across the filter and sequential H_2O_2 and $KMnO_4$ chemically selective gas scrubber traps. These data show that the majority of the total Hg (86% on average) was captured in the final two $KMnO_4$ impingers, suggesting that most of the total mercury existed as elemental Hg at the film-cooler outlet and sample-train conditions. During the non-reducing feed test, however, only 64% of the total Hg was captured in this fraction, compared to 90 to 98% during tests conducted with reductant.

(a) ppmv = parts per million by volume

Table 6.11. Particulate Loss Percentages of Melter Effluents

	Effluent Particulate % for SBW-22 & Additives				
	Sugar		Glycolic	None	Overall Ave
Element	**170 g/L**	**160 g/L**	**280 g/L**	**----**	
Al	100	100	100	97	99
B	100	100	73	55	82
Ba	90	98	99	100	97
Ca	99	100	100	95	98
Cd	100	100	100	92	98
Cr	100	100	100	85	96
Cs	100	100	100	86	96
Cu	100	100	100	91	98
Fe	100	100	100	97	99
Gd	98	97	92	95	95
Hg	1	ND	ND	5	2
K	100	100	100	96	99
Li	100	100	100	91	98
Mg	ND	98	100	92	72
Mn	100	100	100	98	99
Mo	100	100	100	91	98
Na	100	100	100	97	99
Ni	100	100	100	97	99
P	100	100	100	100	100
Pb	100	100	100	95	99
Ru	100	100	100	100	100
S	100	100	100	100	100
Ti	ND	ND	ND	96	24
V	100	100	100	97	99
Zn	93	84	90	78	86
Zr	100	100	100	100	100

Table 6.12. Distribution of Mercury Effluents Across -0060 Sample Train Components

	% Hg Collected By Device		
Reductant & Concentration	**Filter**	**H_2O_2**	**$KMnO_4$**
Sugar @ 170 g/L-SBW	0.95	5.4	93.7
Glycolic @ 280 g/L-SBW	0.30	2.0	97.7
Sugar @ 160 g/L-SBW	0.09	9.9	90.0
None	5.15	31.1	63.8
Average	1.62	12.1	86.3

In addition, the fact that, on average, 12% of the total Hg was captured in the HNO_3/H_2O_2 impingers under strong oxidizing conditions suggests that up to this amount of the total Hg may exist as gaseous $HgCl_2$ at sampling-train temperatures. However, if the high NO_x levels increase the HNO_3 normality of these impingers or otherwise makes this solution more oxidizing, then some of the elemental Hg may also be captured in this fraction. Both the filter and the H_2O_2 sample components recorded maximum

collection fractions under strongly oxidizing process conditions, which suggests that oxidized forms of mercury, specifically HgO and $HgCl_2$, respectively, are indeed being produced at the expense of elemental Hg. This observation is in total conformity with speciation results derived from the previously discussed mercury monitor and will be further supported by secondary waste-stream composition studies to be discussed in the next section.

The only other feed components that exhibit any sort of systematic presence in these gas-scrubber solutions are boron, barium, gadolinium, and zinc. Boron, as mentioned earlier, is an expected gaseous melter effluent, but Ba, Gd, and Zn and their various compounds have not, historically, exhibited semivolatile characteristics under LFCM processing conditions. If, on the other hand, aerosols were penetrating the upstream filter of the multicomponent sampling system, sodium would surely have produced a much stronger systematic trend across the acid baths, which it does not. However, apart from the nature and source of the Ba, Gd, and Zn in these scrub solutions, their contributions to cumulative mass totals are relatively small for most of the processing campaigns characterized. The fact that sulfur did not partition across the 0060 sampling-train components does not necessarily imply lack of volatility, as the sampler's mercury-specific chemical scrubbers are not particularly efficient for SO_2 capture. That was the reason for conducting independent Method 8 sampling campaigns, as was previously discussed.

As a final note, the volatility systematics apparent in Table 6.11 suggest a possible bias associated with the nonreductant feed-processing campaign. The sampling data indicate small but measurable volatile partitioning for just about all feed constituents. Although the processing conditions could conceivably be responsible for this phenomenon, it is doubtful that oxidizing conditions alone could produce universal changes in the nature of the melter source term. Since, in all cases, the indicated volatile fraction is small, effluent cumulative masses and resultant DFs are not significantly affected.

Table 6.13 summarizes total elemental melter DFs obtained by combining the scrub-solution composition with its corresponding particulate catch. Comparing these total values with corresponding particulate DFs previously discussed (Table 6.9) clearly illustrates the dominant role played by the aerosol-loss mechanism. Volatility contributions to the melter-effluent source term were only significant for mercury, the halogens, and boron; all other total DFs are nominally equivalent to their particulate values. These element-specific, total DFs, like their corresponding particulate values, are reasonably close to general expectations and are with few exceptions consistent with previous SBW melter-testing results. Based on previously discussed glass-compositional data, melter mercury DFs should be very close to 1. The fact that a DF of 6 is recorded during nonreductant feed processing is somewhat puzzling but may simply reflect the difficulty associated with mercury sampling and analysis.

Total DFs for the halogens could not be determined since analyses of pertinent particulate samples were not conducted. However estimates of total melter DFs can also be extracted from secondary waste-stream concentration data, provided that the volatiles are efficiently scrubbed and the effluent steams can be representatively sampled. These estimates will be discussed in the following Section.

Table 6.13. Total Individual Elemental Melter DF Values

	Total Effluent DFs: SBW-22 & Additives				
	Sugar		Glycolic	None	Overall
Element	**170 g/L**	**160 g/L**	**280 g/L**	**----**	**Ave***
Al	329	461	430	58	161
B	27	45	107	18	32
Ba	72	17	15	13	19
Ca	230	380	817	82	196
Cd	53	61	104	52	62
Cr	40	120	178	34	58
Cs	9	32	49	38	21
Cu	65	183	203	73	101
Fe	1,560	1,410	1,160	73	250
Gd	453	647	1,870	70	216
Hg	1	2	1	6	1
K	14	54	85	64	34
Li	46	120	306	66	82
Mg	12,000	652	1,390	74	251
Mn	307	376	1,460	35	115
Mo	29	130	172	97	68
Na	17	53	100	53	37
Ni	869	1,520	1,980	349	772
P	216	468	1,220	104	233
Pb	143	220	576	37	98
Ru	20	34	35	59	32
S	15	38	50	36	28
Ti	298,000	123,000	ND	3,930	15,000
V	622	909	1,080	258	533
Zn	68	121	142	30	63
Zr	7,840	42,700	22,600	187	723
*Based on partitioning values.					

6.3 RSM Secondary Waste Streams

For the RSM-2 test, a sampling scheme was developed to obtain as much data as practical about differences in the scrubbing effectiveness of the EVS and HEME when collecting species from the melter off-gas. These data were used to interpret the impact of flowsheet changes. These data were also compared with glass and other off-gas sampling data to obtain an overall picture as to what can be expected from changes in the operational parameters and how the chosen off-gas treatment schemes might be impacted.

To collect the data without impacting the cost and schedule by changing existing equipment, the following sampling and operational scheme was devised. The EVS scrub solution and the collected acid insoluble solids samples were obtained for each of the specified melter-run conditions. The melter feed was stopped to collect the entire volume of EVS scrub solution, and it was subsequently replaced with fresh scrub solution. One molar HNO_3 was chosen as the starting scrub solution for the EVS. The run

time, solution volume, and samples of the solution were taken at the beginning, during, and the end of each run. A batch of fresh scrub solution was used for each set of melter-run conditions (initially 6). While the melter feeding was stopped to change feeds, the scrub solution was drained into 50-L carboys. Using solution pumped from the first carboy, remaining solids were flushed into the last tared carboy. After weighing each carboy, triple 100-mL solution samples were taken from each one. The solids were allowed to settle in the poly carboys for at least 6 h, and the clear solution was decanted for disposal using a tubing pump. Solids were removed by slurrying them with the remaining liquid and pumping the solution into a liter bottle. Solids were allowed to settle for several hours before any more liquid was decanted from them. At the end of the campaign, the same EVS solution was used for multiple conditions due to time constraints. The overall plan for the operation was as follows:

- Evaluate the fate and speciation of mercury in the feed mixture for the different melter redox conditions and reductants.
- Determine the fate of the surrogate feed and other feed constituents, including the reductant, halides, nitrates, sulfur, metals that are hazardous or are surrogates for hazardous metals, and elements that are surrogates for radionuclides.
- Characterize wet-scrubber performance for scrubbing water, Hg species, halides, NO_x species, SO_2, and particulate matter, under the different melter operating conditions but at a single specified scrubber pressure drop.
- Combine the composition, properties, and amounts of all system effluent streams, including the product glass, scrub solution (including dissolved and undissolved solids), off-gas (including PM in the off-gas), and PM deposits recovered from the off-gas piping to establish a process mass balance.

6.3.1 Sampling Schedule and Scrub-Solution Characteristics

During the RSM-2 test, EVS and HEME secondary wastes were accumulated and collected during four separate melter-processing campaigns that sequentially utilized sugar, low and high concentrations of glycolic acid as feed reductants, and a feed that on the average contained no reductant. The corresponding secondary EVS and HEME wastes generated and collected during these processing campaigns are identified by a number reflecting the preceding sequence and shown in Table 6.14.

Table 6.14. Processing Campaigns Monitored by Secondary Waste Streams Studies

Seq#	Processing Campaign
1	SBW-22 Formulation with sugar reductant
2	SBW-22 Formulation with reference glycolic reductant concentrations
3	SBW-22 Formulation with high glycolic reductant concentrations
4	SBW-22 Formulation with no reductant*
* Includes 1.5 h of processing with sugar out of a 27-h total.	

6.3.1.1 Sugar Reductant Campaign (#1)

The first scrub-solution volume was accumulated for the entire operation of the sugar-reducing-conditions iteration. Initially, the solution had a slight tinge of red color that intensified until the solution

was opaque. When the solids settled, the solution was a light green color. In about 46 h, the scrub-solution volume increased from 58 L to just over 70 L. During the same period, the acid concentration decreased from 0.96 N to 0.52 N as the total dissolved solids (TDS) increased from 0.17 to 11.5 g/L. These results are graphically represented in Figure 6.4 and Figure 6.5.

The acid decreased and the TDS increased, mostly because of feed and glass material carryover. The HEME acid concentration and TDS increased from 0.02 N to 0.98 N and from nearly 0 to 110.0 g/L, respectively. For more detail, see Figure 6.4 and Figure 6.5. Sample-collection times for the manual trains (see Section 6.2) are shown as bars across the top of Figure 6.4. As noted in Section 6.3.3, these sampling times were when fewer solids were being transferred to the scrub system. The total HEME solution collected was 2590 g in the 46-h period.

The first scrub solution was collected prior to the initiation of processing with glycolic acid as the reductant. The solids were then allowed to settle, and the solution was sampled and decanted. The final solids slurry and mass balance samples were retained for detailed analysis. During the scrub-solution change, a hand-held mercury vapor analyzer sampled the vapor space above scrub solutions. Results indicated from 0.2 to 0.4 μg Hg/M^3 in the vapor space of the scrub solution samples and dumped solution, but very little mercury was detected in the vapor space of the recirculating scrub tank. Vapor above the contained HEME solutions indicated 0.5 to 0.8 μg Hg/M^3. These values may not be accurate, however, because of possible effects caused by the acid gases on the hand-held analyzer.

6.3.1.2 Glycolic Reductant Campaigns (#2 & #3)

Different scrub solutions were used for each glycolic acid condition. Because of the large change in the added reductant, it was reasoned that off-gas and emission characteristics would be greatly impacted. The scrub solutions remained mostly clear during the use of glycolic acid as the test reductant. The acid concentration decreased at a rate similar to the sugar-reducing run, but the scrub-solution volumes changed less than 0.5 L. As shown in Figure 6.4, the TDS increased from 0.38 to 0.67 in the second scrub and from 0.17 to 0.94 in the third (second glycolic). The acid concentration decreased from 0.98 N to 0.89 N in about 13 h in the first glycolic scrub and from 1.0 N to 0.92 N in the second glycolic scrub in about a 9 h-period (see Figure 6.5). A difference was noted in the glycolic HEME solutions from those of the reduced-sugar condition. The HEME solution acid increased to 1.47 N in the first glycolic solution and to 1.45 N in the second. The amount of HEME solution collected for the first and second glycolic acid batches was 573 g and 476 g, respectively. A small heel remained in the vessel bottom, about 35 mL, but there was an unknown amount of holdup on the media.

6.3.1.3 No Reductant Campaign (#4)

The last scrub solution was used throughout the remainder of the melter run time. A request was received for a changeout before sugar addition was resumed during the last section of the oxidizing condition test. However, not enough time was left in the allotted test window to complete all the shutdown and startup processes.

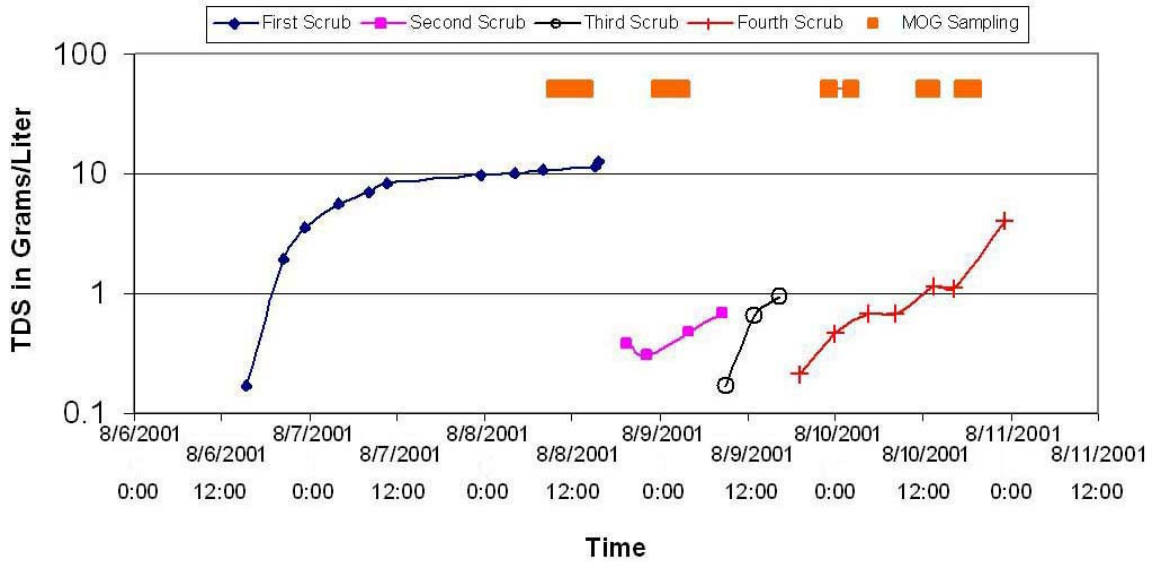

Figure 6.4. EVS Solution of Dissolved Solids Accumulation

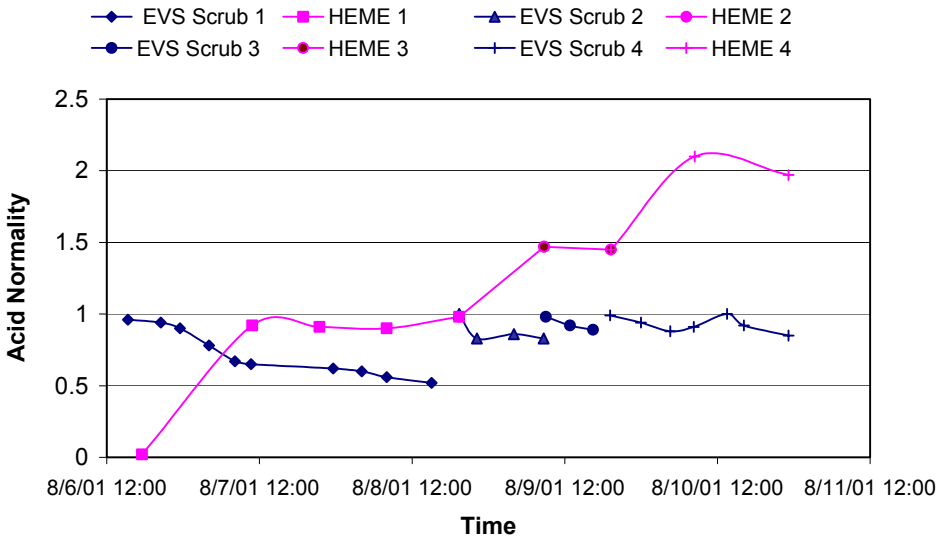

Figure 6.5. EVS and HEME Solution Acidity

The scrub remained very clean until the last 6 h of about 27 h of use. During this time frame, a portion of the solids that had accumulated on the off-gas piping earlier broke loose and was transported downstream to the scrubber. Scrub-solution acidity decreased more slowly compared to the earlier scrub solutions. An upward trend is present in the acid of the EVS solution because of one data point on the afternoon of 8/10/01 (see Figure 6.5). The reason for this is unknown, but it may be a bad analysis. The decanted solution was not as green and had a more yellow cast. The HEME solution also showed this color difference. As with the glycolic acid scrub solutions, the scrub-solution volume showed very little change under oxidizing conditions. The solution collected from the HEME weighed 1418 g (see Section 6.4). Twelve hours after the conclusion of testing, less than 50 mL drained from the HEME. As with the other scrub solutions, the solids were allowed to settle, and the liquid was sampled and decanted.

6.3.1.4 Campaign Summary

All the available solids for each scrub solution were collected in a slurry and split with a riffler into even fractions. One sample was shipped to INTEC for further characterization, and the other is stored at PNNL.

Throughout the test, feed rates were changed, and accumulations occurred in the off-gas lines. Plugs formed, and the lines were unplugged. These variable changes caused fluctuations in the collection rate in the EVS scrub solution. The rates of TDS pickup were smoothed and plotted in Figure 6.6. For most of the test, the rate was less than 0.2 g/h/L. The exceptions occurred during the first scrub, the reducing sugar run, and the final scrub. During the first sugar reductant run, the feed rate was higher than it was later; thus, more water and entrained feed were carried over to the EVS. The rate was above 0.4 g/h/L for more than 11 h. The rate reduced, mostly because of operational problems, and then increased. During the glycolic acid reductant runs, the rate stayed below 0.1 g/h/L. Less feed was entrained, but as will be mentioned later in Section 6.3.2, the volatile materials were released at about the same rate. During most of the final scrub, strong oxidizing conditions existed where either no reductant or a smaller amount of sugar was used. For most of the run, the pickup rate for TDS was well below 0.1 g/h/L but increased near the end to about 0.3 g/h/L. Most of this increase was a result of the off-gas line being cleared of a buildup of entrained feed. There was also a slight increase in the EVS scrub volume. The off-gas manual sampling train was not in operation during this clearing operation but was put into operation afterwards for the final testing.

6.3.2 Analytical Analysis of Scrub-Solution and Collected Solids

Although the melter off-gas processing system was installed to just “clean up” the melter off-gas so that it could be discharged, it functions quite well in removing most of the off-gas chemical constituents that challenge it. The EVS removes the bulk of the material, and the oversized HEME cleans up the entrained solution and solid constituents that escape. It is evident from the data presented above that the HEME concentrates the entrained EVS solution, allowing the water to evaporate into the exiting off-gas. The HEME was warmer to the touch than the surrounding piping, indicating some heat of solution.

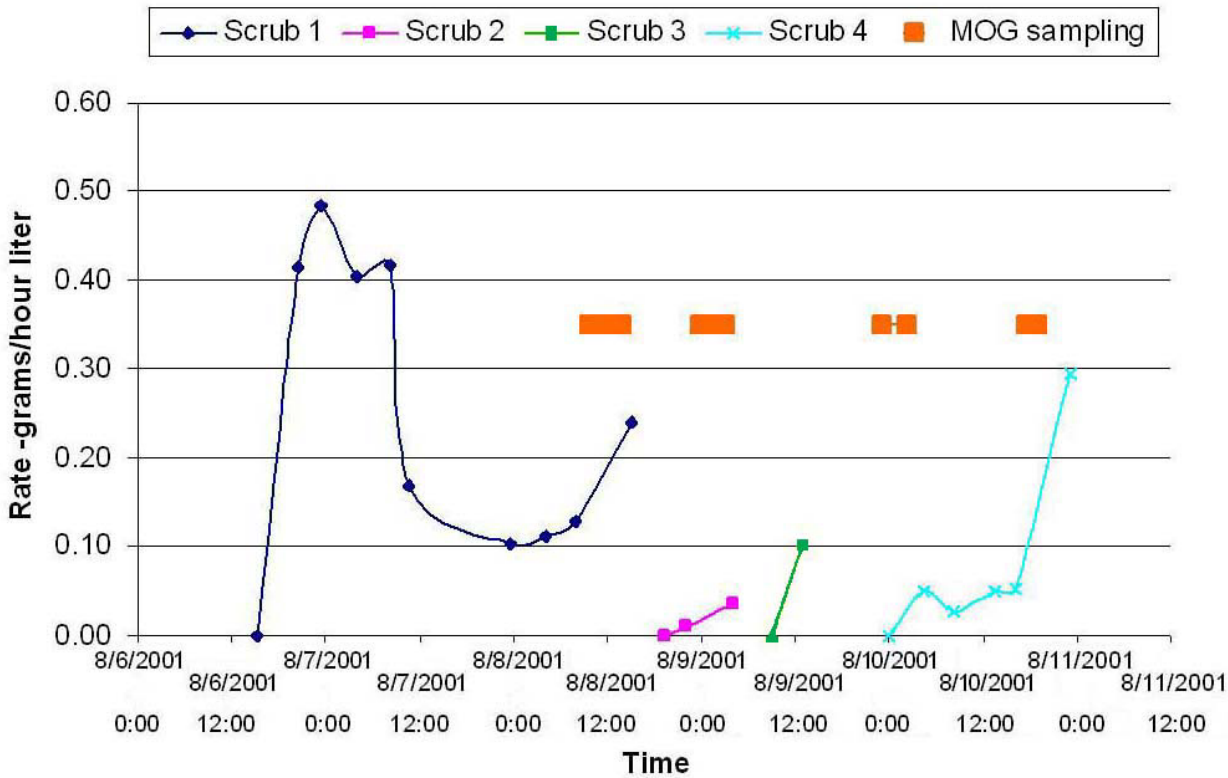

Figure 6.6. RSM-2 EVS TDS Accumulation Rate—g/h/L

In the following sections, scrubber analytical data are presented in both tabular and graphical form. The graphical form presentation is for an elemental accumulation rate in grams per liter of SBW waste fed to the melter and as a percent of element in the feed. Data are presented in the tabular form so specific values can be referenced. No melter decontamination factors are given but can be inferred from the data presented. It is not known how much of each constituent penetrated the scrub system. The properties of undissolved solids will be discussed in more detail in Section 6.3.3.

6.3.2.1 Soluble Secondary Waste Constituents

Table 6.15 summarizes the analyzed soluble species collected by the scrub solutions. The values for the HEME were taken from the total mass collected by the solution during the use of the upstream EVS scrubber. The error introduced to the total is not precisely known because of the solution holdup and differential holdup of the HEME packing. The data indicate a leveling off or an actual reduction of concentrations of the most analyzed constituents (Hg, Cs, Na, Acid). This indicates that the HEME runoff is removing most of the collected species with some delay caused by the holdup. The loss of nitrate in the EVS solution is mostly countered by its increase in the HEME solutions. In the final scrub, a large net increase in the sum of the nitrate is evident. Very little reductant was used in the feed during the collection of the final scrub solutions. A much greater NO_2 concentration in the off-gas was responsible for the observed increase. Most of the NO_3^- pickup was by the HEME, although the EVS also had a net gain. Note that the EVS had a net nitrate loss in the previous three scrub solutions.

For sulfur and phosphorous, total elemental compositions as well as SO_4^{-2} and PO_4^{-3} concentrations were determined. For the non-glycolic runs, scrub solutions #1 and #4, the molar concentration of the sulfate was a high fraction of the total sulfur, but for the glycolic runs, the sulfate was a smaller fraction of the total. In a 1-4 order, the fraction of the total sulfur analyzed as sulfate is 1–0.86, 2–0.51, 3–0.11, 4–0.81. This trend is one of several indications showing the impact of a strong reducing condition in the off-gas during the use of glycolic acid.

The percentage of soluble species collected by the scrub system and found in the HEME solutions is shown as Figure 6.7. Note that the primary collector may not have been the HEME. Some of the EVS solution is transferred to the HEME as aerosol or small droplets.

Most of the species had a higher percentage collected by the HEME during the glycolic acid runs. Several species had more than 50 percent found in the HEME solutions during these reducing conditions. Mercury was one of the species with over 50 percent. During the first and fourth scrub solution periods when glycolic acid was not in use, very few species had over 50% of the total collected in the HEME. Those species were Cs, K, and S during the last scrub solution period. Most of the soluble carbon was collected by the EVS. Percentages of inorganic carbon (iC) and organic carbon (oC) are relatively low compared with the other species collected in the HEME.

As shown in Figure 6.4 and Table 6.5, less dissolved species (in total) were collected during the glycolic acid runs.

6.3.2.2 Overall Secondary Waste Stream Composition

A graphical representation of the percentage of each element fed to the melter that was collected by the scrub system solutions is presented in Figure 6.8. The values also include the solids. The bars are arranged with the highest overall value first, followed by each of the four scrub solutions for each element. The HEME computations are also included.

As may be expected, the elements capable of producing more volatile species are present in a higher proportion. The halogens, which produce acid gases and trihalides from an acid feed, are consistently > 10 percent of the amount present in the feed. Fluorine, which is often tied up with zirconium or an alkaline earth, partitions at > 30 percent and during a glycolic acid run was indicated at slightly over 100 percent. This result may be due to the way feed was prepared. Perhaps the Zr and Ca did not have the same complexing that typically occurs with the fluoride in other melter feeds. In any event, most of the fluorine was released to the off-gas. Chlorine was detected in the KLM analysis of the glass, in some cases in greater proportion than indicated from the feed values. In the last scrub solutions total, chlorine was present at slightly over 90 percent of its theoretical maximum. With smaller concentrations of a substance, a greater percentage error is possible in the overall analysis. In any event, a large proportion of the feed chlorine was trapped by the scrub solutions.

Table 6.15. RSM-2 Scrub Solutions Soluble Elemental Constituents (Values in Grams Collected per Liter of SBW Fed)

	First Scrub Sugar Reducing Condition				Second Scrub Glycolic Acid Reducing Condition				Third Scrub Glycolic Acid Very Reducing				Fourth Scrub Sugar or No Reductant Oxidizing Condition			
	EVS	HEME	Total Collected	% Collected by HEME	EVS	HEME	Total Collected	% Collected by HEME	EVS	HEME	Total Collected	% Collected by HEME	EVS	HEME	Total Collected	% Collected by HEME
Ag	4.94E-05	7.22E-06	0.0001	12.16	0.00E+00	2.98E-05	2.98E-05	100.00	0.00E+00	4.39E-05	4.39E-05	100.00	0.00E+00	8.11E-06	8.11E-06	100.00
Al	0.1265	0.0130	0.1395	8.88	0.0020	0.0161	0.0181	88.87	0.0497	0.0206	0.0703	29.35	0.1400	0.0191	0.1591	11.98
B	0.3207	0.1109	0.4316	24.48	0.1375	0.1299	0.2674	48.58	0.2666	0.1666	0.4332	38.47	0.3942	0.1626	0.5568	29.20
Ba	0.0004	0.0002	0.0006	26.84	0.0000	0.0003	0.0003	95.91	0.0001	0.0001	0.0002	49.31	0.0002	0.0004	0.0005	69.99
Ca	0.2589	0.0538	0.3127	16.40	-0.0206	0.0649	0.0443	146.46	0.0479	0.0825	0.1304	63.26	0.2236	0.0772	0.3008	25.67
Cd	0.0036	0.0027	0.0063	40.82	0.0002	0.0027	0.0029	92.31	0.0038	0.0035	0.0073	48.11	0.0014	0.0038	0.0053	72.76
Cl^-	0.1657	0.0414	0.2070	19.03	0.0244	0.0439	0.0682	64.27	0.0000	0.0562	0.0562	100.00	0.4339	0.0531	0.4870	10.90
Cr	0.0074	0.0041	0.0115	33.77	0.0013	0.0042	0.0055	76.29	0.0029	0.0054	0.0083	65.25	0.0053	0.0053	0.0106	49.87
Cs	0.0182	0.0111	0.0293	35.96	0.0043	0.0125	0.0168	74.16	0.0099	0.0159	0.0258	61.81	0.0098	0.0155	0.0253	61.32
Cu	0.0037	0.0012	0.0048	23.29	0.0052	0.0016	0.0067	23.09	0.0032	0.0020	0.0052	38.02	0.0124	0.0020	0.0144	14.07
F	0.0949	0.0219	0.1168	17.88	0.0701	0.0283	0.0984	28.77	0.2059	0.0363	0.2422	14.99	0.0818	0.0357	0.1176	30.39
Fe	0.0343	0.0035	0.0379	8.88	-0.0033	0.0034	0.0001	NA	0.0173	0.0044	0.0217	20.23	0.0257	0.0037	0.0294	12.67
Gd	2.71E-04	3.33E-05	0.0003	10.43	0.0000	0.0001	0.0001	67.24	0.0001	0.0001	0.0002	55.41	0.0003	0.0000	0.0003	14.58
Hg	0.0654	0.0281	0.0935	28.67	0.0296	0.0355	0.0651	54.51	0.0321	0.0455	0.0776	58.67	0.1388	0.0473	0.1862	25.43
I	0.0016	0.0003	0.0018	13.29	0.0044	0.0003	0.0047	6.98	0.0045	0.0008	0.0053	14.61	0.0029	0.0004	0.0033	11.55
K	0.4187	0.1812	0.5999	28.77	0.0728	0.1992	0.2720	73.24	0.1733	0.2556	0.4289	59.60	0.2060	0.2534	0.4594	55.16
Li	0.3723	0.1053	0.4776	21.00	0.0291	0.1206	0.1496	80.58	0.1355	0.1547	0.2902	53.30	0.2474	0.1463	0.3936	37.16
Mg	0.0918	0.0146	0.1064	13.11	0.0018	0.0162	0.0179	90.20	0.0297	0.0208	0.0505	41.16	0.0780	0.0196	0.0976	20.04
Mn	0.0046	0.0006	0.0052	11.06	0.0001	0.0010	0.0012	87.36	0.0020	0.0013	0.0033	39.84	0.0081	0.0009	0.0090	9.99
Mo	0.0009	0.0007	0.0015	41.48	0.0002	0.0007	0.0009	81.82	0.0004	0.0010	0.0013	70.79	0.0004	0.0007	0.0011	62.29
Na	2.5801	0.8909	3.4710	24.45	0.4408	1.0162	1.4571	69.75	1.0835	1.3038	2.3873	54.61	1.4460	1.2515	2.6976	46.40
Ni	0.0077	0.0015	0.0092	15.47	0.0123	0.0019	0.0141	13.12	0.0081	0.0024	0.0105	22.78	0.0285	0.0010	0.0296	3.45
NO_2^-	-0.122	0.0013	-0.1203	NA	-0.3580	0.0000	-0.3580	NA	-0.4078	0.0000	-0.4078	NA	-0.1907	0.0044	-0.1864	NA
NO_3^-	-8.211	7.3576	-0.8534	NA	-11.3552	8.5720	-2.7833	NA	-9.7326	10.9968	1.2642	NA	0.5834	10.9931	11.5765	94.96
P	0.0085	0.0019	0.0105	17.55	0.0007	0.0026	0.0033	79.55	0.0035	0.0034	0.0069	49.02	0.0065	0.0013	0.0078	16.65

Table 6.15 (Contd)

	First Scrub Sugar Reducing Condition				Second Scrub Glycolic Acid Reducing Condition				Third Scrub Glycolic Acid Very Reducing				Fourth Scrub Sugar or No Reductant Oxidizing Condition			
	EVS	HEME	Total Collected	% Collected by HEME	EVS	HEME	Total Collected	% Collected by HEME	EVS	HEME	Total Collected	% Collected by HEME	EVS	HEME	Total Collected	% Collected by HEME
Pb	0.0039	0.0028	0.0066	39.82	0.0002	0.0028	0.0030	93.20	0.0028	0.0037	0.0064	56.88	0.0034	0.0029	0.0062	46.28
PO_4	0.0411	0.0047	0.0458	9.78	0.0000	0.0064	0.0064	100.00	0.0000	0.0082	0.0082	100.00	0.0000	0.0054	0.0054	100.00
Ru	0.0011	0.0001	0.0012	8.42	0.0009	0.0002	0.0011	18.04	0.0011	0.0006	0.0016	34.03	0.0040	0.0002	0.0043	5.15
S	0.1527	0.0806	0.2334	32.91	0.0217	0.0905	0.1122	80.66	0.0842	0.1162	0.2003	57.99	0.0747	0.1102	0.1850	59.61
Si	0.2589	0.0025	0.2614	0.91	-0.0311	0.0031	-0.0280	NA	0.0998	0.0040	0.1037	3.81	0.1421	0.0029	0.1451	2.02
SO_4	0.4307	0.1762	0.6070	27.66	0.0393	0.1310	0.1703	76.91	-0.0987	0.1681	0.0694	NA	0.2431	0.2453	0.4884	50.22
Ti	0.0003	0.0000	0.0004	7.66	0.0000	0.0000	0.0001	52.75	0.0001	0.0000	0.0002	24.40	0.0003	0.0000	0.0003	10.30
TIC	0.0298	0.0009	0.0306	2.73	0.1512	0.0030	0.1541	1.94	0.1031	0.0038	0.1069	3.56	0.0176	0.0034	0.0210	16.05
TOC	1.1862	0.0517	1.2379	3.98	2.4711	0.0569	2.5280	2.25	2.9422	0.0730	3.0152	2.42	0.4492	0.0772	0.5264	14.67
V	0.1649	0.0248	0.1897	12.47	0.0019	0.0307	0.0326	94.31	0.0452	0.0391	0.0842	46.38	0.0408	0.0382	0.0790	48.32
Zn	0.0012	0.0005	0.0017	27.72	0.0003	0.0006	0.0009	66.48	0.0006	0.0002	0.0008	29.43	0.0011	0.0004	0.0015	27.64
Zr	0.0015	0.0001	0.0016	4.97	0.0002	0.0001	0.0003	39.40	0.0016	0.0002	0.0018	8.92	-0.0001	0.0001	0.0000	NA

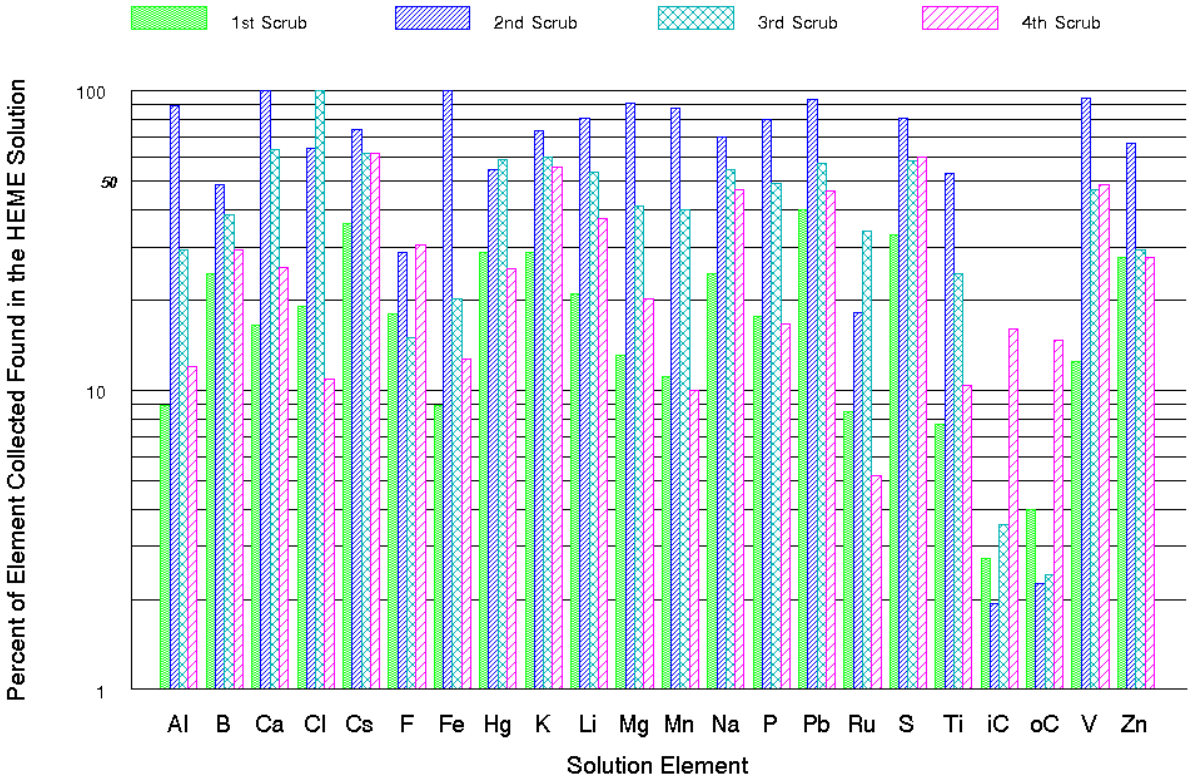

Figure 6.7. Percentage of Elements Scrubbed Found In the HEME Solutions

Most, if not all, of the Hg is expected to be released to the off-gas, but the proportion of oxidized Hg is not known, and the fate of much of the Hg in this test is still not fully understood. The total amount of Hg collected in the scrub was only slightly over 27 percent of that present in the feed (much of the unaccounted for mercury in the off-gas system has subsequently been discovered and quantified as discussed in Section 6.3.4). The data collected by the manual sample trains, and the continuous monitor results indicated that most of the Hg was released to the off-gas. Less Hg as a percentage of feed was collected by the scrub solutions during the glycolic acid runs and a greater fraction was collected by the HEME.

For the balance of the elements, 4 percent of these feed constituents are collected in the scrub solutions. The highest of the alkali metals is as expected: Cs followed by K, Na, and Li. Lithium is under 1% of the feed. This pattern is in the order of the relative volatility of their compounds, especially the meta-borates and halides. The elements collected below 0.1% of the feed, such as Ti and Zr, are not included in Figure 6.8.

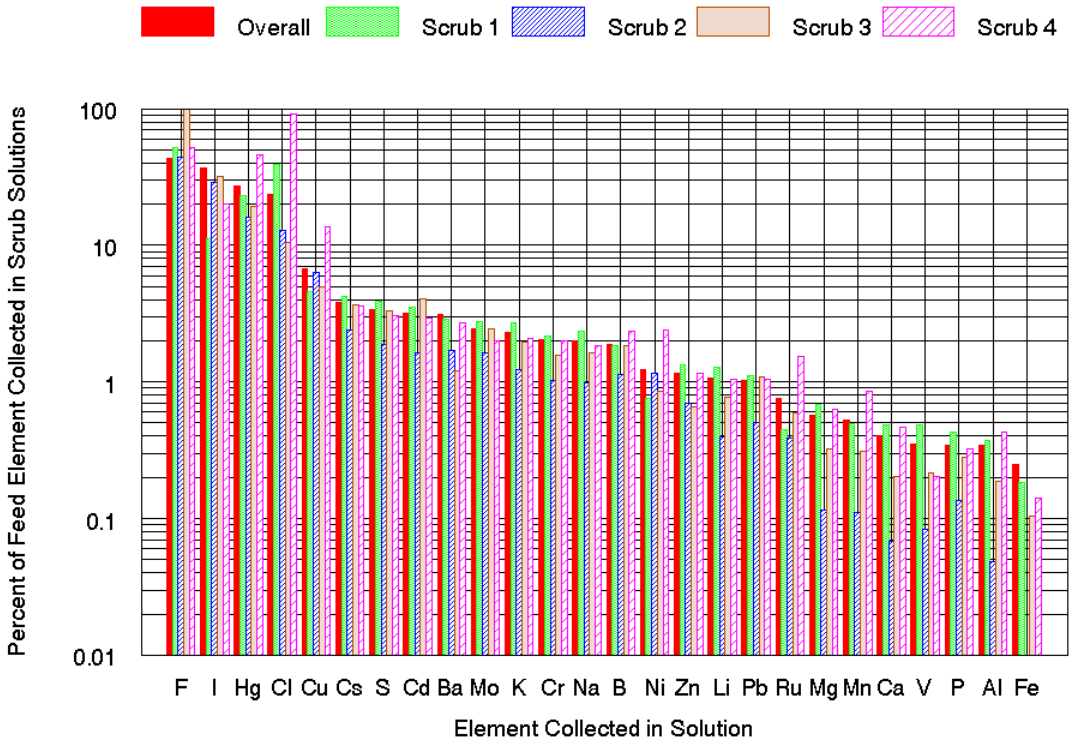

Figure 6.8. Proportion of Feed Elements Collected In Scrub Solutions

Figure 6.9 and Figure 6.10 show in descending order the total scrub system elemental collection rate per liter of SBW fed to the melter. Figure 6.9 contains the higher rates and Figure 6.10 the lower ones. The first bar is for the overall rate, used for sorting, followed in turn by each of the four scrub periods. The graphs allow for sight comparisons of the collection rates, based on a value directly related to the waste being treated.

Sodium appears as the most prevalent constituent, followed by oC. Because Na is one of the primary glass formers as well as the major constituent of the SBW, this result was expected. Most of the sodium losses could have come from the entrainment of feed material. The organic carbon is from the reductant. Note that both oC and iC increased during the glycolic acid runs (Scrub 2 and 3) and were at their smallest during the last scrub period when little or no reductant was present. This also is reasonable. Less sulfate was collected during the glycolic acid runs, although the total sulfur changed little, indicating the reductive nature of the off-gas. Mercury seemed to follow the same pattern as the sulfate. In the final scrub solutions, the Hg was at its highest collection rate of 0.186 g/L-SBW. At this rate, about half of the Hg processed would be collected by the scrub solutions. About 27 percent of the Hg feed was collected. The elemental Hg would not be expected to be collected in the scrub solutions without first being oxidized. As noted earlier, a large source of unaccounted for mercury in the off-gas system was subsequently characterized as discussed in Section 6.3.4.

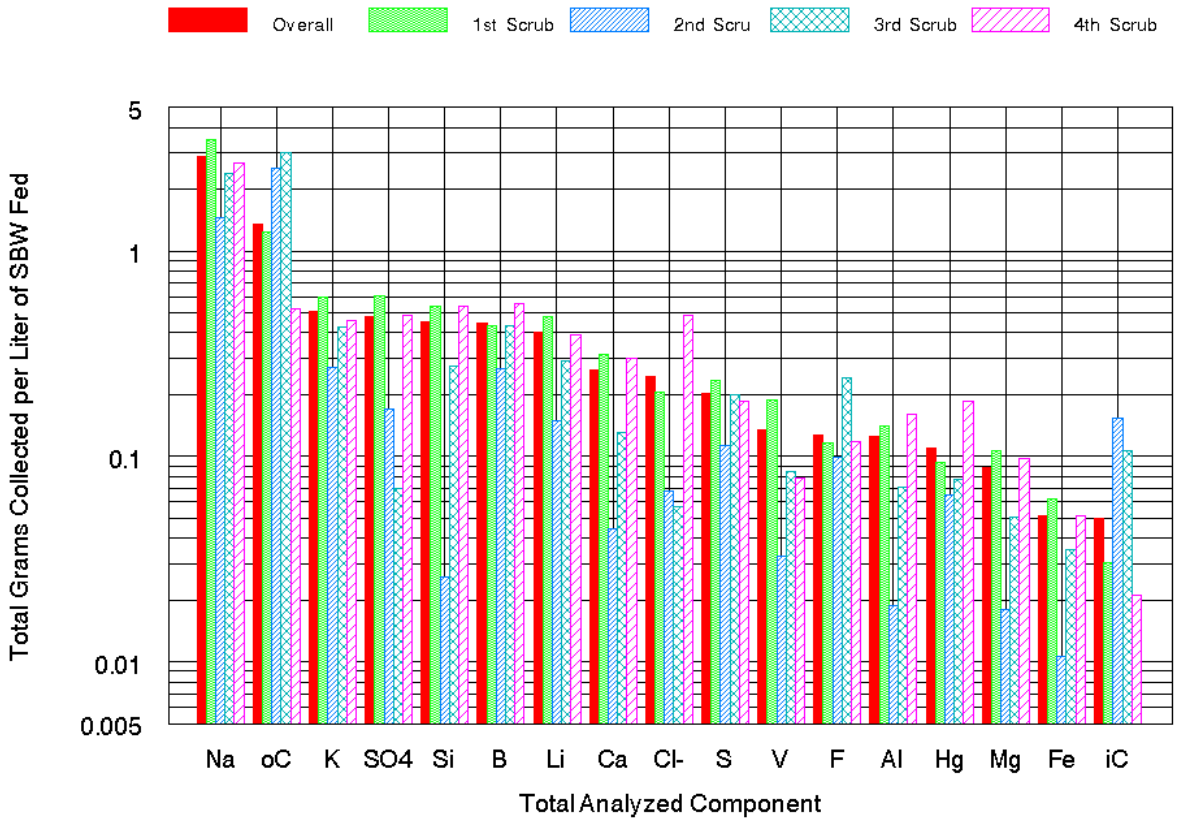

Figure 6.9. Total Scrub Solutions Elemental Accumulation (Higher Values)

Figure 6.10 shows the lower values of the accumulation of the scrub solutions. Cesium had a near constant collection for all four scrub solutions. Many of the other elements shown in Figure 6.10 show an increase in accumulation during the duration of the last scrub solution.

6.3.3 Undissolved Solids: Properties, Composition, Physical Characteristics

Table 6.16 summarizes the overall information on the settled solids collected by the EVS. As seen in the table, the collection rate was much larger when glycolic acid was not in use, and the bulk density of the solids also was greater. This change could be attributed to the nature of the cold cap. The weight loss of the glycolic scrub solids at 200°C was 4 to 5 times the amount lost by the non-glycolic acid scrub solids. This result could be due to more volatile collected organics. The solids from Scrub 2 have the lowest collection rate and the greatest weight loss when heated to 700°C. The amount of solids was very low, and a slight weighing error on the small amount of sample used could have caused the higher percentage loss. It is also possible that more organic was present in the sample aliquot.

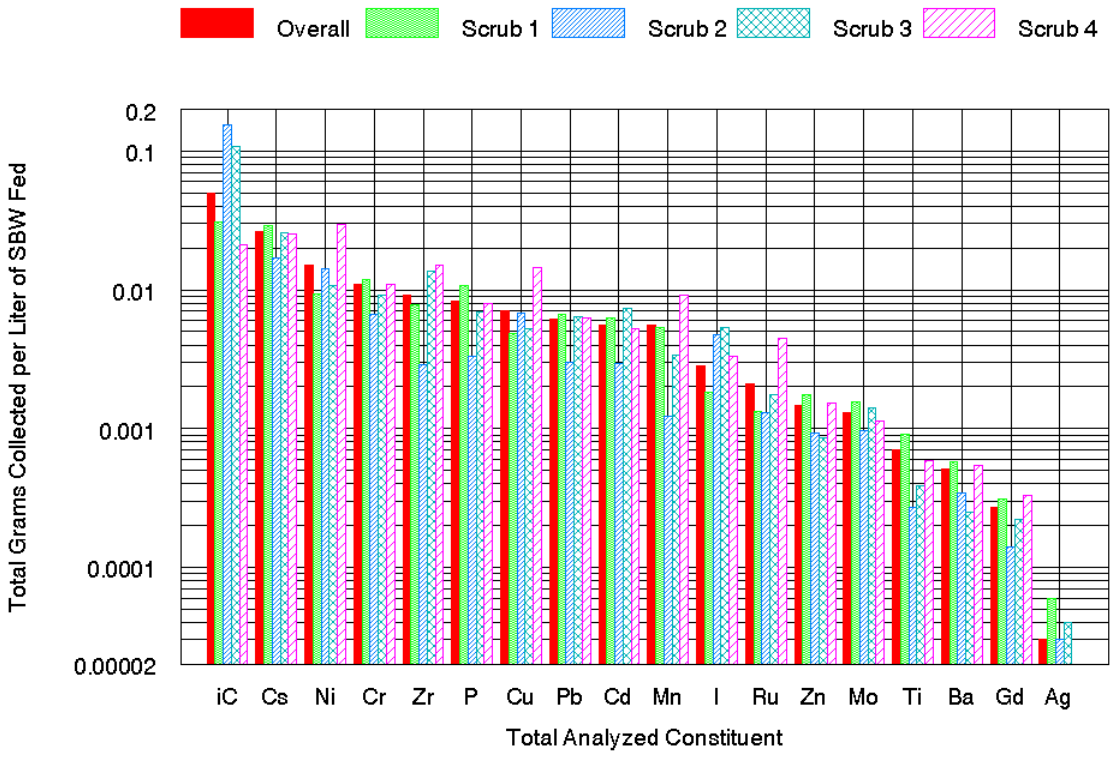

Figure 6.10. Total Scrub Solutions Elemental Accumulation (Lower Values)

Table 6.16. Settled Solids Information

EVS Scrub #	Calc. Total Mass Collected Grams	Dry Density g/cc	% Weight Loss @ 200°C	% Weight Loss @ 700°C	Collection Rate g/L SBW Fed
1-Sugar high	35	0.74	0.57	4.1	0.77
2-Glycolic acid	1.77	0.65	2.2	11.4	0.17
3-Glycolic acid	3.17	0.66	1.8	3.3	0.46
4-None & sugar low	20.6	0.83	0.31	2.3	0.60

6.3.3.1 Chemical Properties

The chemical composition of the settled solids is shown as Table 6.17. These solids were washed three times with demineralized water, dried at 60°C for 12 h, and then submitted for a complete chemical analysis. Not enough sample was obtained to run total organic or total inorganic carbon. The analysis indicated that most of the solids are Si and Fe. A calculation using the overall accumulation rate, assuming that the Si is SiO_2 and the Fe is Fe_2O_3, about 80 wt percent of the solids, can be accounted for with just these two elements. An x-ray diffraction indicates SiO_2 (quartz) and Fe_2O_3 (hematite) are the primary species. An inorganic carbon is also indicated. Carbon, $(VO)_2P_2O_5$, and $Ba_2CdMnFe_2F_{14}$ are

possibly present, as is amorphous material. All four scrub settled solids show the same result. Although Zr is the third most abundant element, and aluminum the fourth in the wet analysis, no ZrO_2 or Al_2O_3 was shown on the XRD analysis. These compounds possibly could have been in the amorphous form.

The source of the silica and hematite is mainly entrained feed. Some of the solids could have come from glass aerosols from melt or partially melted feed. Note that Cl and B collection increased with respect to other elements during the second glycolic acid run. These elements are usually soluble as are several others (such as Cs), all of which also increased during the first glycolic acid run as shown in Figure 6.11. Sodium and K also appear in the solids throughout all four scrub collections. The explanation may be that normally soluble elements are tied up inside glass aerosol particles.

Although the data in Table 6.17 represent the composition of the bulk material collected, a small superfine fraction had to be analyzed separately. As mentioned previously, the settled solids were washed with demineralized water to remove residue scrub solution in preparation for drying and chemical analysis. Washing was done using a centrifuge to enhance settling, so the rinse water could be separated. On the second iteration, solid fines could no longer be separated because a fraction remained suspended. Allowing the solids to settle for a week followed by centrifugation at 4200 revolutions per minute (RPM) produced very little separation. A 0.2-micron filter was then tried on the suspended fractions. This process was successful, but it took more than 12 h using over 20 in. Hg of vacuum. The initial filtrate had to be poured back through the filter after a cake had built up. This material after drying was found to be less than 3 percent of the total solids for the first scrub and less than 1 percent of the other scrub settled solids. This information shows that extremely small solids can adhere to the larger agglomerations under certain chemistry, but when that changes, they may be released. Rinsing out the scrub solution created an unexpected problem. Not enough of the light solids were present for a complete wet analysis on their contents. A scanning electron microscope (SEM) semi-quantitative analysis is shown as Table 6.18. Comparing this tabular data to the values of Figure 6.11 and Figure 6.12 shows that the fine solids are enriched in the other elements over Fe and Si. For many of the elements, the ratios with Fe are about two orders of magnitude higher for the fine material than for the bulk of the solids.

The dried fines look like dried mud cake. Under the microscope, the saltcake surfaces have a metallic sheen. No definite evidence of individual particles is seen at 500×. They are very small, less than 0.5 microns.

6.3.3.2 Physical Characteristics

A picture of the washed settled solids from the last scrub is included as Figure 6.13. This picture was taken at a magnification of 500× using both reflected and transmitted light. The picture shows an agglomeration of smaller particles of at least two different colors. The clear color can be assumed to be mostly quartz, and the red is mostly hematite. This agglomeration can be simply separated by allowing the mass to sit in water, then agitating the mixture. The solids will re-agglomerate and re-settle.

An attempt was made to obtain particle size analysis on these solids, but the resulting slurry water mixture was too opaque for the instrument to function properly, even with a very small amount of sample. Analyzing the solids dry was impossible because of agglomeration. The smallest translucent spherical particles in the picture are from 1.5 to 2.5 microns in diameter. Many of the red particles are even smaller.

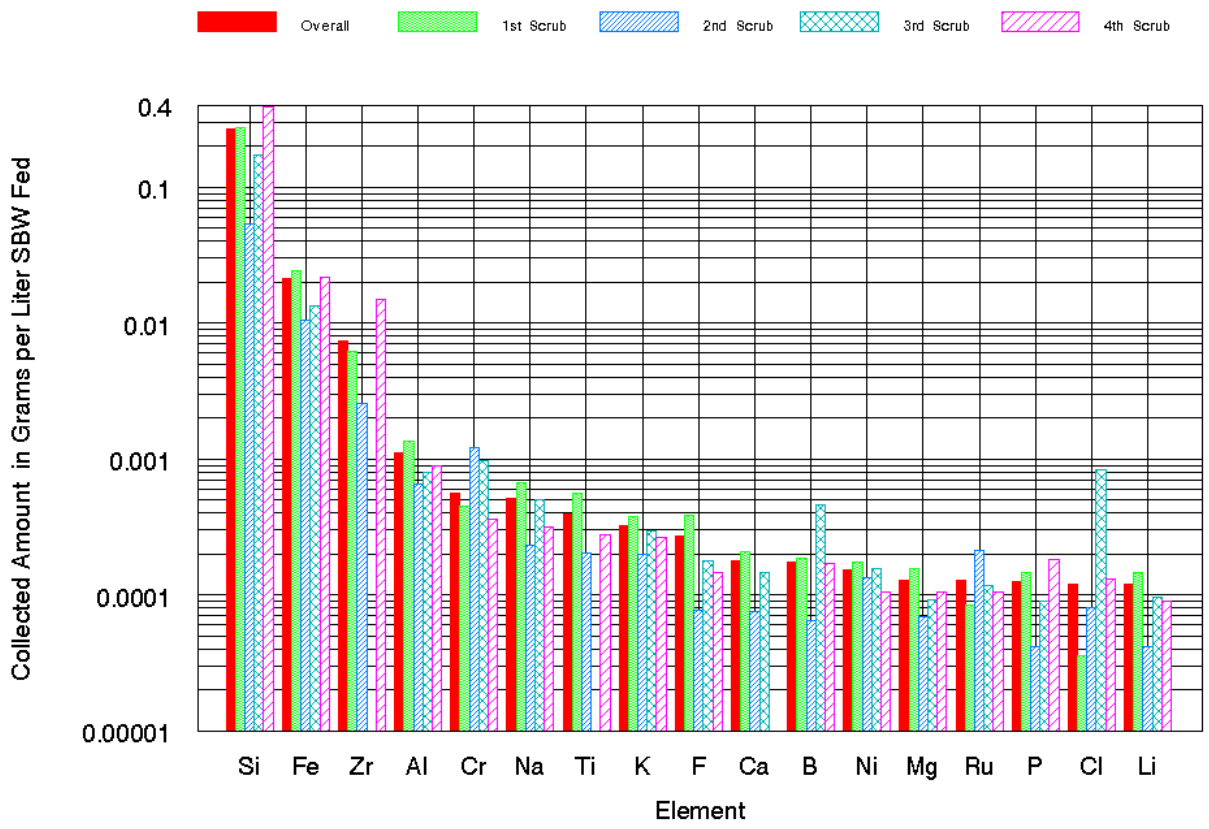

Figure 6.11. Comparison of EVS Settled Solids Elemental Composition (Higher Values)

Table 6.17. EVS Settled Solids Elemental Composition (Values in Grams per Liter SBW Fed)

	First Scrub	Second Scrub	Third Scrub	Fourth Scrub	Overall rate
Ag	ND	3.48E-06	ND	ND	4.45E-07
Al	1.30E-03	6.28E-04	7.60E-04	8.59E-04	1.11E-03
B	1.76E-04	6.14E-05	4.40E-04	9.98E-05	1.73E-04
Ba	1.43E-05	6.51E-06	7.81E-06	3.01E-06	1.04E-05
Ca	1.99E-04	7.19E-05	1.38E-04	1.63E-04	1.77E-04
Cd	1.79E-05	7.38E-06	2.54E-05	1.24E-05	1.66E-05
Cl-	3.37E-05	7.68E-05	8.00E-04	8.54E-05	1.20E-04
Cr	4.34E-04	1.17E-03	9.34E-04	3.44E-04	5.67E-04
Cs	ND	1.37E-05	ND	ND	1.75E-06
Cu	3.54E-05	3.50E-05	2.61E-05	2.29E-05	3.30E-05
F	3.66E-04	7.29E-05	1.69E-04	1.39E-04	2.69E-04
Fe	2.34E-02	1.01E-02	1.28E-02	2.08E-02	2.12E-02
Gd	7.74E-06	2.35E-06	5.14E-06	0.00E+00	5.15E-06
Hg	1.90E-05	3.48E-05	2.80E-05	7.16E-05	3.66E-05
I	ND	ND	ND	ND	ND
K	3.57E-04	1.89E-04	2.82E-04	2.62E-04	3.21E-04
Li	1.39E-04	4.01E-05	9.21E-05	1.05E-04	1.20E-04
Mg	1.50E-04	6.56E-05	8.52E-05	1.01E-04	1.28E-04

Table 6.17 (Contd)

	First Scrub	Second Scrub	Third Scrub	Fourth Scrub	Overall rate
Mn	7.14E-05	4.80E-05	6.14E-05	5.37E-05	6.63E-05
Mo	3.96E-05	6.31E-05	5.54E-05	1.13E-05	3.84E-05
Na	6.36E-04	2.24E-04	4.79E-04	3.03E-04	5.12E-04
Ni	1.68E-04	1.27E-04	1.49E-04	9.96E-05	1.51E-04
NO_2^-	ND	ND	ND	ND	ND
NO_3^-	ND	ND	ND	ND	ND
P	1.40E-04	3.94E-05	8.75E-05	1.25E-04	1.25E-04
Pb	0.00E+00	2.55E-06	0.00E+00	0.00E+00	3.26E-07
PO_4	ND	ND	ND	ND	ND
Ru	8.00E-05	2.02E-04	1.11E-04	1.75E-04	1.27E-04
S	8.48E-05	5.51E-05	7.20E-05	1.08E-04	9.01E-05
Si	2.63E-01	5.16E-02	1.63E-01	3.74E-01	2.69E-01
SO_4	2.63E-04	1.34E-04	3.19E-04	0.00E+00	1.95E-04
Ti	5.34E-04	1.93E-04	ND	2.54E-04	4.06E-04
V	1.04E-04	1.86E-05	ND	2.74E-05	7.03E-05
Zn	2.38E-05	1.10E-05	ND	1.05E-05	1.82E-05
Zr	5.86E-03	2.43E-03	ND	1.44E-02	8.04E-03

Table 6.18. SEM Semi-Quantitative Results of First Scrub Very Fine Suspended Settled Solids

Element	Wt. Percent	At. Percent
O	45.7	65.4
Si	21.4	17.4
Fe	19.3	7.91
Al	3.66	3.1
Mg	1.7	1.6
Cl	2.32	1.5
S	1.68	1.2
P	0.87	0.64
Zr	1.86	0.47
Ca	0.63	0.36
V	0.56	0.25
Ti	0.34	0.16

Although the hematite does not occupy much of the volume, it tends to cast the color on the entire mass of particles.

Another photograph of the settled solids is presented as Figure 6.14. The magnification is at 1000×, and the agglomeration was from the second glycolic acid run EVS scrub (third EVS Scrub). Very small red particles are visible, clinging to the larger, clear, bright ones. Comparison of the two pictures shows that the red color is less represented. The smaller red particles are about 0.4 microns. The larger black mass is possibly a piece of hematite (note the red color at the edges).

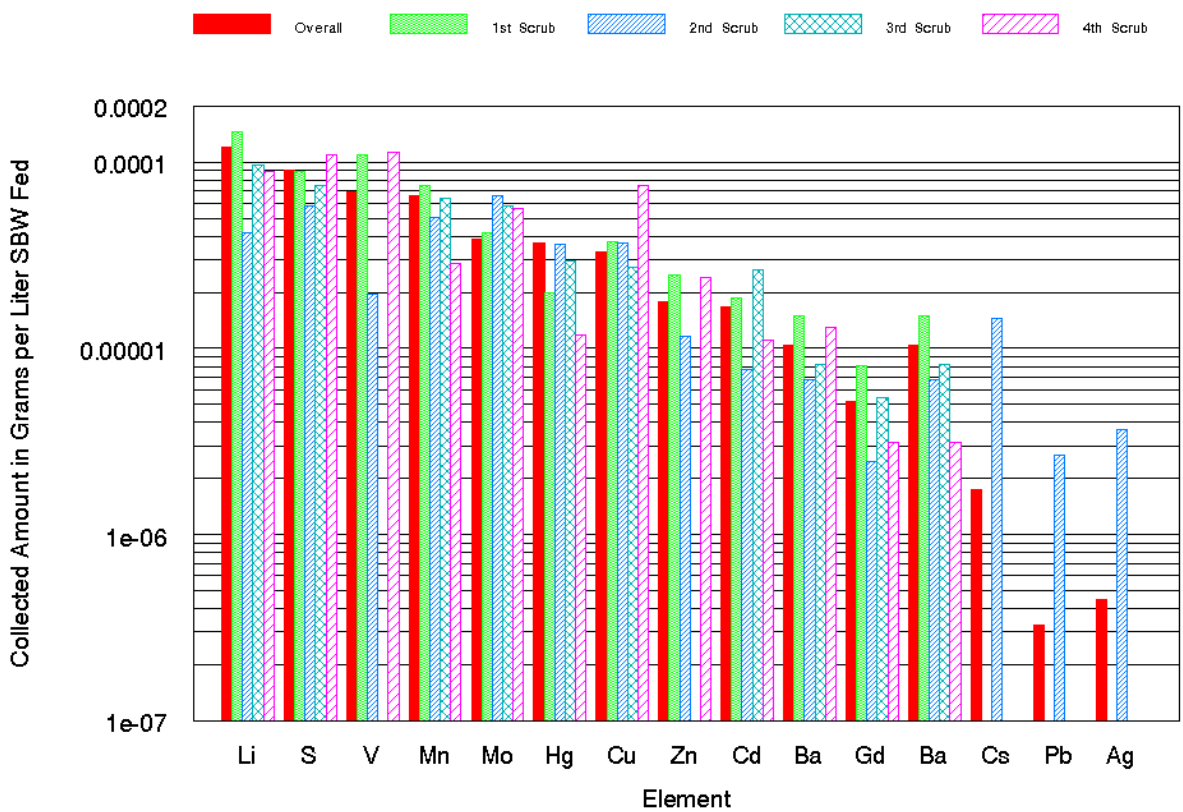

Figure 6.12. Comparison of EVS Settled Solids Elemental Composition (Lower Values)

6.3.4 Melter Feed Constituent DFs

The melter-feed constituent partitioning fractions derived from secondary waste-stream compositions discussed above have been assembled and presented in the form of melter DFs (inverse partitioning factor × 100) in Table 6.19. Included in this tabular data are overall DFs derived from cumulative process data. When compared with the discrete sampling data discussed in the previous section, the process waste-stream values are somewhat lower on the average. Recognizing that the Table 6.19 values represent a long-term average behavior while sampling results are short-term snapshots of melter behavior, the only way perfect agreement could be achieved is if the process had invariant performance characteristics. However, fairly good agreement is recorded for those effluents whose source terms are dominated by fuming or volatile escape mechanisms that are less impacted by campaign-dependent physical processes in the melter, e.g., B, Cs, K, Na, S. Indeed, the agreement between secondary waste stream and off-gas sampling results for sulfur strongly suggests that SO_2 off-gas partitioning is not a significant effluent loss mechanism, as inefficient collection of this effluent by the melter's off-gas effluent scrubbing equipment would otherwise artificially raise waste stream-derived sulfur DFs.

With the exception of iodine, the average DFs recorded for the halogens are found to be similar to the results obtained by the manual sampling trains (see Section 6.2). This agreement supports the earlier assertion (Section 6.2.4) that volatility dominates the melter source term as the waste stream-derived values represent total DFs while the manual trains only measure volatile DFs for the halogens. It should

also be noted that the abnormally high volatile fluorine DF measured with the manual sampling train under oxidizing processing conditions is not at all apparent in the secondary waste stream data. The current result is consistent with expectations based on chemical systematics.

It should also be noted that the relatively high mercury DF recorded in Table 6.19 is due to the fact that the HEME inventory of mercury could not be extracted during RSM testing. However, subsequent leaching of the HEME filter media increased secondary waste-stream mercury recovery from 27% to 73% of the quantity processed (see Section 7.4). By taking account of this additional source of mercury in the secondary waste stream inventory, the overall measured mercury DF is reduced from 4 to 1.4, which is much more in line with sample-train-derived values and with general melter processing expectations.

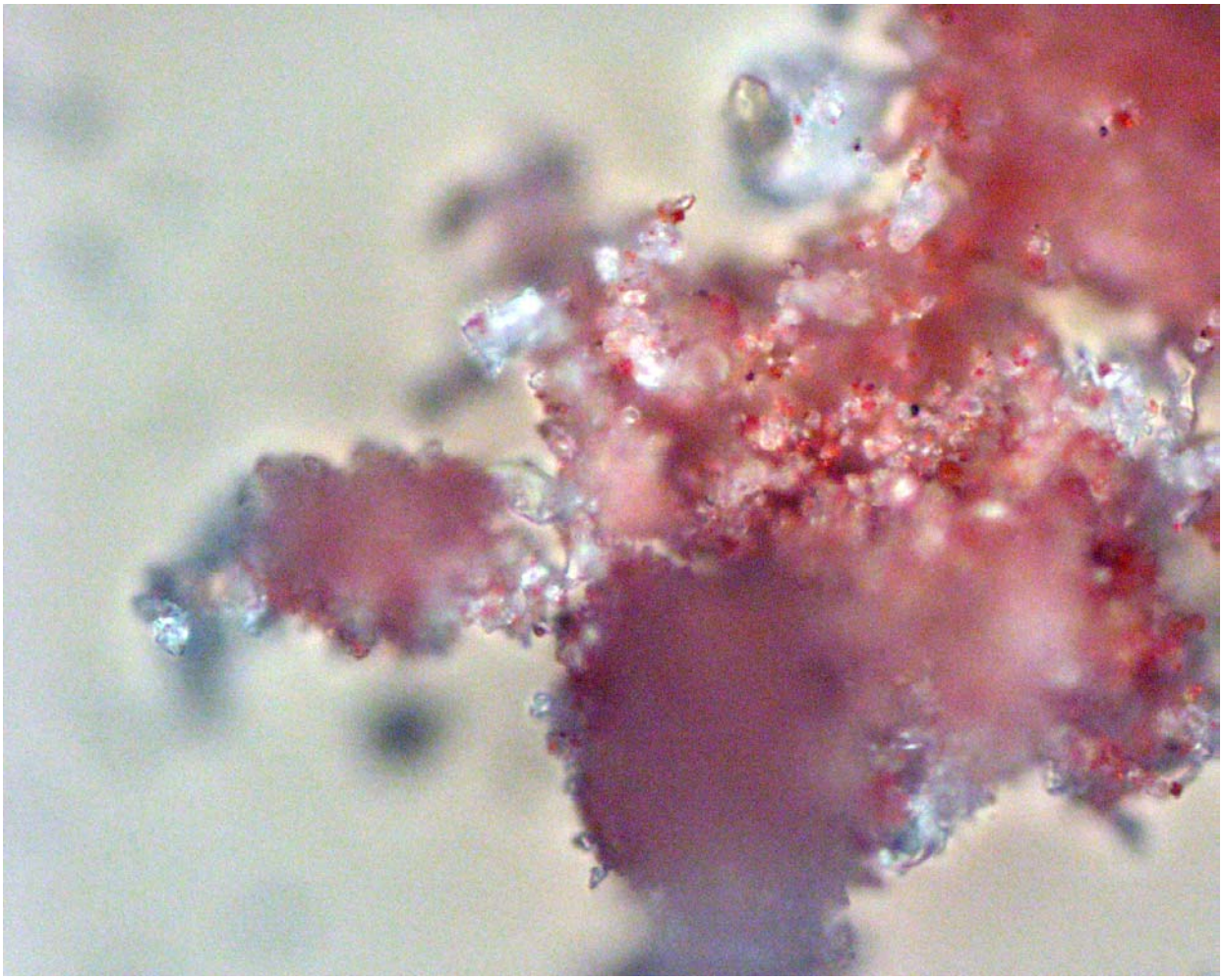

Figure 6.13. Settled Solids from Fourth EVS Solution @500×

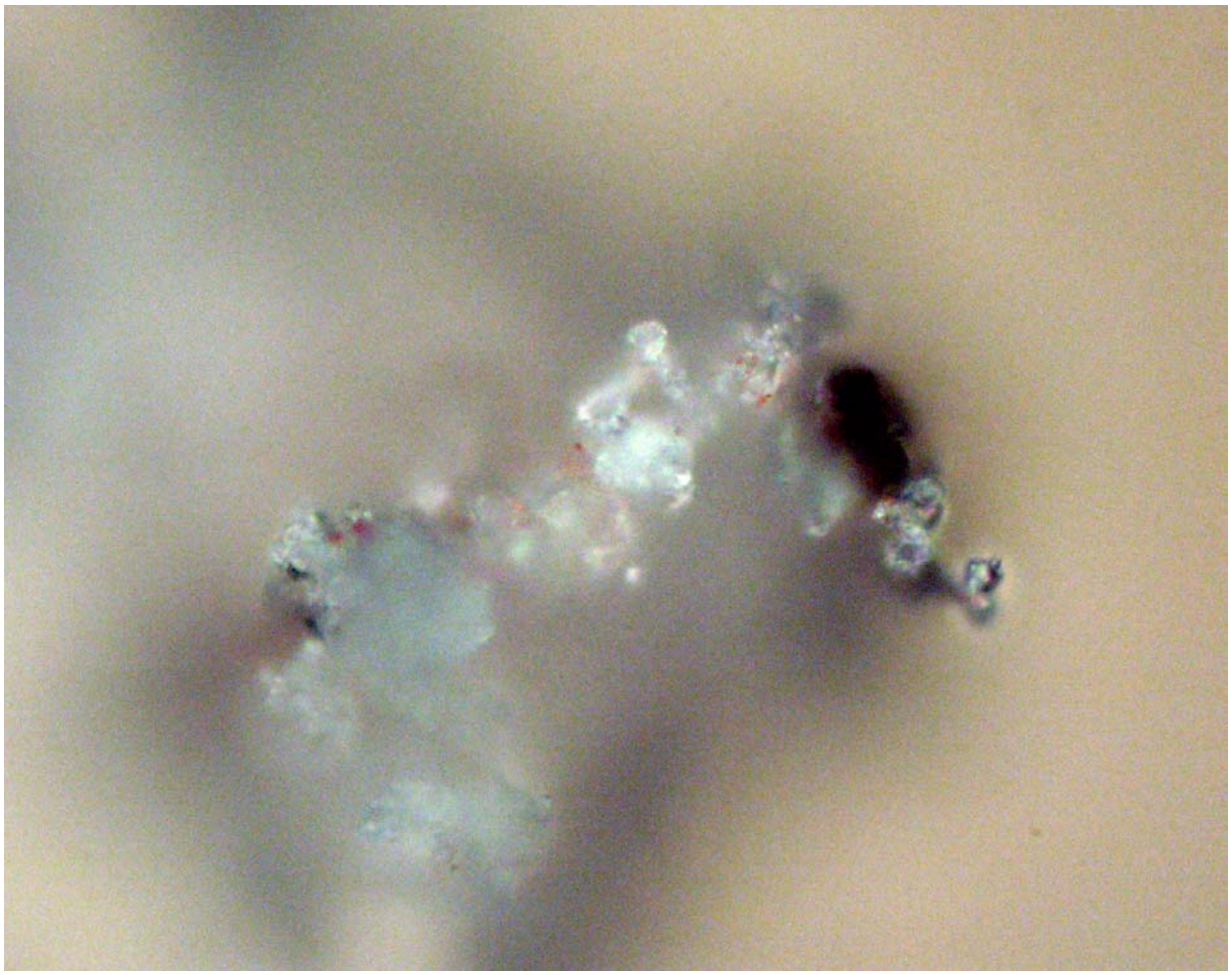

Figure 6.14. Settled Solids from 3rd EVS Scrub @ 1000×

Beyond the similarities of feed-component DFs, but unlike the sampling data that recorded unsystematic melter performance characteristics for the non-reduced feed condition, the waste-stream-derived results indicate non-average melter behavior during the processing of glycolic acid-containing feed at the lower concentrations. With very few exceptions, feed-constituent losses, derived from secondary waste-stream data, were at a minimum during this processing campaign. However, this result is consistent with the fact that an extensive cold-cap structure developed and grew throughout the duration of this processing campaign.

6.3.5 Summary of Secondary Waste-Stream Findings

The data collected from RSM secondary waste streams suggest the following:

- There was a distinct difference in many of the scrub-solution elemental concentrations when glycolic acid was used as the reductant as opposed to the use of sugar or no reductant.
  - More organic, oC, and inorganic, iC, was collected in the scrub solutions.
  - A greater fraction of the captured elements was present in the HEME solutions.
  - Less settled solids were obtained from the EVS solutions.

- As could be expected, the halogens as a group comprised the highest percentage of feed elements collected in the scrub solutions. Fluorine was the highest followed by iodine and chlorine. Mercury was the only other element collected in the scrub solution at greater than 10% of that in the feed.
- The settled solids were mainly composed of SiO_2 and Fe_2O_3.
- The EVS scrub solution did not increase much in volume and decreased in acidity.

Table 6.19. Waste Stream-Derived, Campaign-Dependent Melter DFs

	Melter Process DF vs Feed Additive at SBW Conc				
			Glycolic		
Element	Cumulative Results	Sugar# 8/6-8/8	Low	High	No Reduct*
Al	298	265	1990	525	233
B	53	55	88	54	42
Ba	32	28	48	67	31
Ca	248	207	1460	497	216
Cd	32	28	61	24	34
Cl	3	4	13	15	2
Cr	49	45	79	58	48
Cs	27	24	42	27	28
Cu	15	22	16	20	7
F	2	2	2	1	2
Fe	407	333	1940	593	406
Gd	276	240	545	346	229
Hg**	4	4	6	5	2
I	6	9	3	3	5
K	43	37	81	51	48
Li	94	79	251	130	96
Mg	176	147	868	309	160
Mn	192	201	869	316	117
Mo	42	35	57	40	49
Na	51	43	101	62	55
Ni	81	130	86	115	41
P	294	229	733	350	306
Pb	97	90	198	93	96
Ru	132	211	215	158	62
S	30	26	54	30	33
Si	936	789	16300	1540	790
Ti	3930	3030	10100	7110	4710
V	289	206	1200	463	494
Zn	87	74	141	151	85
Zr	2870	3370	9130	1910	1730

* Processing with sugar @ 250 g/L-SBW for 1.5 of 27 total hours.
** HEME inventory not accounted for.
# Processing with sugar @ 200-, 180-, 46-, 170- & 175-g/L-SBW

7.0 Byproducts, Residuals, Mass Balance, and Volume Reduction

Beyond feed, glass, off-gas effluents, and process-system waste streams, other process products and residuals generated by the RSM test were also characterized for the sake of completeness. Specifically, samples of the melter's molten salt phase and off-gas system deposits formed during RSM processing were analyzed, and a physical accounting of the primary SBW process flowsheet streams and feed-component sulfur were conducted. The results of these evaluations are discussed below.

7.1 Molten-Salt-Phase Composition

Monitoring the RSM melt pool for accumulations of a molten salt phase was an important part of the SBW flowsheet evaluations. This was accomplished periodically by interrupting melter feeding, allowing the melter cold cap to burn off, and probing the melter's glass surface with a ceramic rod. By carefully contacting the molten salt pools on the glass surface with a room-temperature probe, frozen salt deposits could be selectively extracted from the melter. These solidified salt deposits were subsequently dissolved by leaching in distilled water. The resultant salt solutions formed were analyzed and found to be composed primarily of Na_2SO_4 with significant quantities of the alkali halides being present, as is summarized in Table 7.1. By making the reasonable assumptions and associations documented in Table 7.2, Na_2SO_4 was found, in all cases, to represent > 70% of the molten salt's mass with most of the remaining mass, 27 wt%, being accounted for by the alkali and alkaline-earth sulfates and halides. The presence of chromium in the salt layer, ~ < 0.5 wt%, is significantly less than previously observed during RSM-1. This is a reasonable result, given that salt-phase chromium is usually indicative of molten-salt-induced corrosion of the refractories and/or electrodes, and salt accumulations were minimal during the current test. Indeed, the relative weight fractions of chromium to sodium in the molten-salt phases sampled are nominally equivalent to its corresponding ratio in the glass. Consequently, no unusual molten salt-based corrosion of the melter's components is indicated by these compositional data.

7.2 Off-Gas Line Deposits

At the conclusion of RSM-2 testing, the off-gas line from the melter to the EVS was disassembled, inspected, and sampled. Figure 7.1 is a schematic of this segmented off-gas jumper that may be useful in providing perspective for understanding the interrelationship of these pipe segments and the samples obtained from them. Photographs of deposits within these pipe segments are displayed in Appendix H. These photographs indicate that significant pipe-wall deposition did not occur downstream of the film cooler. Beyond the effluent deposition growth at the inlet of the film-cooler that had to be periodically removed, as previously discussed in Section 5.2.2, no significant line narrowing was observed. In total, only about 200 g of material accumulated within the entire run of this off-gas line jumper over the ~ 96-h test period.

Discrete sampling of deposited material at the inlet and outlet of each of the off-gas line segments was conducted and later analyzed. The location and log number of each of these samples are identified in Figure 7.1, and their corresponding compositional data are summarized in Table 7.3. Leach analysis of these deposits allowed important anion information to be extracted from the sample that would otherwise

Table 7.1. Composition of Dissolved Molten Salt Samples*

	RSM-046		RSM-073	
Element	**Wt%**	**mEq/g**	**Wt%**	**mEq/g**
B	0.124	0.114	0.070	0.065
Ba	0.001	0.000	0.002	0.000
Ca	0.124	0.062	0.320	0.160
Ce	0.141	0.030	0.023	0.005
Cl	0.280	0.079	0.120	0.034
Cr	0.309	0.119	0.025	0.009
Cs	0.084	0.006	0.092	0.007
F	0.115	0.061	0.052	0.027
K	2.900	0.741	3.030	0.775
Li	1.820	2.630	2.260	3.250
Mg	0.004	0.004	0.003	0.003
Mo	0.085	0.053	0.084	0.053
Na	18.700	8.110	22.200	9.650
P	0.020	0.033	ND	ND
S	18.300	11.400	22.200	13.900
Si	0.084	0.120	0.056	0.080
Sr	0.032	0.007	0.062	0.014
V	0.505	0.496	0.789	0.774
Total	43.600	24.100	51.400	28.800
* mEq = milli-equivalents				

Table 7.2. Assumed Chemical Forms and Weight Contributions of Salt Constituents

Assumed Form	**Wt% Contrib.**	
	RSM-46	**RSM-73**
Na_2SO_4	72.600	73.300
Li_2SO_4	13.800	14.500
K_2SO_4	8.770	8.320
$CaSO_4$	----	1.120
$SrSO_4$	----	0.164
Cs_2SO_4	----	0.201
KCl	0.809	----
CsCl	0.243	----
$SrCl_2$	0.133	----
$CaCl_2$	0.019	----
$MgCl_2$	0.085	0.051
$BaCl_2$	0.005	0.000
CaF_2	0.542	----
Total (%)	97.000	97.600

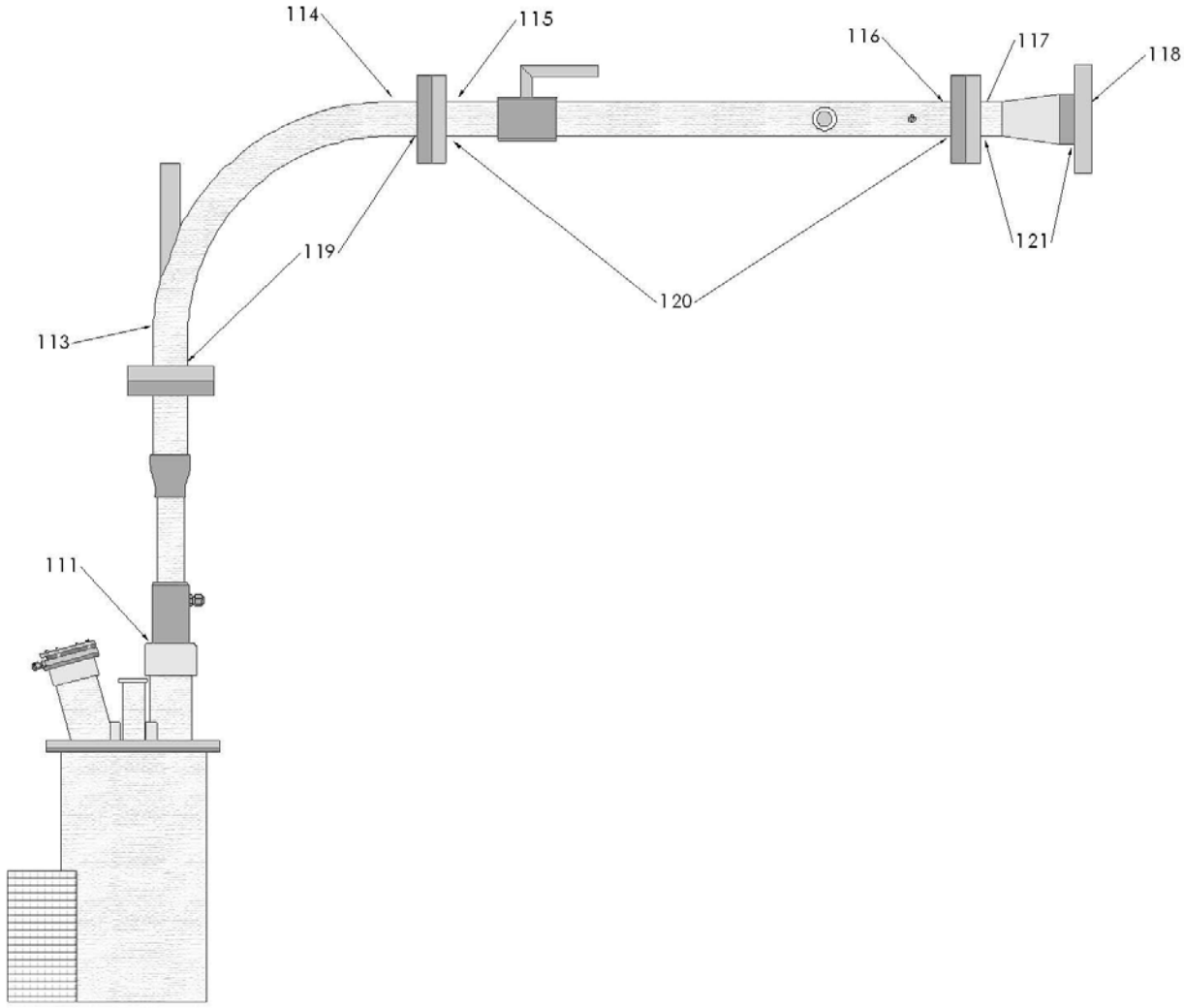

Figure 7.1. Melter Off-gas Jumper Configuration and Sample Site Locations

be unrecoverable after fusions were conducted. Therefore, the composition of analytes that could only be detected in the leachate portion of the sample has been assigned greater-than values.

Except for the film-cooler deposit, the compositions of all the other discrete samples are found to be nominally similar. This is especially true for the composition of the bulk samples that combined all the material within certain jumper segments. Figure 7.1 identifies, by log number, the jumper locations from which materials were collected, combined, and homogenized before being submitted for analyses, and Table 7.3 also summarizes these derived analytical data. Since the mass and composition of material collected from each segment is nominally the same, nothing much distinguishes these bulk materials form generalized process-generated particulate debris, which is, simply, semivolatile enriched feed material.

Table 7.3. Unnormalized Off-Gas Deposit Compositions

	Off-Gas Pipe Deposit Composition (wt%)									
	Discrete, Localized Samples							Homogenized Samples		
Element	**RSM-111**	**RSM-113**	**RSM-114**	**RSM-115**	**RSM-116**	**RSM-117**	**RSM-118**	**RSM-119**	**RSM-120**	**RSM-121**
Al	0.591	0.870	1.480	1.470	1.380	1.100	1.190	2.000	1.390	0.650
B	0.191	0.950	1.880	2.020	2.110	2.050	1.180	3.680	3.780	2.860
Ba	ND	ND	0.006	0.004	0.004	0.005	0.007	< 0.005	< 0.005	0.004
Ca	0.703	1.670	2.210	1.990	1.890	1.640	2.060	1.990	1.950	1.600
Cd	ND	0.031	0.012	0.015	0.025	0.016	0.015	0.031	0.027	0.027
Ce	ND	ND	ND	ND	ND	ND	ND	< 0.007	< 0.007	< 0.006
Cl^-	> 0.120	> 1.100	> 0.810	> 0.470	> 0.810	> 0.800	> 0.200	0.559	0.520	0.648
Co	ND	ND	ND	ND	ND	ND	ND	< 0.004	< 0.003	< 0.003
Cr	0.085	0.127	0.059	0.063	0.061	0.080	0.099	0.060	0.060	0.038
Cs	0.023	0.201	0.072	0.076	0.100	0.095	0.044	0.104	0.095	0.100
Cu	ND	ND	ND	ND	ND	ND	ND	0.005	0.004	0.002
F^-	> 0.150	> 0.320	> 0.550	> 0.620	> 0.600	> 0.600	> 0.540	0.930	0.940	0.360
Fe	2.140	0.403	0.966	0.969	1.410	0.658	0.646	0.938	0.835	0.502
Gd	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Hg	ND	ND	ND	ND	ND	ND	ND	0.012	0.055	0.086
I	ND	ND	ND	ND	ND	ND	ND	< 0.003	< 0.003	0.004
K	0.417	3.220	1.830	1.840	2.290	2.030	1.130	1.580	1.510	1.590
Li	0.255	2.610	1.990	1.940	1.980	2.020	1.160	ND	ND	ND
Mg	0.172	0.546	0.824	0.772	0.571	0.655	0.618	1.120	1.060	0.920
Mn	0.027	0.048	0.076	0.079	0.071	0.047	0.058	0.057	0.057	0.036
Mo	> 0.001	> 0.003	> 0.002	> 0.002	> 0.002	> 0.003	> 0.002	0.003	0.004	0.004
Na	3.320	15.400	9.630	9.610	10.600	11.900	10.700	11.700	10.000	7.220
Ni	0.093	0.033	0.055	0.047	0.044	0.032	0.043	0.016	0.033	0.014
P	ND	ND	ND	ND	ND	ND	ND	< 0.130	< 0.130	< 0.110
Pd	ND	ND	ND	ND	ND	ND	ND	< 0.001	< 0.001	< 0.001
Ru	> 0.002	> 0.114	> 0.004	> 0.005	> 0.006	> 0.007	> 0.002	0.053	0.047	0.031
S	0.152	1.620	0.743	0.671	0.774	0.677	0.609	0.827	0.774	0.744
Si	31.800	5.570	15.400	14.000	11.000	10.600	10.300	12.900	12.400	8.540
Sr	0.019	0.070	0.085	0.081	0.069	0.069	0.062	0.095	0.086	0.069
Ti	0.067	0.025	0.023	0.020	ND	ND	ND	0.007	< 0.009	< 0.008
V	0.643	0.673	0.982	0.771	0.756	0.909	0.853	0.837	0.731	0.608
Zr	0.822	0.227	0.860	0.831	0.534	0.648	0.532	0.592	0.611	0.290
NO_3^-	> 7.800	> 36.000	> 25.000	> 25.000	> 23.000	> 27.000	> 36.000	ND	ND	ND
$SO_4^=$	> 0.420	> 4.500	> 2.000	> 1.600	> 1.600	> 1.500	> 1.200	ND	ND	ND

7.3 Sulfur Processing Characteristics

The fate and behavior of sulfur in the two RSM SBW flowsheet studies conducted during FY 2001 were of major concern. The initial scoping test evaluated the effects of SBW waste loading (30 wt% to 35 wt%) upon molten-salt-phase accumulation using the SBW-9 glass formulation (Goles 2001).

Although steady-state accumulations of melter salt layers did not appear to be a processing limitation, the glass could not fully accommodate the feed-stream sulfur, thus resulting in significant sulfur concentrations in secondary process waste streams. Because waste-stream recycle was part of the conceptual process flowsheet, a new glass formulation, SBW-22, was developed, for the current test, that was to eliminate recycle-induced, feed-stream sulfur buildup by accommodating all available SBW sulfur. If successful, this glass formulation would minimize off-gas system losses and concomitant sulfur buildup in secondary waste streams.

7.3.1 Process Campaigns, Feed Reductants, and Vitreous Sulfur

Since SBW-22 has a finite capacity for sulfur, the SBW waste loading was fixed at a moderately conservative 20 wt% throughout the RSM-2 test. The glass sulfur concentration at this waste loading was 0.9 wt% as SO_3. Since sulfur off-gas partitioning can also be influenced by reductant type and concentration, both sugar and glycolic acid at various feed loadings were evaluated against the SBW-22 feed formulation without reductant. The processing plan that was executed during the current test is graphically summarized in Figure 7.2. The feed-processing campaigns identified by reductant additive and concentration are plotted along with the historical sulfur glass-composition information obtained from ICP-AES analysis at PNNL and SRTC laboratories and from independent x-ray analyses.

As indicated in the graphical display, the initial glass loading of sulfur was ~ 0.7 wt% that was representative of the final RSM-1, SBW-9 glass formulation (0.89 wt% SO_3) that had been diluted by ~ 40% with low-sulfur borosilicate glass. As processing commenced and progressed over the next 2 days, the sulfur gradually built up in the glass. This is totally consistent with visual observations of the melter glass surface that were periodically conducted to monitor for molten-salt-phase development. The noncontiguous pools of molten salt that were observed steadily decreased throughout this period.

Although it would be beneficial to have greater time-resolved glass sulfur data, one could conclude from Figure 7.2 that sugar at 200 g/L-SBW was causing sulfur partitioning at the expense of glass incorporation. The glass composition after 2.6 melter turnovers was nominally representative of the SBW-22 glass formulation (see Table 4.11), but sulfur loading indicated by the ICP-AES analyses had only increased slightly on the average. A larger increase in the x-ray data was recorded, but these data appear to be biased systematically high. Therefore, on the basis of relative trends, all three analytical approaches indicate only a small average increase in sulfur composition. Since significant salt-phase accumulations were not occurring during this period, it appears that sugar reductant at 200 g/L-SBW was reducing the amount of sulfur available to the glass, which is further supported by the secondary-waste-stream composition data that suggest higher off-gas partitioning of sulfur occurred during a period that was inclusive of this processing phase.

In between the sulfur data points at 2.6 and 6.6 melter glass turnovers when feed containing lower levels of sugar were processed, the sulfur content rose to near its target level of 0.9 wt%. All analytical trends show that this sulfur glass composition was stable or slightly increasing throughout all phases of

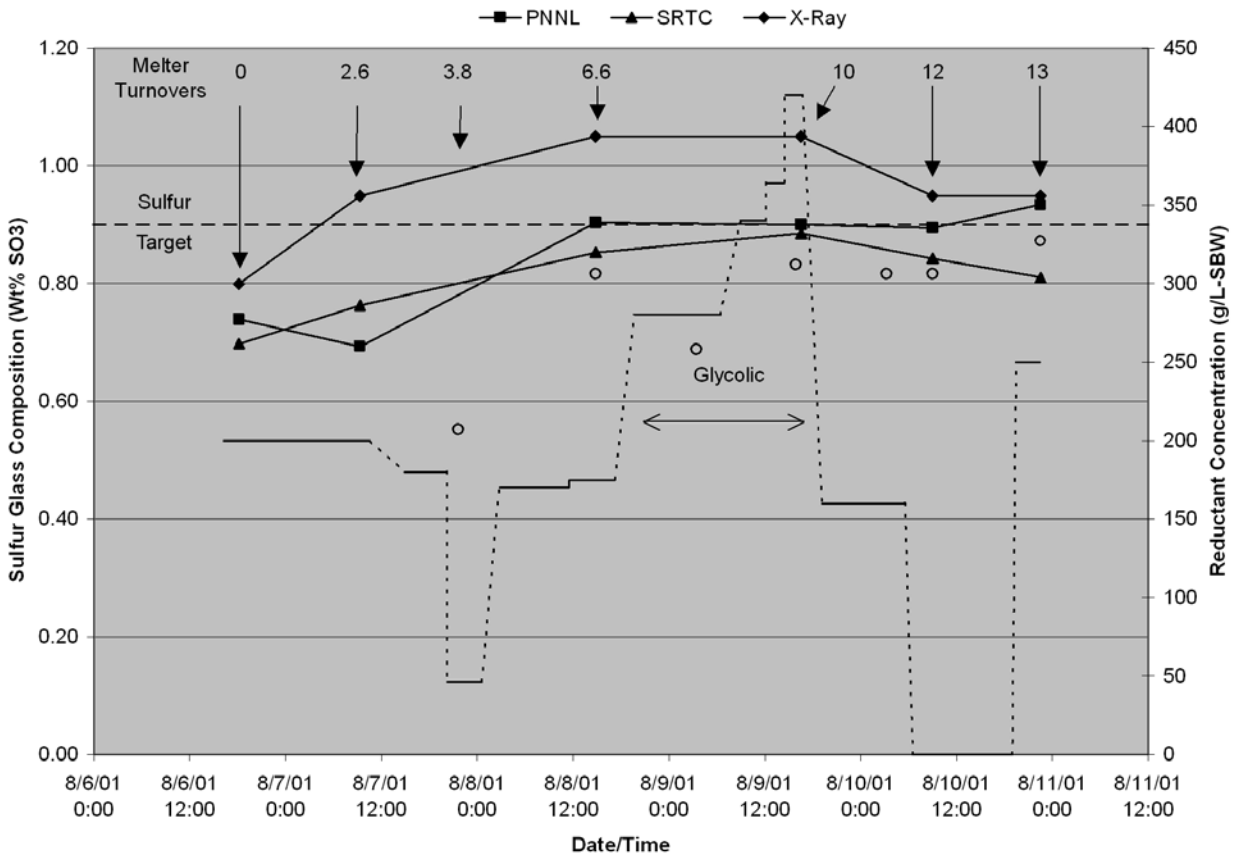

Figure 7.2. Historical SBW-22 Sugar/Glycolic and Glass Sulfur Concentrations

processing using glycolic feed reductant. This is also consistent with previous observations regarding glycolic acid's ineffectiveness as a glass oxidation state modifier.

At the conclusion of glycolic testing, and after a period of processing with sugar reductant and then no reductant, two of the three analytical sets of data suggested a downward trend in sulfur glass loading. This trend continued for one of the ICP-AES data sets as processing continued without reductant followed by a short processing period (1.5 h) involving feed containing 250 g-sugar/L-SBW; however, the other two data sets suggested nominally stable glass sulfur composition.

Since sugar loading used at the conclusion of the glycolic processing campaigns was no higher than that used before glycolic testing, a downward trend in sulfur loading is inconsistent with the previous results. Added to this is the fact that processing without reductant also preceded the results showing the first decline in sulfur glass loading after glycolic testing. Since the combination of these facts is difficult to rationalize, it may be that analytical uncertainties associated with a limited number of samples are creating an impression of analytical variances that are neither real nor significant.

However, impressions that are real are those formed from off-gas studies and physical observations. The off-gas data show no evidence of increased sulfur off-gas partitioning after the processing campaigns using glycolic reductant. In addition, visual observations suggested a continual decline in the number and size of molten-salt-phase pools that were observable on the melter glass surface. Although never completely absent, the presence of molten salt present at the end of the test was quite difficult to identify and observe.

The fact that SBW-22 has an inherently higher sulfur solubility than SBW-9 is evidenced by the limited quantity of salt that precipitates out of the glass during the cooling process. Appendix H contains photographs of the surface of canisterized glass collected during the current test. These photographs clearly show limited salt separation occurring upon cooling of the SBW-22 formulation. Unfortunately, similar photographs for SBW-9 canisterized glass do not exist, but much more extensive salt separation occurred when this glass was cooled and solidified.

7.3.2 Sulfur and Sulfide

New wet-chemical methods have been developed to determine the total sulfur and sulfide content of vitreous materials.[(a)] These new procedures were applied to RSM-2 glasses, and the results obtained are summarized in Table 7.4. Although no sulfide was detected in any of the glasses tested, the total sulfur values derived from this unvalidated analytical procedure are included in Figure 7.2 for completeness (open circles). When compared to the ICP-AES data, these results appear to be biased somewhat low. However, the overall trend of these data is nominally consistent with the previously discussed glass analytical results.

Indeed, these new data also show low sulfur glass retention during the processing of feed containing higher levels of sugar (200- and 180-g/L-SBW) that increased after feeds containing lower concentrations of sugar (46- 170- and 175-g/L-SBW) were processed. If one neglects the data point in the middle of the glycolic processing campaign, these data exhibited a historical trend that is quite similar to PNNL's ICP-AES time-dependent results. Unfortunately, the increase in glass sulfur content that both data sets show after processing feed containing 250 g-sugar/L-SBW is somewhat inconsistent with the higher sulfur-partitioning results accompanying the processing of feeds containing up to 200 g-sugar/L-SBW at the outset of testing. However, both sulfur composition increases are small and, therefore, are probably not significant.

Table 7.4. Total Sulfur and Sulfide Content of SBW-22 Glasses By Wet Chemical Methods

	Total Sulfur Wt% as SO_3 and Sulfide Content of SBW-22 Glasses: RSM-01-2(-XYZ)						
Cmpnd	**RSM-023**	**RSM-040**	**RSM-053**	**RSM-067**	**RSM-081**	**RSM-094**	**RSM-100**
Total S	0.55	0.82	0.69	0.83	0.82	0.82	0.87
S^{-2}	< 22 ppm	< 33 ppm	< 28 ppm	< 33 ppm	< 33 ppm	< 33 ppm	< 35 ppm

(a) DD Siemer, Bechtel, Babcock, and Willcox Idaho, Idaho National Engineering and Environmental Laboratory, Idaho, Falls, Idaho (2001).

7.4 Process Mass Balance

The compositional data of process streams previously discussed and summarized were combined in an attempt to fully characterize the SBW vitrification process flowsheet. Analytical feed compositional data were used whenever possible or reasonable; otherwise, target values were employed. The mass-balance results for the overall test are summarized in Table 7.5. These data have been segregated into three sections, depending upon the completeness of the information available. The first section, containing the majority of feed constituents, is supported by adequate process data. Recognizing the limitations imposed by analytical uncertainties, reasonable mass closure is demonstrated for most of the feed constituents in this group. The extremely good mass-closure results for sulfur strongly suggest, as mentioned earlier, that SO_2 melter off-gas partitioning is not a significant effluent loss mechanism as inefficient collection of this effluent by the melter's off-gas effluent scrubbing equipment would have otherwise created a measurable mass-balance deficit.

The second group of feed constituents represents elements whose compositional data are thought to be compromised by experimental/analytical limitations. The glass data for Cr, Mo, and Ti are unreasonably high because of the contribution from non-feed-stream sources. Refractory corrosion and/or chemical impurity contributions to extremely small feed-constituent concentrations are likely sources of off-normal glass results. The abnormally low mass-closure result for nickel may be due to the formation of nickel carbonyl, a room-temperature gas, that would not only enhance melter losses, but would effectively challenge the efficiency of the melter's off-gas emission abatement system. Analytical uncertainties in both feed and glass composition data limit the ability to provide a meaningful mass closure for the low levels of Ru employed in the RSM-2 test.

The third group of elements in Table 7.5 represents feed constituents for which analytical glass data are not available. The mass-closure data for barium and gadolinium suggest that 97% and 99.6%, respectively, of these processed elements were incorporated in the melter's vitreous product. This is not an unreasonable inference! On the other hand, it is probably unreasonable to suggest that glass accumulations are responsible for the large difference between processed and waste stream accumulations of mercury, fluorine, and iodine. Previous studies have shown that borosilicate glass has very little capacity for these elements (Goles 1990, 1996). Rather, these data suggest that melter losses are dominated by volatility, which has been previously demonstrated (see Section 6.2.4), and that these persistent volatiles successfully challenged the capabilities of the RSM's off-gas processing system or, if captured, were not recoverable from the HEME.

7.5 Process Statistics

During the August 2001 melter-flowsheet evaluation studies, 90 L (24 gal) of SBW simulated waste having a total mass of 110 kg was processed by the RSM, producing 22 L (5.7 gal) of glass having a total mass of 55 kg. Although vitrification results in both mass and volume waste reductions, only the volume-reduction parameter is meaningful since the major mass contributors to the SBW (H_2O and NO_3^-) are nonvitrifiable, volatile species. On the other hand, volume reduction has important waste-disposal implications. This is because most of the hazardous and radwaste SBW components can be incorporated

and immobilized in the melter's vitreous product, and tank waste volumes of SBW are a physical reality. During the current RSM test, an overall SBW waste-volume reduction factor of 4.2 was achieved. This factor is lower than that recorded (7.6) during the previous SBW melter test because of the significantly lower waste loading associated with the SBW-22 formulation.

Table 7.5. Stream-Dependent % Accumulation Per Liter of SBW Processed

		EVS				
Element	**Glass**	**Solution**	**Solids**	**HEME**	**Total**	**Delta**
Al	106.000	0.291	0.0030	0.042	107.0	-6.6
B	109.000	1.320	0.0007	0.556	111.0	-11.0
Ca	104.000	0.306	0.0003	0.098	105.0	-4.8
Cd	108.000	1.490	0.0093	1.660	111.0	-11.0
Cl	66.300	23.500	0.0140	5.170	95.1	5.0
Cs	98.100	1.960	0.0003	1.820	102.0	-1.9
Cu	92.500	5.640	0.0310	0.982	99.1	0.9
Fe	112.000	0.126	0.1020	0.019	112.0	-12.0
K	104.000	1.370	0.0015	0.934	106.0	-6.0
Li	97.900	0.743	0.0003	0.323	99.0	1.0
Mg	106.000	0.462	0.0008	0.105	107.0	-6.9
Mn	106.000	0.445	0.0062	0.071	107.0	-7.0
Na	111.000	1.300	0.0004	0.700	113.0	-13.0
P	107.000	0.274	0.0051	0.061	107.0	-7.4
Pb	98.400	0.537	0.0001	0.498	99.4	0.6
S	99.900	1.850	0.0015	1.540	103.0	-3.3
Si	98.000	0.043	0.0635	0.001	98.1	1.9
Sr	98.700	0.000	0.0000	0.000	98.7	1.3
V	93.400	0.266	0.0002	0.080	93.7	6.3
Zn	99.100	0.793	0.0138	0.339	100.0	-0.2
Zr	99.000	0.004	0.0285	0.000	99.1	0.9
Cr	226.000	1.090	0.1070	0.856	228	-130
Mo	285.000	1.130	0.0693	1.240	287	-190
Ni	55.200	1.110	0.0123	0.124	57	44
Ru	145.000	0.659	0.0459	0.052	146	-46
Ti	5340.000	0.010	0.0097	0.001	5340	-5200
Ba	ND	1.660	0.0634	1.370	3	97
F	ND	43.400	0.1200	13.200	56.7	43.3
Gd	ND	0.308	0.0069	0.048	0.4	99.6
Hg	ND	19.000	0.0091	54.300	73.3	26.7
I	ND	15.200	0.0000	21.800	37.0	63.0

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Appendix A

Test Plan for the RSM-01-2 Vitrification Demonstration for INEEL Sodium-Bearing Waste

Appendix A: Test Plan for the RSM-01-2 Vitrification Demonstration for INEEL Sodium-Bearing Waste

Nick Soelberg, INEEL
Ron Goles, PNNL
John McCray, INEEL
Sylvester Losinski, INEEL

July 27, 2001

Pacific Northwest National Laboratory
Richland, Washington 99352

Idaho National Engineering and Environmental Laboratory
Idaho Falls, Idaho 83415

APPROVALS

PREPARED BY:

_____ _____
PNNL R. W. Goles Date

_____ _____
INEEL N. R. Soelberg Date

APPROVED BY:

_____ _____
RW Goles, Lead PNNL Investigator Date

_____ _____
LM Peurrung, Line Manager, Thermal Processing Group Date

CONCURRENCE:

_____ _____
JM Davis, RSM Equipment Custodian Date

_____ _____
RM Gough, Safety Representative Date

_____ _____
PH Rojas, APEL Building Manager Date

RECEIPT ACKNOWLEDGED:

_____ _____
CR Allen, APEL Building Manager Date

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TEST PLAN FOR VITRIFICATION DEMONSTRATION TESTS OF INEEL SODIUM-BEARING WASTE

1. INTRODUCTION

Over several decades, operations at the U.S. Department of Energy (DOE) Idaho National Engineering and Environmental Laboratory (INEEL, formerly called the Idaho National Engineering Laboratory, INEL, and before that the Nuclear Reactor Testing Station, NRTS) has performed nuclear reactor testing, reprocessing of spent nuclear fuel, and storage, treatment, and disposal of radioactive and mixed wastes that result from reactor operations and nuclear fuel reprocessing operations. Liquid, acidic, and radioactive high-level waste (HLW) and sodium bearing waste (SBW) from spent fuel reprocessing operations has been for the most part calcined in the New Waste Calcining Facility (NWCF) and the earlier Waste Calcining Facility (WCF) to convert the liquid waste into a dry granular calcine that is safer to store. About 1-million gallons of liquid SBW remains stored in the Tank Farm Facility (TFF). Current plans are to vitrify rather than calcine this remaining SBW because DOE has determined to close the NWCF calciner in Fiscal Year 2000 rather than upgrade and permit this facility to meet new regulatory requirements, and because even the calcine is not expected to meet long-term disposal requirements.

The SBW is an aqueous, highly acidic (1 to 3 molar nitric acid) solution containing dissolved and suspended radionuclides, heavy metals, and other species, including halogens. This waste is a listed, mixed waste, containing both radionuclides, hazardous characteristics (corrosivity and characteristic metals), and small amounts of listed organic constituents.

Demonstration vitrification tests are being conducted during Fiscal Year 2001. An initial, small-scale, week-long test was performed in January 2001 using the Research Scale Melter (RSM) located at the Applied Process Engineering Laboratory (APEL) facility in Richland, Washington. A week-long larger, pilot-scale test was also performed in April 2000 at the Clemson Environmental Technologies Laboratory (CETL) at Clemson University in Anderson, South Carolina. Laboratory crucible tests have also been performed prior to and simultaneously with research and pilot-scale demonstration tests, in part to provide initial information for establishing surrogate feed recipes and target melter-operating conditions for the demonstration tests.

This test plan describes the second RSM test series (termed RSM-2) planned for early August 2001.

2. OBJECTIVES

The RSM-2 test series is defined to evaluate the melt rate, off-gas emissions, and the performance of the RSM-2 off-gas system while feeding a mixture of WM-180 surrogate waste, glass formers, and a reductant in proportions designed (for most of the test series) to prevent the formation of any detectable sulfate layer. Some test conditions are also included to evaluate melter performance and off-gas emissions while excluding any organic reductant from the feed and to evaluate higher sulfur feed formulations. Specific objectives of the RSM-2 test series, in order of priority, are summarized as follows:

1. Determine the best reasonably achieved melt rate and melter system performance under three different melter redox conditions, two different levels of sulfur in the surrogate feed, and using two different reducing agents.

2. Confirm the presence or lack of a separate sulfate layer during all operating conditions. The low-sulfate feed mixture was designed based on engineering studies and crucible tests to result in no detectable sulfate layer formation. Perform corrective actions if, in the opinion of the test leader, the presence of a sulfate layer becomes excessive.

3. Evaluate the fate and speciation of mercury in the feed mixture for the different melter redox conditions and reductants.

4. Determine the fate of the surrogate feed and other feed constituents, including the reductant, halides, nitrates, sulfur, metals that are hazardous or are surrogates for hazardous metals, and elements that that are surrogates for radionuclides.

5. Characterize wet scrubber performance for scrubbing water, Hg species, halides, NO_x species, SO_2, and particulate matter, under the different melter operating conditions but at a single specified scrubber pressure drop.

6. Determine the composition, properties, and amounts of all system effluent streams, including the product glass, scrub solution (including dissolved and undissolved solids), off-gas (including particulate matter [PM] in the off-gas), and PM deposits recovered from the off-gas piping. The product glass must meet waste acceptance criteria for high-level waste borosilicate glass.

7. Demonstrate and determine the performance of an online redox electrode compared with the traditional wet chemistry redox analysis technique.

Objective 1 will be accomplished by determining the highest surrogate waste feedrate that can be achieved, as determined by 1) avoidance of an excessive sulfate layer or foaming, 2) coverage of a cold cap of unmelted feed on the surface of the molten glass, 3) a minimum glass melt temperature, measured at two specified locations, and 4) a minimum glass melt electrical conductivity, determined by the input voltage and current, that enables the voltage to stay below a specified maximum value.

The degree of cold cap coverage will be determined by visual observation and by monitoring the plenum temperature. The plenum temperature will be used as an indicator of cold-cap coverage on the melt surface instead of solely relying on visual observation, because the entire melt surface is not visible through the sight port, and the entire extent of cold-cap coverage is not readily determined based on the sight port view. The plenum temperature is assumed to rapidly drop when radiant heat from the melt is blocked as the melt surface becomes almost completely covered by unmelted cold cap. The feedrate and plenum temperature will be correlated to identify a minimum plenum temperature to maintain the plenum, by modulating the feedrate. When the plenum temperature drops below the specified limit, then the

feedrate will be reduced. This approach assumes that the feedrate is limited by the amount and degree of cold-cap coverage and not by the bulk glass melt temperature or conductivity; the maximum feedrate will be defined as the highest feedrate that results in a plenum temperature above a specified limit.

Objective 2 will be accomplished by frequently observing, through the melter sight port, the surface of the melt. At selected times, the melt surface will be manually probed to more rigorously observe any presence of a sulfate layer and to retrieve samples of a sulfate layer if detected. Due to the presence of Hg in the feed, probing activities will be performed according to strict procedures to prevent fugitive release of Hg from melter. Probing activities may be less frequently performed compared to the frequency during the RSM-1 test.

Objectives 3 through 6 will be accomplished using 1) continuous emissions monitoring (CEM), 2) manual off-gas sample collection and analysis, and 3) sampling and analysis of all process effluent streams. CEM and manual off-gas sampling will be performed at two locations in the off-gas system—at the film cooler outlet (which is also the scrubber inlet), and the scrubber and high efficiency mist eliminator (HEME) outlet.

Objective 7 will be accomplished through the installation of a redox electrode on the RSM melter and comparing responses of that electrode with measured Fe^{+2}/Fe_{tot} mole ratios of respective glass samples.

Melter system continuous process monitoring (CPM) will be performed during all test conditions to monitor, record, and control system flowrates, temperatures, pressures, and input power, to assure safe operation within reasonable ranges determined by the test leader that avoids unnecessary risk of damage to the system or of hazards to operating personnel or others.

Not included in the scope of this test are:

- Evaluation of the performance of other reductants besides sucrose and glycolic acid.

- Parametric evaluations of scrubber performance under conditions (such as varied pressure drop) that do not result from the different melter parametric variations (degree of redox, feedrate, type of reductant). Because of how the scrubber system will be operated and sampled, the scrubber will likely not reach steady state during a given test condition.

- *In situ* determination of particle size distribution of the entrained/volatilized particulate matter. INEEL will conduct post-test estimates of particle size by scanning electron spectroscopy or Coulter Counter analysis of filter or bulk particulate matter (PM) sample fractions.

- Evaluations with sample collection and analysis to determine PM emissions for different melter conditions such as reduced cold cap.

- Long-term, steady-state operation for evaluating key components of the system. An approximately 1-week test is insufficient to evaluate the effects of such conditions as corrosion or deposition that may not be noticeable in a 1-week test, but may become important in long-term operation. Furthermore, several feed changes are planned for the melter, and quantitative recovery of the scrub solution is planned for those feed changes. While the melter may re-equilibrate after several hours of operation after a feed change, re-equilibration of the scrubber solution is not expected after each time that the scrub solution is refilled with fresh solution. The effects of these non-steady-state conditions should be considered during data evaluation.

- Quantitative or qualitative measurement of speciated volatile organic compound (VOC) emissions or semivolatile organic compound (SVOC) emissions, including such compounds as polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans. Evaluations of formation or emissions of these

species must be performed in other studies such as tests being performed in the EV-16 melter at Clemson University.

- Evaluations of candidate NO_x control technologies such as selective catalytic reduction (SCR).

3. PROJECT ORGANIZATION AND SCHEDULE

The RSM-2 test program will be performed primarily by personnel from Pacific Northwest National Laboratory (PNNL), INEEL, and Science Applications International, Inc. (SAIC) under subcontract to PNNL. Project activities and performing organizations are listed as follows:

- INEEL provides:
  (a) Project definition and test objectives
  (b) SBW composition and determination of reductant
  (c) Test plan preparation support
  (d) Project schedule definition
  (e) An Hg and $HgCl_2$ continuous emissions monitoring system (CEMS)
  (f) Operation of the Hg/$HgCl_2$ CEMS, including control and data logging
  (g) Onsite test team support of at least 1 engineer or technician on all shifts during testing
  (h) Direction of manual off-gas sampling and analysis
  (i) Sample analysis of all scrub solutions and off-gas train samples
  (j) Hg CEMS, wet scrubber, and off-gas sample data reduction and evaluation
  (k) Test report preparation for the Hg CEMS, wet scrubber, and off-gas sample sections
  (l) Overall test report review and comment.

- PNNL provides:
  (a) Overall responsibility for the performance of the test
  (b) Test facility (RSM system) and support infrastructure (utilities, electrician, machine shop, etc.)
  (c) Feed procurement and preparation
  (d) Facility and test plan preparations
  (e) Interface of the INEEL-provided Hg CEMS with the PNNL sampling system
  (f) A CEMS that includes pre-existing analysis capabilities and an additional SO_2 CEM
  (g) Training for INEEL and subcontractor personnel who will participate in onsite testing
  (h) Subcontracted manual off-gas sampling capability
  (i) Test team direction during the test series
  (j) A minimum of 1 engineer or technician on all shifts during RSM operations
  (k) A minimum of 1 engineer for operating the PNNL-provided CEMS during selected test periods
  (l) A minimum of 1 engineer or technician for manual off-gas sample train operation
  (m) Sample containers for all samples
  (n) Containers (six 50-L carboys) for scrub-solution settling containers
  (o) Reagent (concentrated HNO_3, 15.8 M, in twenty-four
  (p) 1-L bottles, for pre-acidifying scrub solution
  (q) Reagents used in manual off-gas sample train operation and recovery
  (r) Reagents needed for continuous Hg monitoring
  (s) Calibration gases used in calibrating the PNNL-provided CEMS
  (t) RSM process monitor and CEMS and data logging, except for equipment provided by the INEEL
  (u) Feed and glass sample analysis
  (v) All data reduction and analysis except for those items identified above as INEEL's responsibility
  (w) All test report preparation except for those items identified above as INEEL's responsibility.

- SAIC provides manual off-gas sample train equipment and personnel to the extent of:
  (a) Pretest preparation of sample train equipment and procedures
  (b) Specification of manual sampling reagents that PNNL must have available for the test
  (c) Off-gas train sample containers
  (d) All specialized equipment required for sample train operation and recovery
  (e) Appropriate backup equipment to avoid sampling delays in the event of sampling failure
  (f) Sample train operator checklists, data sheets, and data-reduction spreadsheets
  (g) Pretest and post-test equipment calibrations and maintenance
  (h) Equipment calibration records.

Laboratory contact personnel at INEEL are Rick Anselmo (overall) and Jeff Lau (metals analysis). The PNNL laboratory contact person is May Lin Thomas (509-376-5936).

An approximate schedule is shown in Table 1. This schedule includes preparation of an interim test report by September 28, 2001, to meet a DOE Tanks Focus Area milestone for the RSM-2 test series. Since this date is too early for inclusion and evaluation of all laboratory analysis results, this report will be revised, or a second report will be issued after the laboratory data are received and evaluated. The scheduled activities are all on the critical path for project completion. Any delays in these activities could extend this schedule beyond the estimated dates.

Table 1. RSM Test Program Schedule

Activity	Start date	Completion date	Performer
Complete Interoffice Work Order	May	June 14 (done)	INEEL
Draft test plan	May	July 2 (done)	INEEL, PNNL
Procure feed components	June	July 23	PNNL
Finalize test plan	---	Aug 3	INEEL, PNNL
Prepare test facility, including modifications of the off-gas system, the enclosure for Hg control, and PNNL CEMS	June	Aug 3	PNNL
Procure manual sample train subcontract	July 2	July 23	PNNL
Perform manual sample train equipment and procedure preparation	July 23	Aug 3	SAIC
Procure rental SO_2 CEM	July 16	July 27	PNNL, INEEL
Perform pretest shakedown of new and modified RSM equipment	July 30	Aug 3	PNNL, INEEL
Prepare feed mixture(s)	July 30	Aug 3	PNNL
Install and shakedown Hg CEM and manual sample trains	Aug 6	Aug 7	PNNL, INEEL
Conduct test	Aug 6	Aug 10	PNNL, INEEL, SAIC
Cleanup / Waste Disposal	Aug 13	Aug 31	PNNL
Post-test inspections, calibrations, facility cleanup, sample collection, and sample delivery to laboratories	Aug 9	Aug 17	PNNL, INEEL, SAIC
Laboratory analysis	Aug 13	Sep 27	PNNL, INEEL
Continuous process monitor and continuous off-gas monitor data reduction	Aug 13	Aug 31	PNNL, INEEL
Draft interim report	Aug 13	Sep 7	PNNL, INEEL
Review interim report	Sep 7	Sep 21	PNNL, INEEL
Finalize and submit interim report	Sep 24	Sep 28	PNNL, INEEL
Laboratory data reduction and quality control	Sep 17	Oct 12	PNNL, INEEL
Draft final report	Oct 15	Oct 31	PNNL, INEEL
Review draft final report	Nov 1	Nov 15	PNNL, INEEL
Finalize and submit final report	Nov 16	Nov 29	PNNL

4. TEST PROGRAM

The RSM-2 test series is designed for approximately 5 days of continuous, 24-hour-per-day melter operation. A target test condition matrix is shown in Table 2. This matrix is designed to satisfy the test objectives described in Section 2.

Independent variables that will be varied to establish each test condition are:

- Glass redox state, which is measured by the Fe^{+2}/Fe_{tot} ratio, and which is controlled by varying the ratio of added reductant to nitrates in the feed. The ratio of reductants to nitrates in the feed will be varied between (a) 0, in order to assess if any reductant is needed to improve melter performance, considering that the use of an organic reductant will limit treatment system design and permitting flexibility, (b) just enough to react with some of the feed nitrates, with only a slight excess that reduces Fe^{+3} to Fe^{+2} only to the extent that the Fe^{+2}/Fe_{tot} ratio is above its detection limit of about 0.01, and (c) enough added excess reductant that the Fe^{+2}/Fe_{tot} ratio approaches the limit considered to be safe (about 0.3) long-term melter performance.

- Kind of reductant. The evaluation of 2 different reductants (sucrose and glycolic acid) is planned in the RSM-2 test series. These reductants are summarized in Table 3, compared to other potential reductants. Separate batches with different reductants will be prepared in a separate mix or feed tank to facilitate rapid switching between feeds with different kinds or amounts of reductants during the test series.

- Level of sulfate in the feed. Limited testing (toward the end of the planned test series) is planned to evaluate a higher level of sulfate in the feed, in order to bound the possible ranges of sulfate in the actual SBW. At this higher sulfate level, a high level of reductant, with a high degree of melt reduction, is planned in order to better minimize the formation and amount of a separate sulfate layer.

The glass formulation will not be changed during the test series, except possibly to establish the high sulfate feed test condition near the end of the test. The high sulfate condition will be achieved by either changing the glass formulation or by simply adding more sulfate to the low-sulfate formulation.

The ability of this test series to accomplish all planned test objectives depends on how rapidly the melter glass bath, the cold top, and the off-gas respond to step changes that occur for each of the different test conditions. Determining the maximum melt rate, the redox condition of the glass, and the presence/absence of a sulfate layer may require some trial and error operation. Target operating conditions based on design parameters and optimal melter-system operation are shown in Table 4.

Table 2. Target Test Matrix for the January 2001 RSM Test Series

Test Condition (a)	Glass Fe^{+2}/Fe_{tot} Ratio	Reductant	Feedrate	Sulfate layer	Dura-tion	Cumu-lative hours	Sample collection (b)	
							Manual trains	Scrub solution
1 (oxidizing, no sulfate)	0.01-0.05	Sucrose	Highest optimum (~1.5 liters/hr) (c)	None	36	36	x	x
2 (oxidizing, no sulfate)		Glycolic acid			8	44	---	---
3 (oxidizing, no reductant)	Non-detectable	None		TBD (d)	8	52	x	x
4 (reducing, no sulfate)	0.2-0.3	Sucrose		None	24	76	x	x
5 (reducing, no sulfate)		Glycolic acid			8	84	x	x
6 (High sulfate (e), reducing, add Re)	<0.3	Sucrose		TBD	12	96	x	x
Contingency (f)	TBD	TBD		TBD	5	101	---	---

a. The waste loading (% of surrogate SBW in the feed mixture) will be determined from crucible tests to be the highest value that provides a reasonable expectation that stable, sulfate-phase free operation can be established and maintained. This value will be lower than achieved during the RSM-1 test because a sulfate layer was observed in that test. Continuous process monitor (CPM) and CEM data will be logged during each test condition. Process and off-gas sampling will be performed only for selected test conditions when the glass melt has approached steady state.

b. After steady state is reached, approximately 4 hours or more may be required to perform manual sample trains, observe and log CPM and CEM data, and collect the scrubber solution. The test plan allows for the collection and analysis of five separate sets of manual sample trains and scrubber solution samples. These samples will be collected for analysis only during selected test conditions.

c. The feedrate may be adjusted if necessary to try to achieve an optimum 90 to 95% cold cap. This level of coverage will be determined by monitoring the melter plenum temperature. When coverage is nearly complete, the plenum temperature is expected to drop precipitously. This temperature will be determined from historical RSM data and from experimentation during Test Condition 1. After determining this temperature, the feedrate will be increased to the point where the plenum temperature approaches the precipitous decline within an administrative limit determined by the test leader.

d. TBD = To be determined

e. The high sulfate condition may be achieved by simply adding more sulfate to the low-sulfate feed mixture, or it may be achieved by changing the feed mixture and waste loading back to the mixture used in the RSM-1 test.

f. The contingency time is the difference between the scheduled time for the specified test conditions and the total of 101 operating hours based on a nominal test start time of 1200 on August 6, and a nominal end time of 1700 on August 10. This contingency time allows for extending the duration of any planned test conditions, adding yet-to-be-planned test conditions, repeat test conditions, or shortening the total test time, as determined by the test leader.

Table 3. Reductants Included in the RSM-2 Test Series

Reductant	Composi-tion	Mole weight	H:C ratio	O:C ratio	Soluble in ~1 M Nitric Acid	Thermal Decomposition Temperature, C	Comments
Sucrose (a)	$C_{12}H_{22}O_{11}$	342	1.83	0.91	Soluble	186 (melting point)	Baseline reductant, used in the RSM-1 test
Glycolic acid (a)	$C_2H_3O_3$	76	1.5	1.5	Soluble	80 (melting point)	Most volatile/reactive based on O:C ratio and decomposition temperature
Starch (b)	$(C_6H_{10}O_5)_n$	$(162)_n$	1.67	0.83	?	Not available	
Charcoal (b, c)	85-98% C, remainder O and H	$(12)_n$	~0.01	<0.1	Insoluble	500-600	Least reactive and could be very persistent

a. Because of their relatively high H:C and O:C ratios, solubility in nitric acid, and reactivity with nitrates and NO_x species in the cold top and melter plenum, both sucrose and glycolic acid are considered leading candidates as SBW feed reductants. These properties make these two additives relatively easy to blend in the feed and favor reaction of these reductants with nitrates and NO_x in the cold top and plenum. Because of their volatility, reactivity, and high O:C ratios, they may produce lower levels of products of incomplete oxidation compared to those reductants with lower H:C and O:C ratios. However, because of their volatility and reactivity, they may not persist long enough in the melter for complete reaction with nitrates/NO_x, and they may react too readily with gas-phase oxygen or oxygen from other sources such as water.

b. If sucrose or glycolic acid are too reactive (and react unselectively with other sources of oxygen besides nitrates and NO_x) or are too volatile to persist long enough to react with nitrates and NO_x in the cold top or plenum, then less hydrogenated and oxygenated reductants like starch or charcoal may persist longer in the melter to react longer with nitrates/NO_x. Because of their relatively low volatility (especially for charcoal), these may react with species in the glass melt, making it too reducing. This concern, plus less easy feed blending compared to sucrose or glycolic acid, would need to be resolved for continued interest in using these reductants.

c. Neither cornstarch nor charcoal is planned for inclusion in RSM-2; they are included in this table for illustration and comparison of property trends for the other candidate reductants.

At a given set of operating conditions, some operating time is needed to (a) detect changes in some conditions such cold cap coverage and a separate molten sulfate salt layer, and (b) allow time for the melt bath composition to approach a new equilibrium after step changes in the feed composition that affect the melt composition. Based on the RSM bath volume and nominal feedrate, the bath normally requires at least 5.3 hours of operation and tapping after a feed composition change to purge one volume of the old bath composition. Up to three bath volume changes (approximately 16 hours) may be needed to approach a new glass composition and redox equilibrium.

Thirty-six hours is allowed for the first test condition to enable the melter system to establish some stability after startup and verify or change any conditions that are outside the design operating ranges for this test condition and for system operation.

Eight hours per test condition is allowed for test conditions established by changing the reducing agent when the glass composition and redox state is intended to stay constant. This time is not based on the time needed for three glass volume changes, but is nominally based on time needed to make the feed change, verify how the system responds to the change, make corrective actions if needed, perform continuous off-gas monitoring, and perform off-gas sample collection if needed.

Table 4. Target RSM-2 Operating Conditions

Parameter	Target or Design Parameter
Melt surface area, ft^2	0.2
Melt surface area, cm^2	182
Melt volume, ft^3	0.049
Glass density, lb/ft^3	168
Glass specific gravity	2.6
Blended feedrate, liters/hr (a)	1.5
Blended feedrate, kg/hr (b)	2.0
Blended feedrate, lb/hr (b)	4.4
Feed glassformers, kg/liter	0.45
Glassformer feedrate, kg/hr	0.68
Glassformer feedrate, lb/hr	1.5
Melt rate, $lb/hr/ft^2$	8
Glass melt temperature, ^oC	1,150
Glass melt volume, liters	1.4
Glass melt mass, kg	3.6
Glass melt mass, lb	8.0
Glass residence time, hr	5.3
Glass production rate, $kg/m^2/day$	880
Glass Fe^{+2}/Fe_{tot} ratio	<0.3
Plenum temperature, ^oC	500
Plenum pressure, inches water	-0.5 to -1.5
Air inleakage rate, scfm	~1.0 (estimated)
Total melter off-gas flowrate, liters/minute (d)	173
Total melter off-gas flowrate, scfm (d)	6.1
Plenum residence time, sec	1.6
Scrub solution HNO_3 concentration, M	1

a. 1.5 liters/hr was target for the RSM-1 test.
b. Assuming a feed mixture specific gravity of 1.34.
c. Based on a glass specific gravity of 2.6.
d. Standard conditions: 1 atm, 0^0C

If some conditions such as the generation of a separate molten sulfate salt phase occur, some time may be required to take corrective actions. Such corrective actions could include (a) temporarily increasing the amount of feed reductant or adding another reductant (such as starch) to react with the sulfate to form metal oxides and gaseous sulfur species, or (b) decreasing the amount of sulfur in the feed and allowing time for sulfates to dissolve from the sulfate phase into the lower-sulfate-concentration glass product.

When satisfactory operating conditions are maintained stably, then time, while maintaining those conditions, will be required to obtain glass, off-gas, PM, and secondary waste samples for analysis. Depending on the number of particulate/metals sample trains that are collected, the duration of this sample period may be at least 4 hours.

5. DATA COLLECTION AND SAMPLE COLLECTION/ANALYSIS

The test program includes process monitoring and control, data collection, sample collection, and sample analysis.

5.1 Process Monitoring, Control, and Data Collection

Process monitoring, process control, and data collection is performed primarily by the RSM data acquisition and control system that monitors and controls key system components and electronically logs key RSM data. Process data that are not electronically logged by this system must be recorded manually on operator data sheets.

Table 5 shows process data that are electronically logged by the RSM data logging system and manually logged on RSM operator data sheets. These data document operations of the melter, electrode power, the kiln that encloses the melter, glass tapping, and the off-gas system.

Visual observations of the operating behavior of the feed system and the melter, and off-gas system will be done. Any foaming, corrosion, or salt formation will be noted. Accurate records of feed rate will be made. Any operational problems or potential optimizations will also be recorded.

At the completion of each test, the melter system will be shut down according to PNNL procedures. Any devitrification in the containers of glass produced will be noted. The melter tank, other melter components, and the off-gas system will be examined for any wear, pitting, or corrosion. If operating or glass product characteristics suggest the presence of a separate metal phase, the RSM's melt cavity will be examined for possible accumulations of reduced metals.

Table 5. RSM Process Data that Are Electronically or Manually Logged

Parameter	Units	Range	Electronic log	Manual log
Melt Temperature (T1, control, T2, manual log)	° C	1,125 – 1,175	X	X
Plenum Temperature	° C	400 – 600	X	
Feed pump setting	%		X	X
Cold Cap Coverage	%	> 75	---	X
Slurry Pool Coverage of Cap	%		---	X
Cold Cap Flexibility Visible	y or n		---	X
Number of Vents	#		---	X
Cold Cap Thickness	inch		---	X
Phase Separation Noted in laboratory record book	y or n		---	X
Glass Pouring	y or n		---	X
Electrode Potential	volts		X	X
Electrode Current	amps		X	X
Electrode Power	kW		X	X
(Electrode Power) Output	%		X	X
(Electrode) Control Mode	A or M		X	X

Table 5. RSM Process Data that Are Electronically or Manually Logged (Continued)

Parameter	Units	Range	Electronic log	Manual log
Melt Resistance	Ω		X	X
Melt (Electrode) Setpoint Temp	° C		X	X
Kiln Power	kW		X	X
Kiln Temp Setpoint	° C		X	X
Kiln Actual (Middle) Temp	° C		X	X
Kiln Power Output	%		X	X
Kiln Control Mode	A or M		X	X
Discharge Can Power	KW		X	X
Discharge Can Temp Setpoint	° C		X	X
Discharge Can Actual Temp	° C		X	X
Discharge Can Power Output	%		X	X
Overflow Heater Power	kW		X	X
Overflow Heater Setpoint	° C		X	X
Overflow Heater Power Output	%		X	X
Feed Nozzle Cooling Flow	gpm	reg FNT (0.5)	---	X
Film Cooler Air Flowrate	gcfm	reg OGT (1-10)	---	X
Melter Vacuum-Magnehelic	in. H_2O	0.1 -2.0	---	X
EVS HX Cooling Flow	gpm	reg SLT (1-5)	---	X
EVS Scrub Tank Volume	gallon	35 - 50	---	X
EVS Nozzle Pressure	psi	50 - 55	---	X
EVS Scrub Solution pH	pH	> 9	---	X
Feed Pump (tubing) Condition			---	X
Agitator Setting			---	X
Blower Cooling Flow	gpm	1 - 1.5	---	X
Feed Nozzle Temp (FNT)	° C	< 40	---	X
Off-gas Temp (OGT)	° C	< 250	---	X
Post EVS Off-gas Temp	° C	< 50	---	X
Scrub Liquid Temp (SLT)	° C	<40	---	X
Heat Xfer Temp	° C	< 30	---	X
Feed Pressure	psi	<1.0	---	X
Feed Tank Wt	Kg	decreasing	---	X
Overflow Temp	° C	1,000 - 1,100	---	X
Discharge Can Temp	° C	750 - 850	---	X
Glass Scale	Kg	< 10	---	X
Alarm Condition	On/Off	Off	---	X

5.2 Process Sample Collection and Analysis

Process samples that will be collected for analysis include the feed slurry, glass product, accretions of material on the inside of the melter plenum or off-gas pipe, scrubber solution (including undissolved solids (UDS), and SCR catalyst. Table 6 shows these sample matrices, frequency of sample collection, and planned analyses. In general, process samples (except for plenum or off-gas pipe samples, which cannot be collected until those areas are physically accessed either at the end of the test series or during other shut down conditions) will be collected at least for every identified "stable" operating condition that represents a condition that satisfies the test objectives. Sample analyses will be done to determine elemental compositions for mass balances and for determining properties of those melter product streams.

Table 6. Process Sample Collection and Analysis for the RSM-2 Test Series

Sample Matrix	Size	Frequency	Number of samples	Analyses	Lab
Feed slurry blend	≥200 g	Once per distinct feed blend.	7	Elemental (a) (7)	PNNL
				Density (7)	RSM-2 staff (b)
				Loss on drying (7)	PNNL
				Loss on ignition (7)	
Glass product	≥200 g	Every 2-hours or whenever the melter pours, which ever is less frequent. Stimulated pours may not be possible. Split selected samples for leachability analysis.	10-20+	Elemental (a) (7)	
				Fe ratio (10-20+)	RSM-2 staff
				Fe ratio (10-20+) by wet chemistry	PNNL
				Density (7 analyses)	
			7	PCT leachability (7)	INEEL
				TCLP leachability (7)	
Plenum or off-gas pipe accretions	≥200 g	At end of test series or when system is shut down.	3	Elemental (a) (3)	PNNL
Scrubber solution with dissolved solids	≥200 ml per sample	Scrub solution will be collected at the conclusion of every test condition considered to be stable and satisfactory of test objectives. The total scrubber solution will be collected. Its volume will be measured. UDS will be allowed to settle. Supernatant will be decanted from UDS into two samples; remainder will be discarded. An aliquot will be titrated to determine HNO_3 content. UDS will be slurried and split into two samples.	7 archived @ PNNL; 7 analyzed	Total volume (7)	RSM-2 staff
				TDS (7)	INEEL
				TDS composition (a) (7)	
				Density (7)	RSM-2 staff
				Acidity (7)	
UDS from scrubber solution	≥200 ml		7 archived @ PNNL; 7 analyzed	UDS composition (a) (7)	INEEL
				Total UDS (7)	
				PSD (7)	
Total samples and analyses			55-65+	135-155+	

a. Elemental analysis includes 25 elements – 7 RCRA metals (Ag, Ba, Cd, Cr, Hg, Pb, Se), 11 glass formers (Al, B, Ca, Fe, K, Li, Mg, Mn, Na, Ni, Si, and Zn), 1 nonradioactive surrogate of radioactive elements (Cs), 3 halogens (Cl, F, and I), and P, S, and C. Other components of the feed (Cu, Gd, Mo, Ru, Ti, and Zr) are not present in the feed at high enough concentrations to be detectable in the feed or the glass. As, a RCRA metal, was not added to the feed and so is not included in the analysis list.

b. Analyses performed on-site during testing will be done by RSM-2 staff on shift or personnel assigned for sample collection during test operation. For example, some of the INEEL personnel are assigned primary responsibility, when they are not on shift, for sample collection and analysis.

Glass-sample analysis will include not only the elemental composition, but also leachability, Fe ratio, and density. Leachability will be measured using both the Product Consistency Test (PCT), ASTM C-1285-94 (ASTM 1994), and the Toxicity Characteristic Leaching Procedure (TCLP) per EPA Method 1311. The PCT measurement is the standard test method used for determining the durability of high-level waste glasses in the United States, and is a criterion that will be used to determine how the product glass of the actual waste will meet regulatory requirements for high-level waste disposal. TCLP analyses will be done to determine how the hazardous metals in the feed (such as Cd, Cr, and Pb) will be stabilized to meet RCRA disposal requirements.

While feed, glass, scrubber solution, and off-gas pipe accretion samples will be collected according to the frequency indicated in Table 6, only some of those samples, which are considered to be most representative of selected test conditions, will be analyzed. Furthermore, only some of the above-listed analyses may be done on some of those selected samples. For example, some of the glass samples that are analyzed for elemental composition may not be analyzed for leachability or other properties.

5.3 Off-gas Sample Collection and Analysis

Off-gas sampling and analysis will include online, practically online, and offline measurements. True online measurements include off-gas temperature and NO, NO_2, SO_2, and total hydrocarbon (THC) measurements. Measurements of the off-gas flowrate and composition using gas chromatography and mass spectroscopy are not true online measurements. These measurements include continuous or discrete sample gas extraction, and discrete injection of sampled gas into the gas chromatograph (GC) or the mass spectrometer (MS). Analytical GC and MS results are typically available within minutes (≤ 75 s) of sample collection, and so are still as useful as online measurements.

Figure 1 and Table 7 show the off-gas and particulate/metals sampling and analysis activities for this test series. Off-gas measurements will be performed at two locations—the film cooler (FC) outlet and the high efficiency mist eliminator (HEME) outlet. The FC outlet measurements, when corrected for dilution from the film cooler gas, will best represent the actual uncontrolled melter emissions for melter mass balance evaluations and off-gas system design. The HEME outlet location is useful for characterizing the performance of the scrubber system.

5.3.1 Continuous Emissions Monitoring

The CEMs are configured as shown in Figure 2. GC, $NO/NO_2/NO_x$, THC, and SO_2 CEMs will monitor the film cooler outlet off-gas. This system will be configured so that it can also be used to sample off-gas at the HEME outlet location. The single mercury monitor will time-share monitoring for Hg and $HgCl_2$ at the film cooler outlet and the HEME outlet locations, independently from where the other CEMs are sampling.

The CEMs are summarized in Table 8. The Microtechnologies Incorporated (MTI) gas chromatograph is calibrated for the specified species. The GC measurements are available only when the GC sample system and GC are online. The GC has two separate columns and detectors. He is a carrier gas for one column/detector for measuring CO_2 and N_2O. Ar is the carrier gas for the other column/detector for measuring H_2, He, N_2, CO, NO, and O_2.

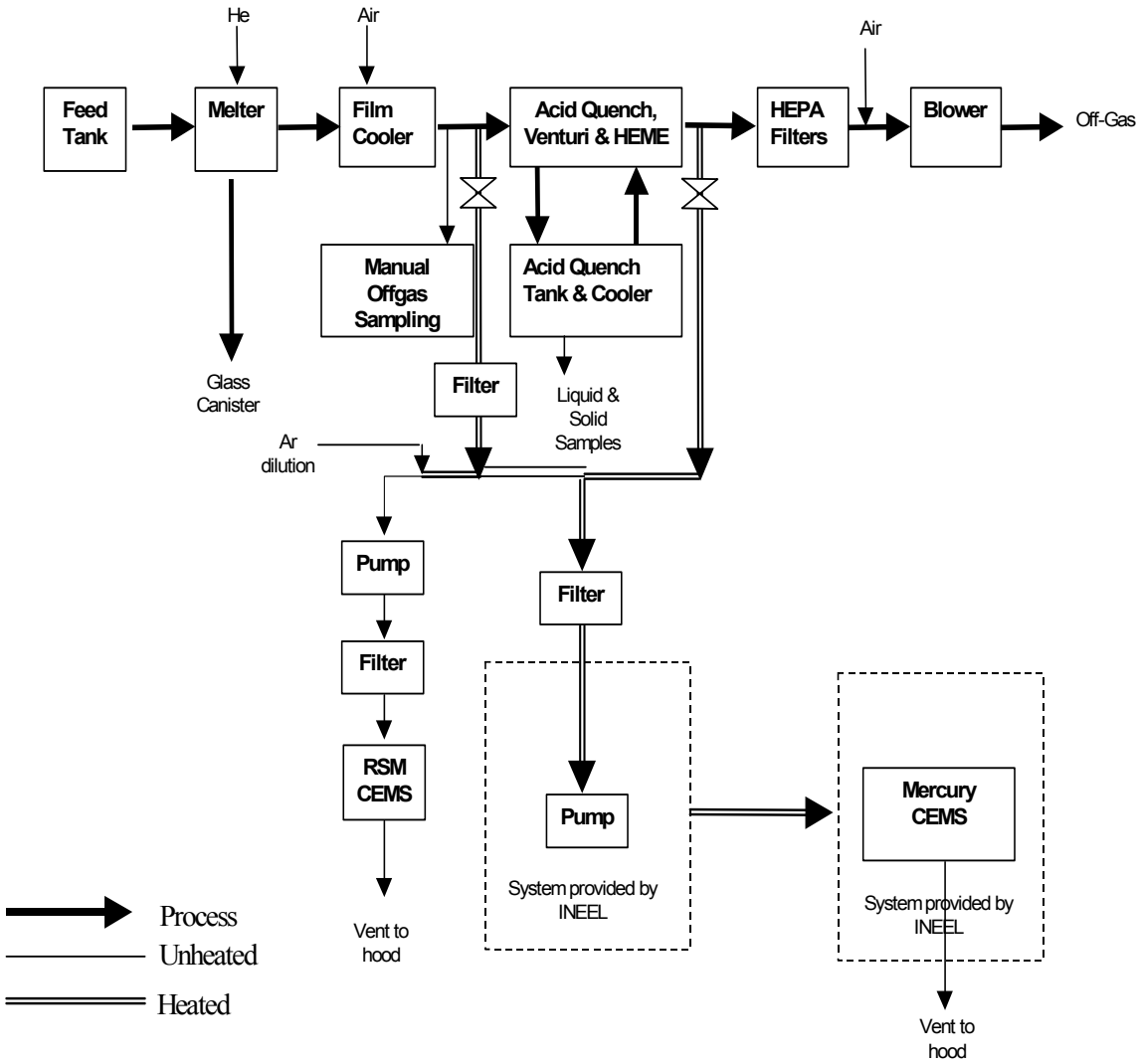

Figure 1. RSM-2 Off-gas Sampling and Analysis Scheme

The Stanford Research System quadrupole mass spectrometer is a general analytical tool that can quantitatively and qualitatively measure certain gaseous species with molecular masses up to 300 atomic mass units (AMU). Species such as CO and N_2 (with molecular weight of 28) and CO_2 and N_2O (with molecular weight of 44) are not easily discriminated using the MS results, so these results must be interpreted carefully using other measurements or process knowledge. The MS is further limited because of limited sensitivity for some species, with background noise in the ppm concentration range. The MS can be calibrated for quantitative analysis based on height of peaks that are qualitatively identified based on molecular weight. To the extent that the MS analysis can provide measurements of sulfur species SO_2, H_2S, COS, and CS_2, Cl species HCl and Cl_2, F species HF and F_2, and HNO_3, these will be reported. However, RSM-1 results indicate that levels of most species such as HNO_3, SO_2, and VOCs are present at levels below the MS detection/interpretation limit.

Table 7. Melter Off-gas Sample Collection and Analysis for the RSM January 2001 Test Series

Off-gas Measurement	Location		Instrument/sample no.			Comments
	FC out	SCR out	GC	MS (a)	Other	
Off-gas flowrate/Velocity	X	X			He balance @ FC outlet;	
% sample gas dilution	---	---			Ar in the dilution gas supply as a tracer; Flowmeter measurement of sample gas and dilution gas flowrates	
O_2	X		X	X		
CO_2	X		X			
CO	X		X			
Ar	---	---	X			
He	X		X			
N_2	X		X			
NO	X	X	X		Chemiluminesce @ FC out: Chemiluminesce @ HEME out	
NO_2	X	X				
N_2O	X		X			Which column/detector?
HNO_3	X			X		Sampling temp below BP/moisture reactive
Total SO_2 and SO_3	X				5 nonisokinetic Method 8	
COS						
CS_2						Sampling temp below BP
H_2S						
SO_3						Sampling temp below BP
Total S species	X				CAE pulsed fluorescence	Use tube furnace to oxidize reduced S species to SO_2
THC	X				THA/FID	
CH_4	X			X		
VOCs	X			X		Specific species need to be identified.
H_2	X		X			
PM					5 nonisokinetic M0050 trains	
HCl	X			X		
Cl_2	X			X		
Nitrates	X					
Nitrites	X					
HF	X			X		
F_2	X			X		
Total F	X				5 nonisokinetic M0050 trains	
HI	X			X		
I_2	X			X		
Total I	X				5 nonisokinetic M0050 trains	
Hg	X				INEEL CEM	
$HgCl_2$	X					
Metals incl. Hg and $HgCl_2$ (b)	X				5 Single-point, isokinetic M0080 sample trains	

Acronyms:
FC = Film cooler
GC = gas chromatograph
MS = Mass spectrometer
CAE = Clean Air Engineering
TECO = Thermo Electron Co.
THA = Total Hydrocarbon analyzer
FID = Flame ionization detection

a. RSM-1 results indicate that levels of most species such as HNO_3, SO_2, and VOCs are present at levels below the MS detection/interpretation limit.

b. Elemental analysis includes 28 multiple metals are 6 RCRA metals (Ag, Ba, Cd, Cr, Hg, Pb), 19 glass formers (Al, B, Ca, Cu, Fe, Gd, K, Li, Mg, Mn, Na, Ni, Pb, Ru, Si, Ti, V, Zn, Zr), 1 nonradioactive surrogate of radioactive elements (Cs), P, and S. RCRA metals As and Se were not added to the feed and so are not included in the analysis list.

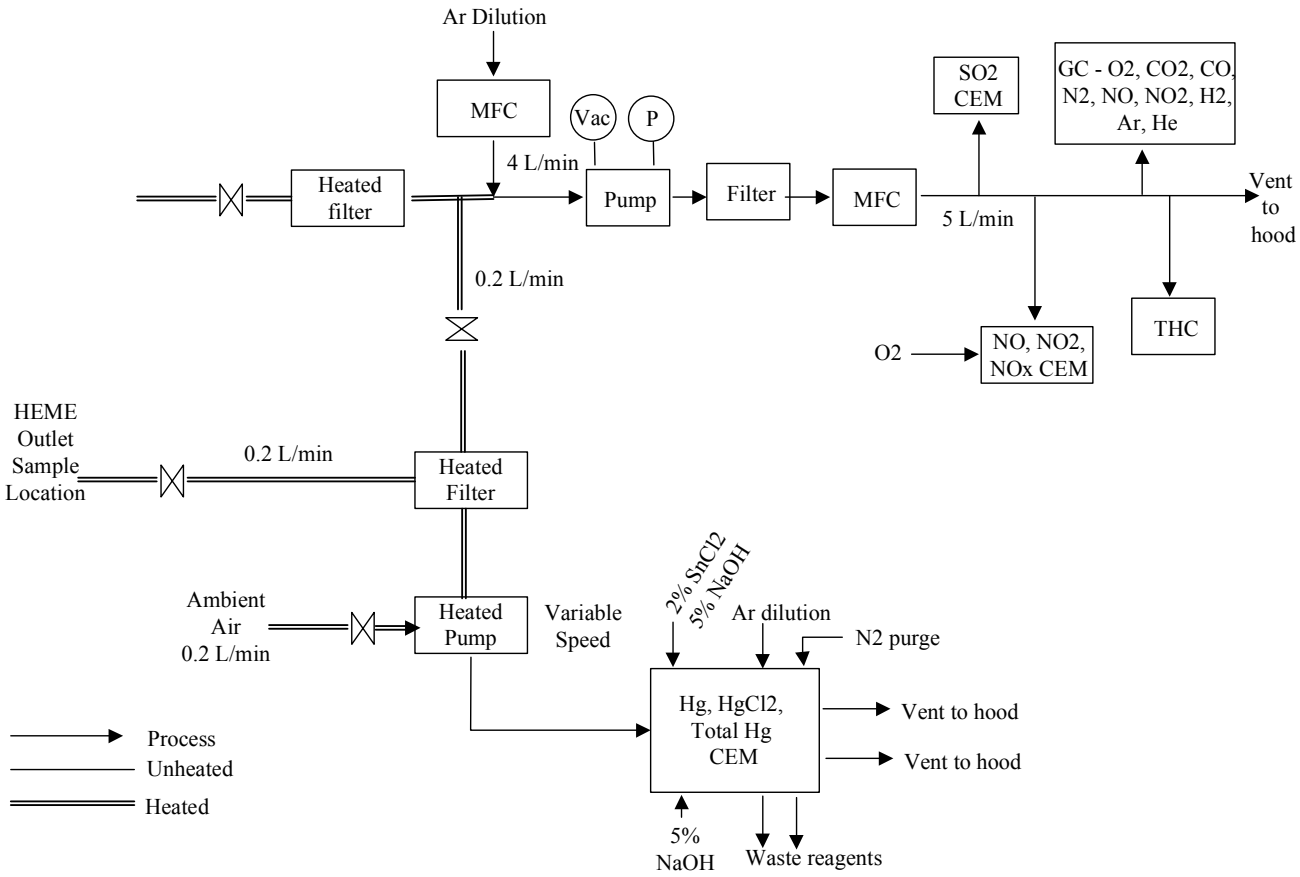

Figure 2. Sample Conditioning and Analysis Scheme for the RMS-2 CEMS

The mercury analyzer (Figure 3) is a PS Analytical Sir Gallahad unit capable of measuring total Hg and elemental Hg. Oxidized Hg is determined by difference. This unit operates semicontinuously in 5-minute cycles, alternating between the total Hg and the Hg^o modes. In each cycle, a 1-minute average data point for either total Hg or Hg^o is determined. Sample gas is withdrawn continuously through a heated sample line by a heated vacuum pump. The sample gas is split evenly. One stream is continuously passed through an impinger containing 2-w/v % $SnCl_2$/ 5 w/v % NaOH, where oxidized forms of mercury are reduced to Hg^o. The other stream flows continuously through an impinger containing 5-w/v % NaOH, where oxidized forms of Hg are scrubbed from the gas into the impinger solution.

In the first cycle, the sample gas from the $SnCl_2$/NaOH impinger is passed into a cold-wall condenser to remove water vapor and through a bed of gold-coated sand where the total Hg^o is sorbed. This is the collection cycle for the total Hg measurement. The sorbent is then heated and purged with N_2 to release the total Hg into an atomic fluorescence measurement cell. This is the heating/measurement cycle for total Hg. All forms of Hg in the sample gas are reduced to Hg^o in the $SnCl_2$/NaOH impinger and detected in this cycle.

In the next cycle, the sample gas from the NaOH impinger is passed through the cold wall condenser and into the gold-coated sand adsorber. Since the NaOH impinger removes oxidized Hg species from the sample gas, only Hg^o is collected in the gold sorbent. When thermally desorbed into the measurement cell, only Hg^o is detected.

Table 8. CEMs Used in the RSM-2 Test Series

Species	Procedure	Instrument/ Detection Principle	Detection Limit	Sample Conditioning
O_2, CO_2, CO, NO, NO_2, Ar, H_2, He	Vendor/ PNNL procedure	MTI GC with 2 columns/detectors, He carrier gas in one of the columns Ar carrier gas in other.	1 ppm, depending on dilution factor	Continuous filtered sample gas extraction followed by sample dilution; injection of discrete <1 mL sample aliquots into GC
NO, NO_2, NO_x	EPA 7E	Rosemount chemiluminescence	~1 ppm	Continuous filtered sample gas extraction followed by sample dilution.
THC	EPA 25A	Flame ionization detection (FID)	~1 ppm	Continuous filtered sample gas extraction followed by sample dilution.
Gaseous species <300 molec-ular weight	Vendor/ PNNL procedure	quadrupole mass spectrometer	~1,000 ppm	Continuous filtered sample gas followed by sample dilution; continuous, direct injection of <1-mL/m sample stream aliquots into MS. Qualitatively detect species based on molecular weight; quantify detected species based on calibrated peak height; process knowledge or other analyses must be used to discriminate between species with similar molecular weight.
Total SO_2	EPA 8C	Pulsed fluorescence	10 ppm	The EPA 8C procedure is modified to use a tube furnace to oxidize reduced S species to SO_2; the tube furnace may be bypassed to speciate SO_2 from total S species
Hg, $HgCl_2$	Vendor/ INEEL procedure	Atomic fluorescence		Continuous filtered sample gas extraction followed by sample dilution, scrubbing, and moisture removal.

The concentration of oxidized mercury is determined by difference. The NaOH in the impingers is used remove NO_2 from the test gas in order to maintain reducing conditions in the impingers and to protect the mercury analyzer. The impingers are continually supplied with solution by a Masterflex-type pump.

The instrument alternates between measuring total and elemental mercury and displays the data in $\mu g/m^3$ in the form of a trend graph, and stores the data in an Excel file. During every 10-minute period, the instrument reports a single 1-minute average total Hg value, a single 1-minute average Hg^o value, and total oxidized Hg, determined by difference between the two other values. The two 1-minute average values are measured 5-minutes apart, so the reported value for oxidized Hg is not a true difference of simultaneous total Hg and Hg^o values.

The mercury analyzer is calibrated using a mercury vapor injection system supplied by the vendor. This system consisted of a vessel containing liquid mercury from which measured volumes of mercury-saturated air, at a measured temperature, are withdrawn by hypodermic syringe and injected into an N_2 carrier gas by means of a sample port. The analyzer software calculates the mass of mercury injected, which is converted to a mercury concentration by using both the measured carrier gas flowrate and the time interval used for sampling.

Prior to sampling the melter off-gas, room air will be sampled into the mercury analyzer to set sampling flowrates and establish a baseline. A manual data sheet will be used for recording sampling flowrates, condenser temperatures, and observations and comments.

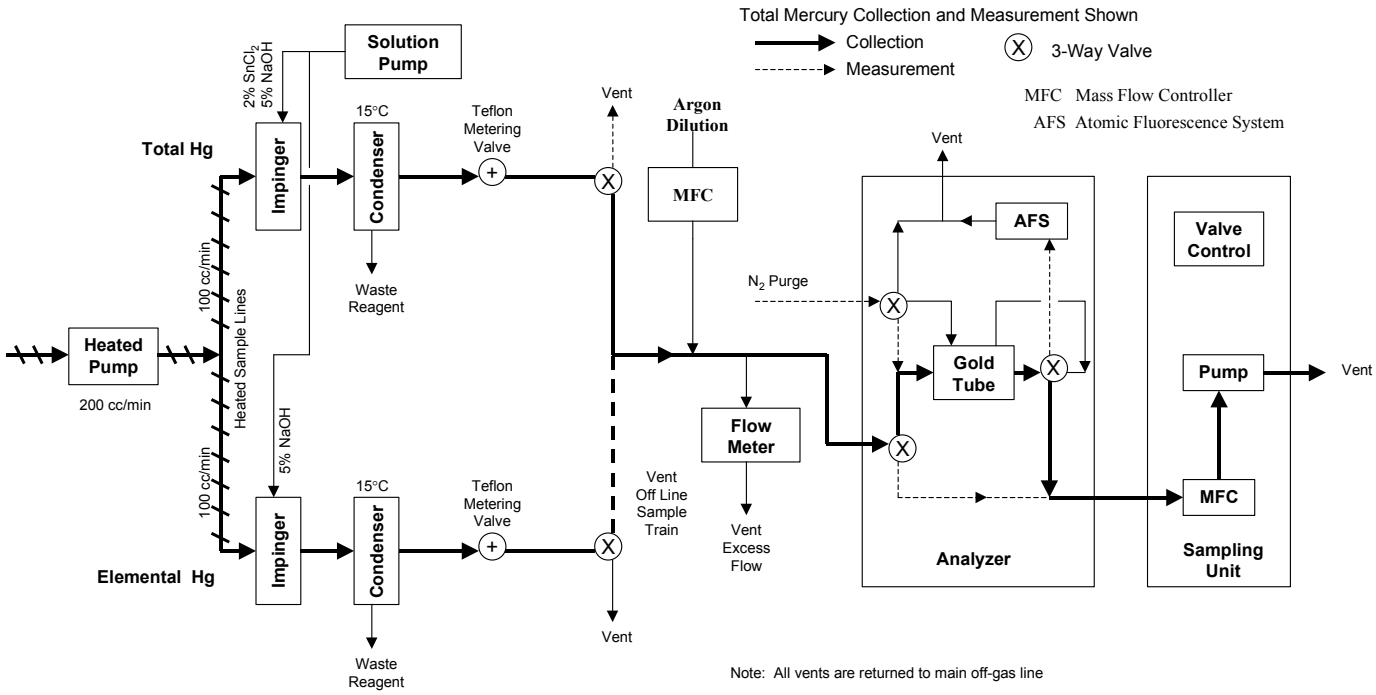

Figure 3. Mercury Monitor Flow and Analysis Schematic

5.3.2 Manual Off-gas Sampling and Analysis

Manual off-gas sampling and analysis will be performed to provide off-gas emissions data in addition to that obtained from the CEMS. The scope of manual off-gas sampling and analysis is shown in Table 9. All manual off-gas sampling will be performed at the film cooler outlet location in order to best characterize the melter outlet emissions upstream of the off-gas system.

5.4 Sample Analysis Procedures and Equipment

Process and off-gas samples will be analyzed, as applicable, for elemental composition, species composition (for gaseous effluents), leachability, density, particle size, viscosity, and oxidation state (as discussed Sect. 5.2). Multiple analyses of various different sample matrices will be used. Some analyses are online during test operation, while others incur delays of a few minutes to several hours or days, depending on the kind of analysis, sample preparation required prior to analysis, and location of the analytical equipment. Table 10 briefly describes the different analyses that will be included in this test program.

Table 9. Manual Off-gas Sampling and Analysis

Sample train procedure (a)	Measure-ment	Number of:			Analytical procedures
		Trains	Samples per train and matrices	Total samples/total analyses	
0050 modified for PM and PSD	HCl, Cl_2, PM, F, I, nitrates, nitrites, PSD	5 + 1 blank	4 per train: 1. AR – acetone rinse 2. Filter – glass fiber filter 3. H_2SO_4 – 0.1 N liquid 4. NaOH – 1 N NaOH	24 samples, 36 analyses: PM: 6 AR, 6 Filters PSD: 6 AR, 6 Filters Anions (b): 6 H_2SO_4, 6 NaOH	9056/9057 (IC for HCl, Cl_2, F, nitrates, nitrites); Method 5 (gravimetric for PM); SEM for PSD (visually count particles and estimate size in SEM fields magnified up to 10,000x).
0080	Multiple metals including Hg (c)	5 + 1 blank	6 per train: 1. FH – Front half 0.1 N HNO_3 2. Filter – glass fiber filter 3. BH – 5% HNO_3/3% H_2O_2 4. MT – 0.1 N HNO_3 5. $KMnO_4$ – 4% $KMnO_4$/ 10% H_2SO_4) 6. HCl – 8 N HCl	36 samples, 42 analyses: Metals: 12 (1 combined FH and filter, 1 BH per train) Hg: 30 (1 combined FH and Filter, 1 BH, 1 MT, 1 $KMnO_4$, and 1 HCl per train)	6010B (ICP) for multiple metals, 7470 (CVAAS) for mercury
8 modified for SO_3 measurement	SO_2 and SO_3	5 + 1 blank	2 per train: IPA – 80% isopropyl alcohol H_2O_2 – 3% H_2O_2	12 samples, 12 analyses: SO_3: 6 IPA SO_2: 6 H_2O_2	Method 5 (barium-thorin titration), modified by recovering the IPA impinger for SO_3 analysis.
Totals		13 +3 blanks	---	72 samples: 90 analyses	

Acronyms:
AR = Probe and filter front half acetone rinse
ICP = Inductively-coupled argon plasma spectrometry
CVAAS = Cold vapor atomic absorption spectrometry
PM = Particulate matter
IC = Ion chromatography
PSD = particle size distribution
SEM = Scanning electron micrograph
IPA = Isopropyl alcohol
FH = Front half, acid rinse of the probe and filter front half
BH = Back half, nitric acid impingers
MT = Empty impinger, after the nitric acid impingers

a. Isokinetic single-point, glass-lined probe, heated filter, impingers that contain sorbent solutions.

b. Anions = Cl^- (represents HCl in the H_2SO_4, and Cl_2 in the NaOH samples), F^-, I^-, nitrate, nitrite, and phosphate.

c. 28 multiple metals are 6 RCRA metals (Ag, Ba, Cd, Cr, Hg, Pb), 19 glass formers (Al, B, Ca, Cu, Fe, Gd, K, Li, Mg, Mn, Na, Ni, Pb, Ru, Si, Ti, V, Zn, Zr), 1 nonradioactive surrogate of radioactive elements (Cs), P, and S.

Table 10. RSM-2 Sample Analysis Methods for the Process Samples

Analysis	Sample matrix	Analysis Method	Detection limits	Sample preparation (solids)	Analysis Description
Cations	Solid or liquid	EPA 6010B (ICP) for most metals; AAS for Cs, Na, and K at INEEL; ICP-MS for Cs at PNNL; 7470A (CVAAS) for Hg. (at INEEL: ALD 2900 for 6010B (ICP-AES) and also AAS, ALD 2800 for 7470A CVAAS for Hg. (at PNNL: AIAL-01 for ICP-OES for most metals; PNL-ALO-280 for ICP-MS for Cs	Variable	NaOH, Na peroxide, or KOH fusion followed by total digestion; or avoid fusion by digestion with HF if needed (EPA 3010, 3015, or 3051), preferred to avoid loss of Hg. Hg digestion per 7470A (liquids) and 7470B (liquids)	Analysis of total amount of element, regardless of speciation
			Variable	Li-tetraborate fusion followed by total digestion or avoid fusion by digestion with HF if needed (EPA 3010, 3015, or 3051), preferred to avoid loss of Hg	
Anions		IC	≥1ppm	Total digestion (of solids) per EPA 3050 followed by analysis per 9056	Cl^-, F^-, I^-, nitrate, nitrite, sulfate, and phosphate
TOC		TOC	TBD		Combustion, CO_2 detection
LOI		PNL-ALO-236 reference to CRC handbook of Chemistry and Physics, 67th Edition			Heat in air atmosphere to 110°C for moisture, 305°C for water of hydration, 505°C for $MgCO_3$, and 905°C for $CaCO_3$, weigh residue each time.
Fe ratio	Glass	Fe ratio probe	0.05		
		Wet chemistry analysis	0.05		
Leacha-bility		TCLP (EPA Method 1311, at ALD: ALD 7998 for TCLP prep, followed by ALD 2800 (for Hg) and ALD 2900 (for other metals), PCT (ASTM C-1285-94)	---	Crush and size-segregate	
Density	Solid	---	---		Measure displacement of water by a known mass
% H_2O	Liquid	EPA Method 5; PNL-ALO-236	---		Dry at 100°C to dryness, desiccate, weigh to constant weight, subtract residue mass from total
TDS		EPA 160.1 (ALD 7975 at INEEL)			Dry at 100°C to dryness, desiccate, weigh to constant weight
Density		---			Measure mass of known volume in tared container
Acidity		Titration to a pH endpoint			Titrate with NaOH to a desired pH endpoint, measure the volume and molarity of NaOH.
Mass	UDS	EPA 160.2 (ALD 7972 at INEEL)			Filter, dry at 100°C to dryness, desiccate, weigh to constant weight
PSD					Wet coulter counter

AAS	Atomic absorption spectroscopy	LOI	Loss on ignition
AES	Atomic emission spectroscopy	OES	Optical emission spectroscopy
AIAL	Advanced Inorganic Analysis Laboratory at PNNL	PCT	Product Consistency Test
ALD	Analytical Laboratory Department at the INEEL	PSD	Particle size distribution
ALO	Analytical Laboratory Operations at PNNL	TCLP	Toxicity Characteristic Leachability Test
CVAAS	Cold vapor atomic absorption spectroscopy	TDS	Total dissolved solids
IC	Ion chromatography	TOC	Total organic carbon
ICP	Inductively-coupled argon plasma	UDS	Undissolved solids

6 SURROGATE SBW AND FEED MIXTURE

The feed mixture includes surrogate SBW in the specified waste loading compared to added glass-forming frit and added reductant.

6.1 SBW Description

Liquid SBW is a waste product of past spent nuclear fuel reprocessing activities. The waste is a highly acidic (~1 molar nitric acid) aqueous solution of sodium nitrate containing significant amounts of aluminum, potassium, some toxic metals, and radionuclides. Due to its relatively high concentration of sodium, this waste is referred to as SBW. This waste contains not only dissolved matter, but also undissolved solids that have generally settled and formed solid or sludge-like tank heels. At present, the mass and composition of the tank heels are not included in target composition of the RSM tests.

Added to this waste is also some liquid high-level waste that has been recycled back to the tank farm from processing activities, such as calcination, and other wastes generated from decontamination and solvent recovery efforts. This waste is a mixed waste, containing not only radionuclides, but also RCRA hazardous characteristics (corrosivity and hazardous metals) and listed wastes (organic solvents). The actual concentrations of any organics is not well known, but is expected to be low (under 1 weight %).

The surrogate SBW for the RSM tests needs to simulate the actual waste as closely as reasonably possible. Instructions for blending the surrogate SBW are shown in Appendix A. Table 11 summarizes the target composition of the surrogate SBW, based on the instructions in Appendix A.

The surrogate SBW will not contain any radionuclides. Surrogates of some of the actual radionuclides will be used if non-radioactive isotopes of the radionuclides do not exist. Waste radionuclides and those that will be represented by stable isotopes or chemical surrogates are summarized in Table 12. Some elements that were not detected in the SBW were added to the RSM-2 surrogate waste, if they are used for chemically similar surrogates for radionuclides. The amounts of some of these surrogates, and stable isotopes of radionuclides, are in some cases increased to levels in the surrogate waste that are higher than found in the SBW, in order to enable measurement in the glass and off-gas system at levels above their detection limits for the analytical methods used for sample analysis.

Some elements thought to be in the SBW are excluded from the RSM-2 surrogate feed mixture. These elements are noted in Table 11. These elements were excluded if they are present in low concentrations in the SBW, are not used in the test as radionuclide surrogates, are not expected to impact the glass product characteristics, are not of concern in melter system mass balances, are not expected to impact melter or off-gas system performance, and are not expected to impact the characteristics or fate of secondary waste streams.

6.2 Glass Formulation

The product glass needs to meet regulatory limits for high-level waste borosilicate glass. The target glass for SBW vitrification is an iron-enriched borosilicate glass. The target composition for this glass is shown in Table 13.

Table 11. INTEC Tank WM-180 Waste and Simulant Compositions

Constituent	Reagent used to make simulant	Constituent concentration in WM 180 SBW, mole/liter (a)	Included at the indicated level in RSM-2 Simulant	Comments
Non-radioactive Metals				
Aluminum	$Al(NO_3)_3*9H_2O$	6.63E-1	Y	---
Arsenic	As_2O_3	4.99E-4	**N**	Chem hazard, no impact on glass product
Barium	$Ba(NO_3)_2$	5.58E-4	Y	---
Beryllium	BeF_2	7.77E-6	**N**	Chem hazard no impact on glass product
Boron	H_3BO_3	4.53E-4	Y	---
Cadmium	$Cd(NO_3)_2*4H_2O$	7.54E-4	Y	---
Calcium	$Ca(NO_3)_2*4H_2O$	4.72E-2	Y	---
Chromium	$Cr(NO_3)_3*5H_2O$	3.53E-3	Y	---
Copper	$Cu(NO_3)_2*3H_2O$	6.97E-4	Y	---
Gadolinium	$Gd(NO_3)_3*5H_2O$	1.77E-4	Y	---
Iron	$Fe(NO_3)_3*9H_2O$	2.17E-2	Y	---
Lead	$Pb(NO_3)_2$	1.31E-3	Y	---
Lithium	$LiNO_3$	3.39E-4	Y	---
Magnesium	$Mg(NO_3)_2*6H_2O$	1.20E-2	Y	---
Manganese	$Mn(NO_3)_2$	1.41E-2	Y	---
Mercury	$Hg(NO_3)_2*H_2O$	2.02E-3	Y	---
Molybdenum	Mo in HNO_3	1.93E-4	Y	H_2MoO_4 used?
Nickel	$Ni(NO_3)_2*6H_2O$	1.47E-3	Y	---
Potassium	KNO_3	1.96E-1	Y	---
Ruthenium	$Ru(NO)(NO_3)_3$	1.25E-4	Y	---
Sodium	$NaNO_3$	2.06	Y	---
Titanium	$TiCl_4$	5.78E-5	Y	---
Uranium	$UO2(NO_3)_2*6H_2O$	3.36E-4	**N**	No available nonradioactive surrogate
Zinc	$Zn(NO_3)_2*6H_2O$	1.05E-3	Y	---
Zirconium	ZrF_4	6.63E-5	Y	---
Non-Radioactive Anions				
Cl	HCl	3.00E-2	Y	---
F	HBF_4	1.19E-2	Y	---
Nitrate	HNO_3	5.27	Y	---
Phosphate	H_3PO_4	1.37E-2	Y	---
Sulfate	H_2SO_4	6.98E-2	Y	---

a. From Christian, J., personal communication to J. McCray, "Simulant Makeup Spreadsheet for Report," June 2001.

Table 12. Nonradioactive Surrogates Used Instead of Radionuclides in the RSM-2 Test Series

Radio-Nuclide	Stable Surrogate	Reagent used to make simulant included in RSM-2	Constituent concentration in WM 180 SBW, mole/liter (a)	Included in RSM-2	Comments
^{3}H	H	H_2O	56	Y	
^{60}Co	Co	$Co(NO_3)_2*6H_2O$	1.93E-5	**N**	Excluded, because the level would need to increase to 2.03E-2 (~1,000x higher than Co in WM-180 waste) to be detectable.
^{90}Sr	Sr	$Sr(NO_3)_2$	6.76E-03	Y	Sr level was increased to 6.76E-3 (~60x) to meet detection limits.
^{99}Tc	Re	Re in 5% HNO_3	5.92E-6	Y (during test condition 10 only)	Re is a surrogate for ^{90}Tc. The Re concentration was increased to 1.59E-3 (~300x higher than Tc in the WM-180 waste) for Re to be detectable. Re will be added in only a portion of the test (test condition 10) to evaluate affect on Cs partitioning.
^{129}I	I	KI	1.30E-4	Y	
^{134}Cs ^{137}Cs	Cs	$CsNO_3$	7.73E-6	Y	The Cs concentration was increased ~300x to 2.22E-3 for Cs to be detectable.
^{154}Eu	Eu	$Eu(NO_3)_3*6H_2O$	3.90E-03	**N**	Excluded, because the Eu level would need to increase to 3.90E-3 (~100,000x higher than Eu in WM-180 waste) to be detectable.
^{234}U ^{235}U ^{236}U ^{238}U ^{237}Pu ^{238}Pu ^{239}Pu	Ce	$Ce(NO_3)_3*6H_2O$	3.36E-4 (total U)	**N**	Ce is a surrogate for U and Pu. Ce was excluded because partitioning data for U and Pu is not needed from RSM-2 test, and because the Ce level would need to be increased about 7x for Ce to be detectable in the glass.
^{241}Am	Nd	$Nd(NO_3)_3*6H_2O$	1.08E-7	N	Nd is a surrogate for Am. Excluded, because the Nd level would need to increase to about 3.90E-3 (~40,000x higher than Am in WM-180 waste) to be detectable.

Table 13. Target Glass Composition (20% SBW)

Oxide	Wt%
SiO_2	54.4
Na_2O	13.8
Al_2O_3	5.5
B_2O_3	4.9
Li_2O	4.9
CaO	4.5
V_2O_5	3.9
ZrO_2	2.0
K_2O	1.5
Fe_2O_3	1.5
All Others	9.8

6.3 Feed Mixture Description

The feed mixture includes surrogate SBW in the specified waste loading compared to added glass-forming frit and added reductant (sucrose). In order to satisfy the test objectives, separate batches of feed mixtures will need to be prepared to provide feed mixtures of different compositions to perform the target test matrix shown in Table 2.

The maximum reductant concentration is limited by the oxidation state of the product glass. Excess reductant will tend to reduce not only nitrates but also glass oxides. The oxidation state is indicated by the Fe^{+2}/Fe_{tot} ratio, which should be maintained under 0.3. The maximum concentration for sucrose, based on previous melter tests, is 170 g of sugar per liter of surrogate SBW. This maximum value is about 78% of the amount needed to stoichiometrically react C with nitrates to reduce the nitrates to N_2.

The composition of the feed mixture will not be based on sample collection and analysis of the feed mixture. Instead, the feed mixture composition will be based on calculated weighted averages of compositions of the separate feed components.

6.4 Glass-Forming Additive Composition

Table 14 shows the composition of the glass-forming chemicals used as a feed additive to produce a regulatorily acceptable product glass.

Table 14. Frit Composition for the RSM-2 Test Series

Frit	Oxide		Glass Former		Mass Ratio
Oxide	**Wt%**	**MW**	**Chemical**	**MW**	**(g-Chem /g-frit)**
B_2O_3	6.03	69.6	H_3BO_3	61.8	0.11
Fe_2O_3	1.52	159.7	Fe_2O_3	160	0.02
Li_2O	6.11	29.9	$LiOH*H_2O$	42.0	0.17
SiO_2	67.95	60.1	SiO_2	60.1	0.68
CaO	5.02	56.1	$Ca(OH)_2$	74.1	0.07
MgO	1.75	40.3	$Mg(OH)_2$	58.3	0.03
Na_2O	4.29	61.98	NaOH	40.0	0.06
V_2O_5	4.88	181.9	V_2O_5	181.9	0.05
ZrO_2	2.44	123.2	ZrO_2	123.2	0.02

7. RSM FACILITY DESCRIPTION

The Research Scale Melter (RSM) facility is located in the Applied Process Engineering Laboratory (APEL) building in Richland, Washington. Figure 4 shows the RSM system. Table 15 shows RSM dimensions and other operational features.

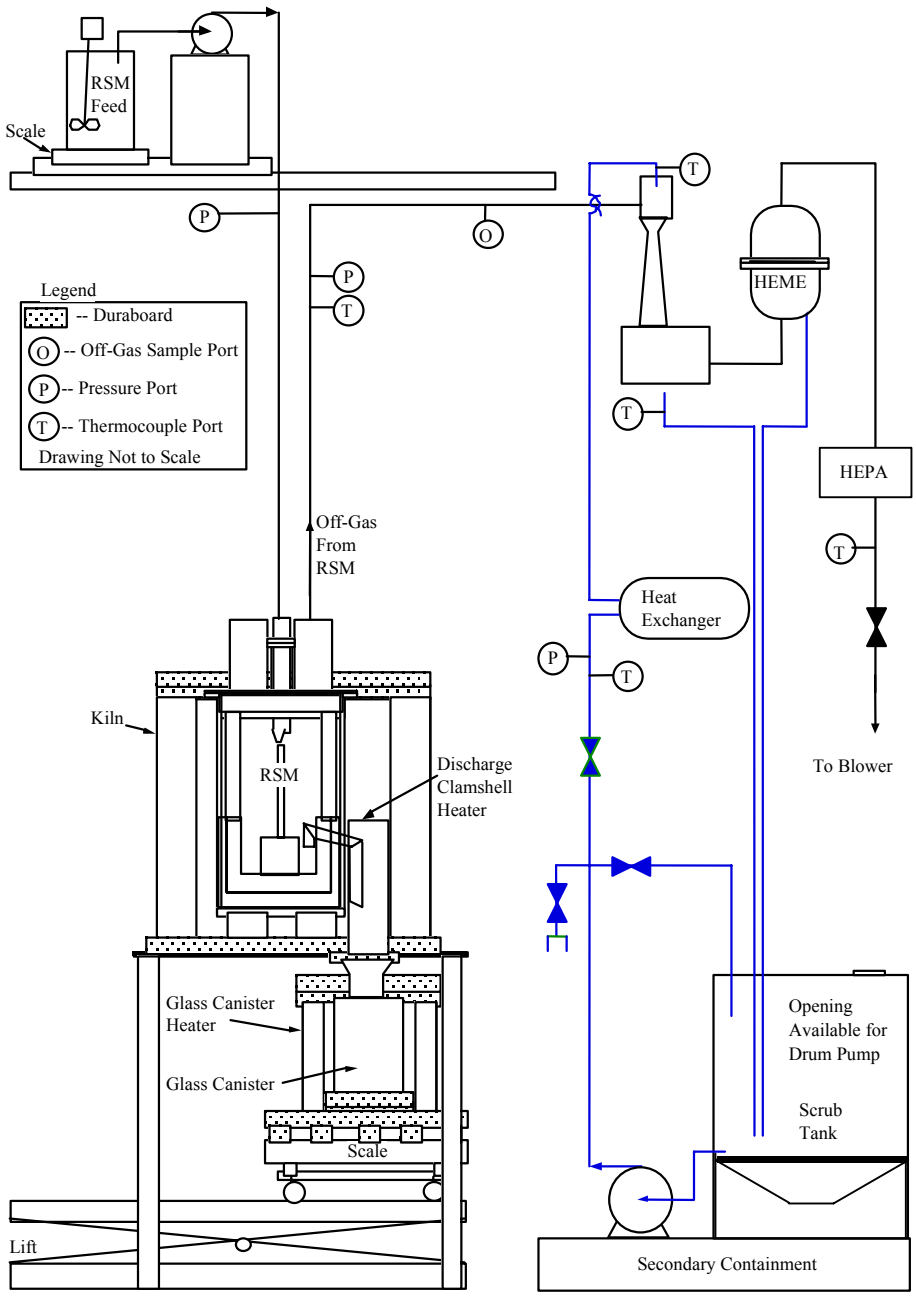

Figure 4. Research-Scale Melter Test Apparatus
(*Not shown is a HEPA filter that was installed for this test downstream of the HEME*)

Table 15. RSM Dimensions and Operational Features

Parameter	Value
Melter cavity diameter	15 cm
Melter cavity height	17 cm
Melter inside volume	4.5 liters
Nominal glass depth	7.6 cm
Maximum operating temperature	1,200°C
Nominal operating Temperature	1,150°C
Electrode Dimensions	7.6 cm × 7.6 cm
Electrode Material	Inconel 690
Electrode distance from bottom	0 cm
Electrode current (average)	90 A
Electrode voltage (average)	35 V
Electrode current density (average/maximum)	1.6/2.0 A/cm^2

The RSM is a small joule-heated melter that is capable of processing melter feed on a continuous basis. This capability is key for determining the relationships between the properties of the feed and the properties of the final glass produced. Production of glass in a continuous manner is also more representative of a full-scale system. Testing in the RSM allows for quantitative measurement of the off-gas stream and the performance of parametric studies (e.g., changing one feed component at a time to determine its effect on the process) in a relatively short time frame.

Melter feed is delivered from a feed tank to the RSM feed nozzle by a peristaltic pump. An agitator in the feed tank keeps the slurry well mixed. The feed tank sits on a scale that is monitored by the computer data acquisition and control system. Pump speed (and thus the rate at which feed is introduced into the melter) is controlled from the computer.

The body of the RSM is an Inconel® closed-ended cylinder lined with Alfrax® refractory and containing a Monofrax® K3 refractory melt cavity. An Inconel overflow tube discharges molten glass into a stainless steel canister. An electric kiln surrounds the melter body and minimizes heat loss from the melter body during operation. The discharge section is heated to facilitate pouring of the glass. The stainless steel canister sits inside a smaller kiln maintained between 700°C and 900°C to promote uniform canister filling. Two top-entering Inconel 690 electrodes suspended in the glass supply joule-heating power to the RSM.

Melter off-gas is treated by an off-gas treatment system consisting of a film cooler, venturi scrubber (caustic scrub solution), heat exchanger, high efficiency mist eliminator (HEME), and high-efficiency particulate arrestor (HEPA, also known as high efficiency particulate-air) filter.

A data acquisition and control system monitors and controls the electrodes, the melter and discharge canister kilns, the melter, the heater for the discharge section, and the peristaltic pump for the feed system. Data collected include the voltage and current for major electrical components, temperature at various locations in the system (e.g., molten glass, plenum space in melter, melter kiln, off-gas treatment system), pressure in the melter, and weight of the feed tank. Data are typically archived every minute but are displayed at more frequent intervals to assist the operators.

The melter has been enclosed in a walk-in hood designed for containing any fugitive Hg emissions from the melter that occur when processing feeds that contain Hg. A schematic of the hood is shown in Figure 5.

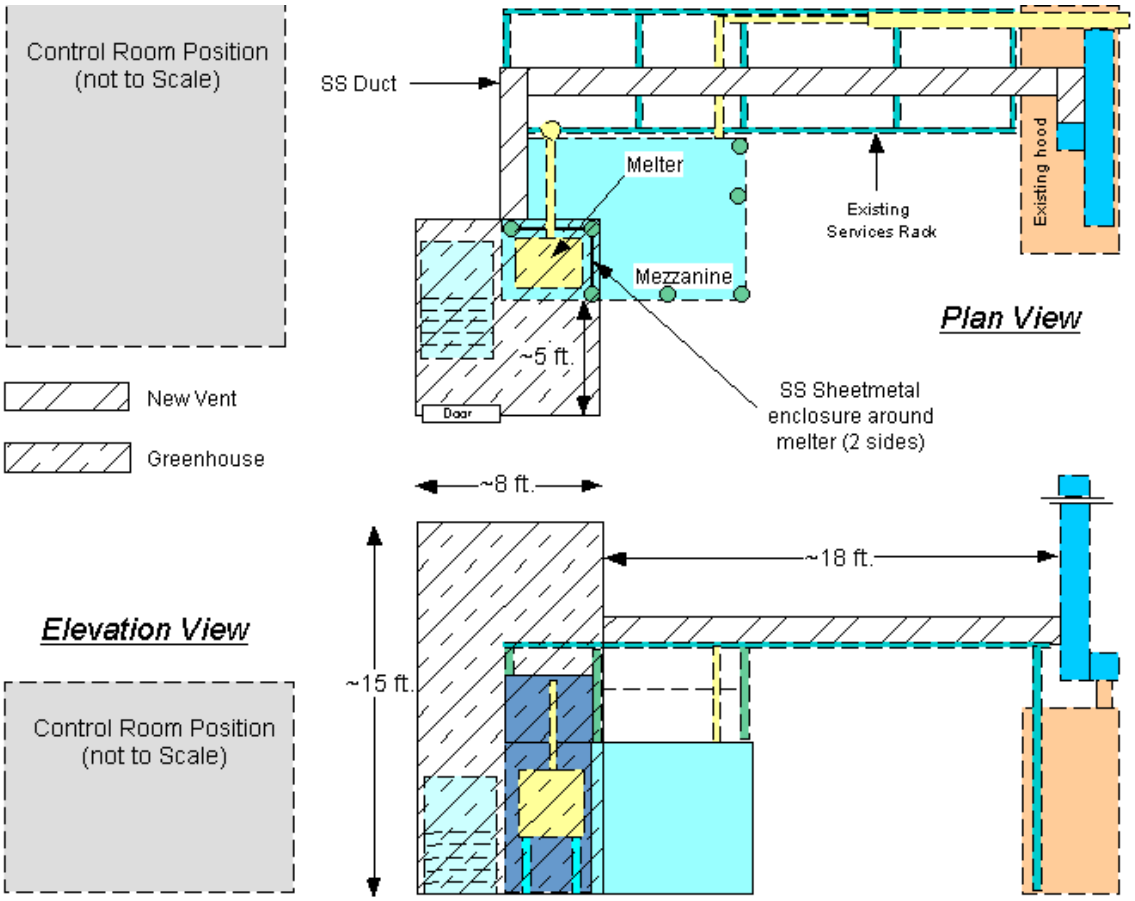

Figure 5. RSM Hood Design

8. QUALITY ASSURANCE

All laboratory data, general observations, and details of the activities performed per this test plan will be recorded in a Laboratory Record Book (LRB) or data entry sheets. Subsequent notebooks will be cross-referenced.

Changes to this test plan will be documented on the workplace copy. Changes may be entered by the test leader, shift leader, or responsible engineer (initialed and dated) and approved by the principal investigator as indicated by initial and date. Changes that may affect successfully achieving the primary test objectives will be concurred upon by the INEEL project staff and noted by initial and date. An explanation of any changes should be noted in the LRB. PNNL standard laboratory practices will be followed throughout the testing.

8.1 Data Quality Objectives

The data quality objectives (DQOs) establish the degree of quality assurance/quality control (QA/QC) required to meet the data quality needs of the test objectives. Table 16 summarizes the DQOs for each test objective previously discussed in Section 2 of this test plan. This table also shows whether each objective is critical (C) or non-critical (NC). Finally, the type of data for each objective is identified as either quantitative (Quan) or qualitative (Qual). Additional discussion regarding specific measurements is given below.

Table 16. Data Quality Objectives for RSM Test Objectives

Objective	C/NC	Quant	Qual	DQOs
1 & 2	C	x	x	The test data and observations must enable determination of the degree of a sulfate layer and melt foaming, cold-top coverage, minimum glass temperature for reasonable operation (tapping and melt mixing), and minimum melt electrical conductivity, as indicated by the ability to maintain the needed heat input at nominal voltage and current levels.
3-6	C	x		Analyses of product streams must be done using standard methods with established QA/QC procedures and with sufficient sensitivity to determine the fate of feed materials, including glass formers, nitrates, sulfur, chlorine, iodine, Hg, and radioactive surrogates; replicates performed to obtain statistical confidence intervals; accuracies of calibrated M&T are summarized in Appendix A.
7	NC	x	x	Comparison data between the redox probe measurements and redox sample analyses must enable determination of the accuracy and precision of the redox probe. Operating performance of the probe (such as ease of use, maintenance requirements, operating life, calibratability, etc.) will be used to indicate useability in operation.

8.2 Quantitative Measurements

Feed and Product Stream Masses—The mass of each stream will be measured with an analytical balance or load cell, user calibrated to ±2% full scale accuracy, with the calibration documented in the laboratory record book. Duplicate samples will not be available since this is the measurement of the total mass of the stream.

Feed (additives) and Product Stream Compositions, Product Durability—The composition of each stream will be measured by obtaining representative samples from each stream and submitting the samples to an analytical laboratory for the required analyses. Accuracy of the analyses will be ensured by the analytical laboratory performing the analyses through the use of standard methods with approved QA/QC procedures. Selected samples will be run in duplicate for each stream and each analytical technique to determine the precision of the measurements. Precision of two measurements will be expressed as the relative percent difference (RPD), which is the absolute value of the difference between the two measurements, divided by the average value of the two measurements, expressed as a percentage.

Process Measurements—The accuracy of process measurements will be ensured through user and/or manufacturer calibration of the test instrumentation and data-acquisition system. Verification of the DAS system to the accuracy indicated in Appendix A will be accomplished using a calibrated signal source to simulate the signal from the measurement equipment to the DAS system. The output of the measurement instrument will be verified by user or manufacturer calibration. All verification and calibrations will be documented on data sheets or in the laboratory record book. Since the data are recorded over time, duplicate measurements are not applicable.

Off-gas Measurements—Gas compositional analysis will be conducted primarily using the user-calibrated CEMS described earlier. The CEMS will also be used to characterize total melter flowrates by measuring the concentration of a He tracer gas that will be continuously injected, at a fixed rate, into the film cooler's air supply. Some off-gas measurements will also be performed using manual sample trains, described earlier. Where applicable, the manual sampling will be done isokinetically to minimize any bias in the results of measurements made of the PM entrained in the off-gas.

8.3 Qualitative Measurements

By their nature, qualitative measurements cannot have quantitative measures of data quality. However, the quality of these measurements can be ensured by establishing guidelines for recording qualitative measurements. Qualitative measurements (observations, comments, descriptions, etc. of system operations and/or abnormal events) should be recorded directly in the LRB, as soon as possible after the occurrence. Descriptions should be as detailed as possible, and referenced to a time or other quantitative process measurement, which will allow correlation of the observation to the quantitative process data. All entries should follow established QA/QC procedures for recording data in the LRB.

9. ENVIRONMENTAL, HEALTH, AND SAFETY

Hazards associated with the operation of the RSM are documented in SOP #80 (Tixier 2000). Operations willl be conducted in a safe manner. Procedures and practices will be established to prevent the inadvertent or uncontrolled release of environmental contaminants. PNNL will take all reasonable precautions to protect the safety and health of its employees and members of the general public, and will comply with all applicable safety, health, and environmental regulations as set forth by local, state, and federal authorities.

9.1 Flammability Mitigation

Different kinds and amounts of organic reductants added to the melter feed will be evaluated during this test. In other joule-heated melter operations with other kinds of high level waste feeds, reductants react with the nitrates in the feed, enabling gas generation from nitrate decomposition to occur more rapidly in the cold top than may otherwise occur. When the nitrate decomposition and resultant gas generation occurs more rapidly in the cold top, foaming of the melt itself (caused by gas generation in the upper layer of the melt itself) is minimized. This enables a higher relative feedrate. The ability to reduce foaming by adding reductants to the feed will be evaluated in this test. Added reductant also enables operator control of the glass redox state, which affects the melter performance and final glass properties.

Organic reductants will react in the melter cold top and plenum space to thermally decompose, react with nitrates, and also react with other sources of oxygen such as water (and water vapor) and oxides in the feed. Although CO_2 and H_2O will be the dominant oxidation products of the reductant, some products of incomplete oxidation, such as CO, H_2, and volatile organic compounds (VOCs), are formed. While the highly oxidizing plenum conditions minimize the formation of H_2 and VOCs, CO at levels of around 0.1% can be formed.

In general, flammability concerns in the melter plenum and associated off-gas system are avoided if the plenum temperature is maintained above the auto-ignition temperatures of the potential organic vapors. Any generation of flammable mixtures would safely combust in the refractory-lined plenum before sufficient potential energy could be built up to cause dangerous pressure surges in the plenum. According to safety control practice at the Savannah River Site's Defense Waste Process Facility (DWPF), a minimum plenum temperature of 300°C assures a sufficiently high temperature. Flammability concerns in the melter plenum will be avoided during the test series by maintaining at least this temperature in the melter plenum.

Beyond the use of plenum temperature control of flammables, the RSM test will comply with the NFPA 69 standard. According to NFPA 69, the lower flammable limit (LFL) of a vapor must be maintained below 25% using engineering controls if automatic interlocks based on the flammable gas concentration are not used. This means that to maintain a guaranteed safe condition in the off-gas system, sufficient dilution air must be added in a controlled manner such that the LFL does not exceed 25%. The worst-case off-gas system concentration of CO (where all available carbon is converted to CO) that could occur under maximum melter feeding and sugar loading conditions and minimum film-cooler flowrate (6 scfm) is 1.2% (9.4% of LFL). Contributions of H_2 and VOCs to the combined LFL are relatively small, since levels of those species are typically well below the concentration of CO. Nevertheless, in order to assure safe operating conditions, active monitoring of combustible off-gas constituents will be continuously conducted through use of a total hydrocarbon analyzer and a gas chromatograph for H_2 and CO.

In the event that H_2, CO, or H_2+CO should exceed 25% of their flammability limits (4%, 12.5%, and 8.25%, respectively) under baseline operating conditions, film cooler dilution air will be increased, and/or

feed rates will be reduced to control off-gas concentration at safe operating limits (25% of LFL). Total hydrocarbon concentrations will be controlled to 0.75% (25% of ethane's LFL). If active, sustained flammability-limit control cannot, for any reason, be maintained, feeding operations will be terminated. Feed termination followed by cold-cap burn-off must precede any designed interruption of the film cooler's air supply.

9.2 Personnel Safety

Safe operation of the RSM and personnel safety is ensured when personnel follow training by cognizant PNNL safety staff. At a minimum, this training includes reading and understanding the following documents:

1. Research Scale Melter SOP #80
2. The Research Scale Melter Test Plan
3. All applicable test instructions
4. Applicable Material Safety Data Sheets
5. Training on the uses and limitation of Mercury Monitor
6. All IOPS training materials assigned by Cognizant Space Managers for all laboratories (including Lab 177) to be accessed by RSM-2 participants.

These training activities will be documented in sign-off record sheets when each training activity is completed. After completing all reading assignments, operating staff will be required to attend a complete system walk through. The walk through will include a review of the test plan and the RSM SOP.

There may be times when the melter will need to be opened (to clear the off-gas line, etc.). Prior to opening the melter, feeding will be stopped to allow the cold cap to be fully incorporated into the glass melt. This will ensure that no noxious or hazardous gases are present in the system when it is opened. Additionally, the melter will be maintained at a slight vacuum so that no gases are expelled into the work area.

9.2.1 Noxious Gas Releases

In the unlikely event that an off-gas processing system failure occurs, noxious gases generated by the inventory of unprocessed feed material within the melter (i.e., the cold cap) will, to some extent, be released to the experimental area hosting the RSM. Since the RSM is contained within a walk-in hood, all released gases will be contained and exhausted through the facility's ventilation system. The various operational scenarios that could lead to a process exhaust failure and the emergency responses required are detailed in RSM-SOP-80. However, the presence of the exhausted enclosure about the melter will minimize the consequence of these accident scenarios should a pressurized event occur.

Although the melter hood enclosure is designed to contain and exhaust process gasses that could conceivably be released during melter processing, mercury contamination within the hooded enclosure could result from such a process upset condition. To accommodate this possibility, response actions have been defined based upon the magnitude of the mercury-vapor concentration resulting from surface contamination of the melter and its enclosure.

A portable mercury analyzer will be used to monitor mercury vapor concentrations within the melter enclosure at least once a shift or as needed to evaluate conditions subsequent to a process upset or whenever handling secondary wastes that might generate mercury vapors. Hood entry constraints will be based upon measured mercury vapor concentrations using this instrument as summarized below. These

tabular limits set below are conservatively based upon threshold limiting values (TLVs) established by the American Conference of Governmental Industrial Hygienist (ACGIH) (and OSHA's permissible exposure limits [PEL's]) for the most restrictive class of mercury compounds: Alkyl forms mercury.

Hg Conc. (mg/m^3)	**Operational Constraint/Action**
$(Hg)_v < 0.005$	None but ALARA
$0.005 \leq (Hg)_v < 0.01$	Exposure time < 4-h/shift/man
$0.01 \leq (Hg)_v < 0.03$	Masked entry only
$(Hg)_v \geq 0.03$	Mask entry only. Evaluate cleanup options.

Mercury monitoring should be used to assess the safety whenever potentially contaminated process waste streams are manipulated. Active source or workplace ventilation may be used in the short term to ensure that breathing areas are maintained at a safe level, i.e., $(Hg)_v < 0.005$. However, long-term work involving mercury-contaminated material must be performed within an exhausted hood enclosure.

In the event that process materials are spilled, the mercury monitor must be used to assess whether mercury vapors are present. If present, the contaminated surface must be cleaned with mercury spill-kit material after the process materials are removed. Latex gloves shall be worn whenever there is a possibility of contacting mercury-contaminated materials or surfaces.

PNNL's Industrial Hygiene group will be apprised of all changes in exposure status as established by the tabular concentration limits discussed above. The point of contact regarding mercury-related health and safety questions is Scott Nickerson, who can be contacted at 375-2377.

Although the health hazards associated with mercury should not and have not been minimized or underestimated, it is instructive to demonstrate that the cumulative quantities of mercury involved in the RSM-2 test are rather small. It is expected that 80-L of simulated waste having a mercury concentration of 0.002 M will be processed over the duration of the RSM-2 test. This corresponds to a total mercury mass of 32 g or to a 2.5 cc volume if in elemental form. Moreover, from previous testing, it is expected that only a very small fraction of mercury evolved from the melter during RSM-2 will be in the elemental form.

9.2.2 Personnel Protective Equipment

During routine RSM testing activities, required personnel protective equipment includes safety glasses and protective gloves. Leather gloves are required when performing operations at and around the kiln, melter, and off-gas line between the melter and the EVS. Because of the corrosive process stream conditions, a full-face shield and latex gloves must be worn when conducting sampling, pH adjustments, or transfer operations involving corrosive and potentially mercury-contaminated secondary waste streams. Nitric acid is the main chemical hazard in the SBW simulated waste, melter feed and in secondary waste streams generated by the RSM's off-gas processing system. Because the high acidity of these process streams (pH~0), a full-face shield, goggles, lab coat (or other suitable outer wear), and protective gloves are also required when in proximity of the agitated waste and feed tanks, when sampling, handing, or transferring waste or when performing maintenance or disassembling the melter feed system, as the transfer line could be under pressure, e.g., a plugged feed line. In the event of a feed spill, spill-kit materials must be immediately deployed to mitigate corrosive conditions and possible mercury contamination. All personnel protective equipment (PPE) used with waste and feed streams must be inspected and washed free of all waste and/or feed residues that may be present *before* the PPE is removed. When required, respirator masks with appropriate mercury-adsorbing cartridges shall be used

only by mask fit-qualified PNNL staff; all other hood entries, when measured mercury vapor concentrations are ≥ 0.01 mg/m^3, are prohibited. Mercury samplers will also be deployed within the melter enclosure to provide a measure of integrated daily exposures to airborne mercury vapor. Other safety-related equipment needed in support of melter operations are discussed in SOP #80.

9.2.3 Melter Electrical Safety

It may be necessary during RSM testing to probe the melter's glass pool for the purposes of extracting molten and/or glass samples or to mechanically stir the melt to help expedite the dissolution of molten sulfate salts. This can only be done with an insulating, alumina probe if power to the electrodes is not, first, physically locked out by the operator. Nevertheless melter electrode voltage shall be reduced to zero before probing with an alumina rod unless the requirement is waived by safety. Details concerning the probing of the melter with an alumina rod are provided in RSM test instructions; however, because of the electrical hazard associated with the electrode power source, a stainless steel or other conductive probe material may not be used unless the operator is authorized and trained to use lockout and tagout procedures, and they are appropriately applied to the electrode breaker disconnect switch.

9.2.4 Material Safety Data Sheets

Material Safety Data Sheets (MSDSs) associated with the RSM feed will be maintained at the RSM unit during testing. Reading and understanding of the MSDS will be required for all operators and will be documented via a training sheet.

9.2.5 Medical Requirements

Not Applicable, except as required l for respirator use.

9.2.6 Confined Space

Not Applicable.

9.2.7 Respiratory Protection Requirements

If required (see Sect 9.2.2), respirator masks with appropriate mercury adsorbing cartridges shall be used only by mask-fit-qualified PNNL staff.

9.3 Waste Minimization/Management

The following waste minimization practices will be followed:

- The chemical Management System (CMS) will be checked before ordering new chemicals.
- Waste will be accumulated in satellite accumulation areas until the project is completed, after which waste will be disposed of in accordance with PNL-MA-8.
- Waste will be stored in separate containers as appropriate to minimize volume of highly toxic waste and toxic waste, etc.
- Surrogate feed preparation procedures will be designed to avoid, where possible, toxic components.

9.4 Emergency Response

Actions for Emergency Response are detailed in SOP #80. Personnel who need to comply with SOP #80 shall be trained and understand the appropriate actions to be taken in the event of an emergency, know how to respond to alarms in the APEL High Bay, or be under the direct control of PNNL staff who have had the required training. Building actions are discussed in the APEL Facility Emergency Procedure. Actions specific to the operation of the RSM are contained in SOP #80.

10. REFERENCES

American Society for Testing and Materials (ASTM). 1994. *Standard Test Methods for Determining Chemical Durability of Nuclear Waste Glasses: The Product Consistency Test (PCT),* ASTM C1285-94.

Tixier, J. 2000. Standard Operating Procedure #80 (SOP#80), "Research Scale Melter Safe Operating Procedure," Revision 5, Pacific Northwest National Laboratory, Richland, Washington (effective date November 11, 2000).

Research-Scale Melter Measurement and Testing Equipment and Test Instruction

DESCRIPTION	M&TE DESCRIPTION	LOCATION	CALIBRATION BASIS	CALIBRATION LEVEL
Analog Gauge	EVS Nozzle Pressure - Ashcroft (0 - 80 psig)	South side of RSM stand	NA	Indication Only
Analog Gauge	Melter Vacuum - Magnehelic (0 - 10 in. wc)	On top of RSM	Na	Indication Only
Analog Gauge	Off-gas Header Vacuum - Magnahelic (0 - 80 in. wc)	Southwest corner of RSM stand	NA	Indication Only
Analog Gauge	Pre-HEME Vacuum - Magnahelic (0-5 in. wc)	South side of RSM stand	NA	Indication Only
Analog Gauge	RSM Off-gas Line Vacuum - Magnahelic (0-15 in. wc)	Southwest corner of RSM stand	NA	Indication Only
Interface module	Discharge Can Power	SCR Panel	Integral to SCR	
Interface module	Electrode Current	SCR Panel	Integral to SCR	
Interface module	Electrode Potential	SCR Panel	Integral to SCR	
Interface module	Electrode Power	SCR Panel	Integral to SCR	
Interface module	Kiln Power	SCR Panel	Integral to SCR	
Interface module	Kiln Power Output	In Kiln	Integral to SCR	
Pres. Transducer	Ejector Venturi Scrubber Diff. Pressure	North side of RSM stand	NA	Indication Only
Pres. Transducer	Feed Pressure	Feed line near valve panel	NA	Indication Only
Pres. Transducer	Film Cooler Diff. Pressure	North side of RSM stand	NA	Indication Only
Pres. Transducer	High Efficiency Mist Eliminator Diff. Pressure	North side of RSM stand	NA	Indication Only
Pres. Transducer	Melter Pressure	North side of RSM stand	Site Calibration Services	
Pres. Transducer	Total Off-gas System Diff. Pressure	North side of RSM stand	NA	Indication Only
Rotometer	Blower Cooling Flow	West wall near blower	NA	Indication Only
Rotometer	EVS HX Cooling Flow	Southeast corner of RSM stand	NA	Indication Only
Rotometer	Feed Nozzle Cooling Water Out Temp.	TE-2006 near water discharge header	NA	Indication Only
Rotometer	Film Cooler Air Supply Flowrate	Northeast corner of RSM stand	NA	Indication Only
Type K Thermocouple	Discharge Canister Temperature	In Can Furnace	Vendor certification	+/- 2% full scale
Type K Thermocouple	Feed Nozzle Temperature	TE-20010 near water discharge header	NA	Indication Only
Type K Thermocouple	Heat Xfer Temperature	TE-2006 South side of RSM stand	NA	Indication Only
Type K Thermocouple	Kiln Temp Bottom	In Kiln	NA	Indication Only

Research-Scale Melter Measurement and Testing Equipment and Test Instruction (Continued)

DESCRIPTION	M&TE DESCRIPTION	LOCATION	CALIBRATION BASIS	CALIBRATION LEVEL
Type K Thermocouple	Kiln Temp Middle	In Kiln	NA	Indication Only
Type K Thermocouple	Kiln Temp Top	In Kiln	NA	Indication Only
Type K Thermocouple	Melt Temperature (T1 - Control)	In Melter	Vendor certification	+/- 2% full scale
Type K Thermocouple	Melt Temperature (T2)	In Melter	Vendor certification	+/- 2% full scale
Type K Thermocouple	Off-gas Temp Exiting Film Cooler	TE-6815A	NA	Indication Only
Type K Thermocouple	Overflow Heater Temp	In RSM Overflow	Vendor certification	+/- 2% full scale
Type K Thermocouple	Plenum Temperature	In Melter	Vendor certification	+/- 2% full scale
Type K Thermocouple	Post EVS Off-gas Temperature	TE-2003 South side of RSM stand	NA	Indication Only
Type K Thermocouple	Scrub Liquid Temperature after Heat Exchanger	TE-20010 South side of RSM stand	NA	Indication Only
Type K Thermocouple	Water Header Temperature	Southeast corner of RSM stand	NA	Indication Only
Weigh Scale	Feed Tank Weight	Feed station stand	Site Calibration Services	
Weigh Scale	Glass Scale Weight	Under RSM Kiln	NA	Indication Only

RSM-01-2 TEST INSTRUCTION

This test instruction provides additional guidance to testing staff during the execution of the INEEL SBW RSM test, RSM-01-2, that will be referred to, from this point forward, as RSM-2.

Objectives

Waste Processing, Off-gas Emission Characterization: For the RSM-2 test, melter feed with a fixed SBW waste loading will be processed using various concentrations of sugar and glycolic acid, in separate processing campaigns, to assess the impact reductant and reductant concentration upon the melter's processing rate, off-gas emission characteristics and sulfur behavior. Melter-feed processing will be evaluated under the reference condition where reduced Fe^{+2} can just be detected, under extreme oxidizing conditions where no reductant is used and at the maximum acceptable reductant concentration where reduced Fe^{+2} accounts for 30% of available iron in the vitreous product. Glass oxidation state will be determined by wet chemical, colorimetric procedure and tracked electrochemically using melter glass-probe measurements.

The melter feed to be used in RSM-2 was specifically formulated to accommodate, in the product glass, the sulfur present in surrogate SBW. However, the nature of reductant used and its concentration can affect cold-cap chemistry and resultant feed-component partitioning. This will be important not only for sulfur, but also for volatile halogens and mercury compounds. Consequently, the melter source term for volatile as well as condensed phase effluents will be discretely sampled during steady-state processing conditions and the EVS's secondary waste stream will be completely collected and fully characterized during selected processing campaigns.

Limited testing is also planned to evaluate a higher level of sulfate in the feed, in order to bound the range of sulfate in the actual SBW. At elevated sulfur feed levels, the higher reductant concentrations identified in the Test Plan will be used in order to minimize the possibility of forming a separate molten sulfate layer. This testing phase will also help determine whether the new feed formulation can accommodate a higher loading of reference SBW without creating a separate sulfate phase.

The reductant concentrations to be used to achieve glass oxidation levels previously discussed will be defined by INEEL prior to the test. Based on testing to date, the equivalent sugar addition levels to be used to obtain reference and maximally reduced glasses are 135 g/L and 170 g/L, respectively.

Glass Oxidation Tracking: An attempt to track the oxidation state of the molten glass in the RSM's melt cavity will be conducted throughout the duration of RSM-2 testing by periodically measuring the electrochemical potential between immersed platinum electrodes. The electrodes are composed of platinum wires running through alumina tubes that extend from the melter lid to near the bottom of the RSM's melt cavity. Each of these identical electrode probes will be purged with different gasses—air (oxygen) for the reference probe and argon for the active oxidation state-sensitive electrode. Because of the large ac-voltages present during active processing, Redox tracking measurements can only be made when Joule heating is interrupted. The DVM used to collect this electrochemical data will be permanently connected to the platinum probes and will need to be appropriately ranged and/or powered up or down to suitably match electrical conditions in the melt cavity. Note: failure to appropriately modify the state of the DVM when resuming Joule heating may very well ruin the voltage-measurement device.

Glass Oxidation Measurement: An INEEL wet-chemical, colorimetric laboratory procedure will be used, contemporaneously, throughout all phases of RSM-2 testing to establish the oxidation state of the product glass. This work will be performed in the 202/204/212/214 laboratory complex by INEEL staff that have been appropriately trained and have been granted unescorted access to these laboratories.

Melter Off-gas Sampling: At various times (see Test Plan) during RSM-2 testing, discrete sampling of melter-generated aerosols and condensable effluents will be conducted. This will occur, without exception, during steady-state processing periods. It is the operator's task to maintain these conditions throughout the sampling campaign.

To initiate sample collection, a sampling probe connected to a filter and a series of impinger bottles will be inserted into the off-gas sampling port located just downstream of the film cooler within the walk-in hood. An isolation valve equipped with inlet slip fittings will be used to reduce the impact of sample probe insertion and extraction activities upon melter vacuum. However, prior to probe insertion or extraction, the melter vacuum should be manually increased, if necessary, to 2-in. wc to compensate for a temporary increase in off-gas system inleakage. The off-gas vacuum control valve should be dynamically adjusted during these probe insertion and extraction periods to maintain this melter vacuum.

Condensate Sample Collection: The collected EVS's secondary waste stream will be manipulated offline to harvest all settled undissolved matter. This separation will be conducted in a secondary containment within the feed preparation hood. To accomplish this, the condensate supernatant will be drawn off the settled solids with a peristaltic pump that will deliver this liquid directly to a waste drum. The settled solids along with residual supernatant will then be transferred to an appropriately sized vessel for subsequent analysis.

Feed Stream Sulfur Content: Because of the fixed waste loading associated with RSM-2 testing, waste component concentration is not, in general, an operational variable. However, the melter feed content of sulfur may be parameterized toward the end of the RSM-2 test to evaluate the impacts of using the upper-bound value for the range of sulfur in SBW. To accomplish this, the melter-feed composition of sulfur will be shimmed by adding sulfuric acid. The impacts of this change upon off-gas composition, melter glass composition, and the possible accumulation of a molten salt phase will be of special interest during this phase of testing.

Salt Layer Formation: Detection of a sulfate phase will require careful and frequent observations of the cold cap and glass surface. From past experience, it is known that the salt layer will appear translucent and fluid (like water). The edges of the cold cap or liquid and solid splatter that lands on top of a salt layer could look similar to the appearance of something being placed in a deep fryer. If a layer is thought to be present, a valuable validation test is to probe the glass surface. To do this, interrupt the feed to the melter, and, once the slurry has evaporated, turn off the power to the electrodes and quickly extend a nonconductive alumina probe through the view port into the area where the salt is thought to be present. Remove it quickly to minimize the heating up of the probe. Resume current flow to the electrodes and feeding. Examine the probe for signs of salt adhering to the probe. **Note: because of the electrical hazard associated with the electrodes, a stainless steel or other conductive probe material may not be used unless you are authorized and use lockout and tagout procedures to lock out the electrode breaker disconnect switch.**

If excessive sulfate has accumulated, the shift leader will confer with the INEEL shift member and agree to suspend this feeding phase. The cold cap will be allowed to melt into the glass, and a second confirmatory probe sample will be obtained. Any salts collected should be washed from the probes with as little DI water as possible and the DI water and salt sample retained as a test sample. If glass can be made to pour from the melter, a short pour sequence should be executed and a glass sample obtained and saved. Do not pour any more glass than is necessary to account for glass in the riser piece and the glass sample itself. The sample will be analyzed to establish a maximum sulfate concentration and redox state. If glass cannot be obtained via the discharge, obtain a sample via the view port (again using a ceramic alumina probe unless lockout/tagout is applied).

To dissipate the sulfate, attempt first to stir the sulfate into the glass melt. This must be done in short durations with the electrode power turned off. If no obvious reduction in sulfate layer occurs, a reductant slurry will be made and fed to the melter using the feed pump to meter the slurry into the RSM. Because of possible mercury contamination, the addition of dry reductant through the melter's view port will not be allowed.

After the sulfate layer has been removed, a glass sample is to be removed from the melter via the viewport (again using a ceramic alumina probe unless lockout/tagout is applied) and archived. The feed material in the feed tank should be shimmed to increase reductant or reduce waste loading, and melter feeding should be resumed.

Reductant Levels: The initial reductant level will have been established prior to starting the run. However, the test plan specifies that more oxidizing feeds will first be processed before evaluating feeds containing higher reductant loadings. Consequently, the heels of all unprocessed feeds containing lower reductant loadings should be transferred to a holding vessel where its reductant level can be shimmed for subsequent use during the processing of more reduced feeds.

Supplemental information on test activities, schedules, and documentation are described below.

Test Activities

1. Feed Batching Activities: With the exception of the first batch, feed batches will be sized to provide Test Plan-prescribed processing periods. The first hours of operation will establish the volumetric feed rate possible, e.g., one to two liters per hour. This value will be used to calculate the subsequent volumes of feed to be prepared and the quantity of glass formers (here glass formers includes the reductant) to be used. At 20% waste loading, there is expected to be a 40% to 50% volume expansion in feed volume when the glass formers are added to the SBW waste simulant. The attached Feed Batching Sheet (Attachment 1) will be used and completed for the preparation of each feed batch. The steps are:

    a. Test engineer to enter into the Feed Batching Sheet:
        i. Time and date
        ii. A sequential feed batch number (Starting with "RSM-01-2-1")
        iii. The SBW target batch volume
    b. Transfer volume of SBW simulant to SBW transfer tank—The SBW "master batch" will be stored in the large RSM feed tank. A poly line connected to the bottom of the transfer tank will be used to dispense feed to the make-up tank. It is required that splash protection, including coat and overalls, full-face shield, safety goggles, and acid-resistant gloves be used for this task.
    c. Estimate the volume and measure the SBW gross weight (tare weight is written on the transfer tank: 2.30 kg).
    d. Use SBW density and weight to confirm/calculate the actual SBW volume.
    e. Calculate the glass former weights by entering these data into the Batch Sheet.
    f. Weigh glass formers and combine into the glass former containers.
    g. Shift engineer or lead test engineer review and sign off the Feed Batching Sheet.

2. Melter Feed Tank Transfer: To refill the melter feed tank perform the following (note: two staff are required for this activity):

a. Place two buckets next to the feed station. One should have approximately 1 inch of water to use to wet paper towels needed for wiping spills. The second will serve as a receptacle for any used paper towels.
b. Don PPE (goggles, face shield, gloves and lab coat, or other protective overgarments).
c. Note the melter feed tank weight and time and record on the batch sheet. Note: be sure to remove all tools, buckets, etc. from the scale before recording the feed tank weight.
d. Place the SBW transfer tank on the transfer stand and secure it.
e. Place the glass former containers within easy access.
f. Place the copus blower intake hose at the feed station.
g. Reduce the agitator speed of the melter feed tank until no splashing is occurring.
h. Extend the SBW transfer hose into the lid opening and open the valve at the base of the SBW transfer tank. Note: while turning the valve, use the other hand to hold the valve body to prevent it from possibly pulling the tubing from the poly tank. Allow contents to transfer into the melter feed tank. **OBSERVE CLOSELY THE MELTER FEED TANK LEVEL AND CLOSE THE VALVE IF THE LEVEL APPROACHES 6 in. FROM THE TOP OF THE TANK.**
i. After contents of the transfer tank have emptied into the feed tank, rotate the restraining strap away from the tank and raise and tip the transfer tank to allow any remnants of SBW to drain into the melter feed tank. Close the valve and carefully remove the transfer line from the melter feed tank and raise the open end up to prevent any drips. Use a wet paper towel to wipe the end of the tube and insert the opening into a nitrile or vinyl glove to prevent dripping while carrying the transfer tank back to the walk-in hood.
j. Read and record the melter feed tank weight and time on the batch sheet.
k. Rotate the agitator shaft angle to the vertical position and secure. This orientation allows a larger vortex to form.
l. Increase the agitator speed to develop a vortex around the shaft. **IF SPLASHING DEVELOPS, REDUCE THE SPEED UNTIL SPLASHING DOES NOT OCCUR.**
m. Remove the tank lid and slide it into the plastic bag next to the tank to prevent any feed splatter on the tank lid from spreading.
n. Turn on the copus blower and hold the intake at the tank edge.
o. The second staff member will slowly sprinkle the glass formers onto the surface of the melter feed tank slurry being careful to assure that a thin even layer is deposited without piles or agglomerations forming.
p. Replace lid, turn off copus blower, clean up any spills, and remove tools and other material from the weigh scale (the bag used to hold the tank lid can remain).
q. Inspect each other for any signs of feed splash stains and wipe with a damp paper towel.
r. Note the time and melter feed tank weight and record on the batch sheet **and the logbook**.

3. Melter Feed Tank Transfer: To completely change feed reductant, follow steps in "2." after completing the following
   a. Turn feed tank agitator off
   b. Drain melter feed tank contents into an appropriately sized vessel
   c. Close drain valve and remove receiver tank to a hood and initiate agitation
   d. Follow steps in "2." above.

4. <u>EVS Condensate Tank Sampling:</u> During specific segments of RSM-2 testing, as outlined in the Test Plan, collection and subsequent segregation of the quench scrubber's waste stream will be conducted. This process will be initiated by completely draining the condensate tank of its contents using a spray lance to wash out any residual insoluble matter. The tank will then be

charged with ~60-L of 1M nitric acid solution before beginning the campaign. To accomplish this the following steps should be taken.

a. Suspend feeding to the melter and water flush the feed nozzle.
b. When the cold cap has been completely consumed, reduce the melter vacuum to ½-in. wc.
c. Turn off EVS pump and estimate the condensate volume.
d. Close EVS drain line.
e. Position condensate waste receiver vessel(s) at the condensate tank and connect drain and exhaust lines (if an exhaust line is incompatible with the receiver, position a Copus blower in the proximity of the vessels lid to exhaust displaced gases).
f. Drain the condensate tank using a spray lance to wash out all insoluble.
g. Close the tank drain valve and remove receiver vessel(s).
h. Add ~50 L of water and 3.9 L of concentrated HNO_3 to the condensate tank and adjust the volume to 60 L with water.
i. Open the EVS's drain valve
j. Turn on the EVS pump.
k. Increase melter vacuum to 1-in. wc to 2-in. wc and resume feeding.

At the conclusion of the testing campaign for which all condensate and undissolved solids will be harvested, the following steps should be conducted to collect the EVS's waste stream.

a. Suspend feeding to the melter and water flush the feed nozzle.
b. When the cold cap has been completely consumed, reduce the melter vacuum to ½-in. wc.
c. Turn off EVS pump.
d. Close EVS drain line.
e. Position condensate sample receiver vessel(s) at the condensate tank and connect drain and exhaust lines (if an exhaust line is incompatible with the receiver, position a Copus blower in the proximity of the vessels lid to exhaust displaced gases).
f. Drain condensate tank using a spray lance to wash out insoluble.
g. Close tank drain valve, remove receiver vessel(s) to the feed makeup hood and allow suspended solids to settle.
h. Add ~50-L of water and 3.9-L of concentrated HNO_3 to the condensate tank and adjust the volume to 60-L with water.
i. Open the EVS's drain valve
j. Turn on the EVS pump.
k. Increase melter vacuum to 1 in. wc to 2 in. wc and resume testing activities.

5. Sample Identification: Mark sample containers with the following:

a. RSM-01-2-
b. Sequential Sample Number
c. Date & Military Time
d. Sample description, e.g., *Feed, Condensate, Glass Sample*
e. Initial of operations staff obtaining sample.

6. Routine data sheets and sample logs are attached for information. Data sheets #1, #2, & #3 are to be filled out every hour on the hour.

ATTACHMENT 1: FEED BATCHING CALCULATION SHEET

Batch Sheet No. RSM-01-2- 1

Prepared by: R. W. Goles

Date: 8/6/01 Time: 8:00

Reductant (select):	Sugar	X	Glycolic	
Glass Oxidation	Hi		Low	X

SBW target volume, Liters: 20

Note: shaded areas require value to be input

SBW transfer tank gross wt:	27.1	kg
SBW transfer tank tare wt:	2.30	kg
SBW transfer tank net wt:	24.80	kg
SBW Density:	1.25	kg/L

Calculated volumed transferred: Net wt. / density = 19.84 Liters

Target glass waste oxide fraction: 0.2

Glass Former Addition Calculation

Glass former weights: 114.6 gm WO/L * (1 gm glass/ X gm WO) * (gm total GFO/ gm glass) * Y L SBW * (gm GF chemical / gm total GFO)

Waste oxide loading, g/L:	125.6	= A
Fraction waste oxide loading:	0.2	= B
Fraction glass former loading:	0.8	= C
SBW Volume, liters	19.84	= D
(A/B) * C * D =	9971.27	= E

23.7

Glass formers	F		Batch totals: (E * F)		Initial after weighing
gm silica / gm total GFO =	0.680	=>	6775.5	grams	
gm LiOH*H2O / gm total GFO =	0.172	=>	1711.4	grams	
gm boric acid / gm total GFO =	0.107	=>	1067.6	grams	
gm Ca(OH)2 / gm total GFO =	0.066	=>	661.3	grams	
gm NaOH / gm total GFO =	0.055	=>	552.1	grams	
gm V2O5/ gm total GFO =	0.049	=>	486.6	grams	
gm Mg(OH)2 / gm total GFO =	0.025	=>	252.5	grams	
gm ZrO2/ gm total GFO =	0.024	=>	243.3	grams	
gm Fe2O3 / gm total GFO =	0.015	=>	151.6	grams	
gm sugar/L SBW	135	=>	2678.4	grams	

Initial melter feed tank weight: _____ kg Time: _____

Melter feed tank wt. after SBW addition: _____ kg Time: _____

Melter feed tank wt. after glass formers addition: _____ kg Time: _____

Completed by (sign & data): _____

Reviewed & Approved by: _____

ATTACHMENT 2: SAMPLE LOG SHEET

RSM-01-2 Sample Log								
Sample Number	Date	Time	Initials	Sample Source	Size	ARF #	Archived	Comments
RSM-01-2-								
RSM-01-2-								
RSM-01-2-								
RSM-01-2-								
RSM-01-2-								
RSM-01-2-								
RSM-01-2-								
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ATTACHMENT 3: OFF-GAS SAMPLING DATA SHEET

Off-Gas Sampling Data Sheet #3

RSM-01-2	By:								
	Date:								
	Time:								
DESCRIPTION	**units**								
Gas Chromatograph									
He	ppm								
H_2	ppm								
O_2	ppm								
N_2	ppm								
NO	ppm								
CO	ppm								
CO_2	ppm								
N_2O	ppm								
NOx	ppm								
NOx Analyzer Computer									
Time	hh:mm								
NOx	ppm								
NO	ppm								
NOx Voltage	Volts								
NOx Instrument									
Range selector	value								
Mode selector	value								
Sample Pressure	psig								
Sample bypass flow	sccm								
Ozone Pressure	psig								
SO_2 Instrument									
Mode	1st Fld								
Range	ppm								
Reading	ppm								
MKS Mass Flow									
Ar Carrier, Chan-1	lpm								
He Tracer, Chan-2	lpm								
Total Sample , Chan-3	lpm								
Sample Pump*									
Vac Gauge	"Hg								
Pres Gauge	psig								
Gas Cylinders Tank Pres#									
O_2	psig								
Ar GC	psig								
Ar Sweep	psig								
He GC	psig								
He Tracer	psig								
Fid Analyzer (in Hood)									
Conc. (multi-digit display)	value								
Range (single digit display)	value								
Pump Outlet Pres	psig								
Air Pres (reg next to pump)	psig								
Smple Pres, (Internal reg)	psig								
Fuel Pres, (internal reg)	psig								
time	hh:mm								

ATTACHMENT 4: DATA SHEET #1: PRIORITY & ELECTRICAL DATA

RSM-01-1										
	By:									
	Date:									
	Time									
DESCRIPTION	UNITS	Range								
Priority Data										
Melt Temperature (T1, control	° C	1125 - 1175								
Melt Temperature (T2)	° C	1125 - 1175								
Plenum Temperature	° C	400 - 600								
Feed pump setting	%									
Cold Cap Coverage	%	> 75								
Slurry Pool Coverage of Cap	%									
Cold Cap Flexibility Visible	y or n									
Number of Vents	#									
Cold Cap Thickness	inch									
Phase Separation (Note in LRB	y or n									
Glass Pouring	y or n									
Electrical Data										
Electrode Potential	Volts									
Electrode Current	Amps									
Electrode Power	kW									
Melt Resistance	?									
Melt (Electrode) Setpoint Tem	° C									
(Electrode Power) Output	%									
(Electrode) Control Mode	A or M									
Kiln Power	kW									
Kiln Temp Setpoint	° C									
Kiln Actual (Middle) Temp	° C									
Kiln Power Output	%									
Kiln Control Mode	A or M									
Discharge Can Power	kW									
Discharge Can Temp Setpoint	° C									
Discharge Can Actual Temp	° C									
Discharge Can Power Output	%									
Overflow Heater Power	kW									
Overflow Heater Setpoint	° C									
Overflow Heater Power Outpu	%									
Time	hh:mm									

ATTACHMENT 4: DATA SHEET #2: ROUTINE STATUS SHEET

RSM-01-1

		By:								
		Date:								
		Time:								
DESCRIPTION	**units**	**range**								
Feed Nozzle Cooling Flow	gpm	.5 - 1(reg FNT)								
Film Cooler Air Supply	scfm	1-10 (reg OGT)								
Melter Vacuum-Magnehelic	in. H_2O	0.1 - 2.0								
EVS ? P	in. H_2O									
Film Cooler ? P	in. H_2O									
HEME ? P	in. H_2O									
System ? P	in. H_2O									
EVS HX Cooling Flow	gpm	1-5 (reg SLT)								
EVS Scrub Tank Volume	gallon	35 - 50								
EVS Nozzle Pressure	psi	50 - 55								
EVS Scrub Solution pH	pH	> 9								
OG Control Valve position	%									
OG Control Valve mode	A or M									
Feed Pump (tubing) Condition										
Agitator Setting	%									
Blower Cooling Flow	gpm	1 - 1.5								
Feed Nozzle Temp (FNT)	° C	< 40								
Off-Gas Temp (OGT)	° C	< 250								
Post EVS Off-Gas Temp	° C	< 50								
Scrub Liquid Temp (SLT)	° C	<40								
Heat Xer Temp	° C	< 30								
Feed Pump Setting	%									
Feed Pump Control Mode	A or M									
Feed Pressure	psi	<1.0								
Feed Tank Wt	Kg	decreasing								
Overflow Temp	° C	1000 - 1100								
Discharge Can Temp	° C	750 - 850								
Glass Scale	Kg	< 10								
Alarm Condition	On/Off	off								
time	hh:mm									

Appendix B

Appendix B: Test Instruction for Preparing 100 L of Sodium Bearing Waste Simulant

TEST INSTRUCTION FOR PREPARING 100L OF SODIUM BEARING WASTE SIMULANT

Objective: To prepare a sodium bearing waste (SBW) melter feed simulant to use in PNNL RSM testing for INEEL.

Start Date: August 1, 2001

End Date: August 1, 2001

Laboratory Record Book Used 57721, pp. 60 to 65

This test instruction has been reviewed in collaboration with the Responsible Scientist.

Author: Renée Russell

Responsible Scientist: Ronald W. Geeb

Test Operator(s): [illegible] BDM
W. Buchmiller
Renée Russell

Balance Calibration Record:

Balance Calibration # 743886 Last Cal. 2-21-01 Due 8-01

Balance Calibration # 4375375 Last Cal. 8-30-00 Due 8-01

Chemical Inventory List:

Chemical	Manufacturer	Lot #	Amount Used
$NaNO_3$	NOAH	37467/3.1	17.50 kg
$Mn(NO_3)_2$ soln	Baker	32669	296.55g
$Mn(NO_3)_2$ soln	ChemWest	Bar Code# 49243	208.05g
KNO_3	Aldrich	01414JF	23.75g
KNO_3	Noah Tech.	22804/1.1	1959.35g
$Ca(NO_3)_2 \cdot 4H_2O$	Noah Tech	60765/1.1	1113.9g
$Cd(NO_3)_2 \cdot 4H_2O$	Aldrich	09911LN	23.25g
$Ni(NO_3)_2 \cdot 6H_2O$	Aldrich	00703EF	42.79g
ZrF_4	Alfa Products	070886	1.12g
$Cr(NO_3)_3 \cdot 9H_2O$	Fisher	973985B	43.43g
$Cr(NO_3)_3 \cdot 9H_2O$	Aldrich	07227AU	66.56g
$Ba(NO_3)_2$	ChemWest	Bar Code# 78011	1.47g
$Ce(NO_3)_3 \cdot 6H_2O$	Aldrich	06824DN	14.58g
$Co(NO_3)_2 \cdot 6H_2O$	Mallinckrodt	4544	0.55g
$Cu(NO_3)_2 \cdot 3H_2O$	Alfa	D05K32	16.85g
$Gd(NO_3)_3 \cdot 5H_2O$	Ventron Alfa	110674	7.68g
$Pb(NO_3)_2$	Allied Chem.	1838	39.51g
$Pb(NO_3)_2$	Fisher	510372	3.77g
TiO_2	Spectrum	42018A04	0.46g

Chemical Inventory List:

Chemical	Manufacturer	Lot #	Amount Used
$LiNO_3$	not listed	Bar Code# 49198	2.35g
$Mg(NO_3)_2 \cdot 6H_2O$	Mallinckrodt	6002	108.30g
$Mg(NO_3)_2 \cdot 6H_2O$	Baker	402125	119.46g
$Mg(NO_3)_2 \cdot 6H_2O$	Mallinckrodt	Bar Code# 74415	80.75g
$Ru(NO)(NO_3)_3$	not listed	Bar Code# 139163	18mL
$Ru(NO)(NO_3)_3$	Strem Chem.	134634-S	30mL
$Ru(NO)(NO_3)_3$	Aldrich	12715 JF	36.5mL
$Sr(NO_3)_2$	Johnson Matthey	C01E	143.1g
$Zn(NO_3)_2 \cdot 6H_2O$	Baker	641084	31.22g
KI	Chem West	Bar Code# 67867	2.16g
$CsNO_3$	Johnson Matthey	D15 D16	31.66g
$CsNO_3$	Alfa	L20E08	11.62g
H_3BO_3	Baker	F44704	2.80g
H_2MoO_4	Baker & Adamson	F043	3.12g
H_3PO_4	Fisher	001143	94mL
H_2SO_4	Fisher	007402	388mL
$Hg(NO_3)_2 \cdot 2H_2O$	Noah Tech	63009/1.1	72.66g
HBF_4	Baker	T40607	216.74g
$Fe(NO_3)_3 \cdot 9H_2O$	Noah Tech	21528/1.2	878.0g
HNO_3	Fisher	002648	2.5L
HCl	Mallinckrodt	2062 KVKY	245mL
HNO_3	EM	37346	2.5L

1. Add 504.6 g of 50% $Mn(NO_3)_2$ solution and 30.15 L of 2.2M $Al(NO_3)_3$ to an appropriate sized vessel.

Amount of $Mn(NO_3)_2$ solution added: 504.6 g

Amount of 2.2M $Al(NO_3)_3$ added: 38.85 kg (d = 1.29 g/mL)

2. Add the following salts to the solution in the amounts indicated:

Chemical	Target Wt.	Actual Wt.	Initial	Date
$NaNO_3$	17.49 kg	17.50 kg	[illegible]	8-1-01
KNO_3	1983.0 g	1983.1 g	RR	8-1-01
$Ca(NO_3)_2 \cdot 4H_2O$	1114.0 g	1113.9 g	RR	8-1-01
$Cd(NO_3)_2 \cdot 4H_2O$	23.26 g	23.25 g	RR	8-1-01
$Ni(NO_3)_2 \cdot 6H_2O$	42.78 g	42.79 g	RR	8-1-01
$Fe(NO_3)_3 \cdot 9H_2O$	877.9 g	878.0 g	RR	8-1-01
ZrF_4	1.11 g	1.12 g	RR	8-1-01
$Cr(NO_3)_3 \cdot 9H_2O$	110.0 g	109.99 g	RR	8-1-01
$Ba(NO_3)_2$	1.46 g	1.47 g	RR	8-1-01
$Ce(NO_3)_3 \cdot 6H_2O$	14.59 g	14.58 g	RR	8-1-01
$Co(NO_3)_2 \cdot 6H_2O$	0.56 g	0.55 g	RR	8-1-01
$Cu(NO_3)_2 \cdot 3H_2O$	16.84 g	16.85 g	RR	8-1-01
$Gd(NO_3)_3 \cdot 5H_2O$	7.68 g	7.68 g	RR	8-1-01
$Pb(NO_3)_2$	43.29 g	43.28 g	RR	8-1-01
$LiNO_3$	2.34 g	2.35 g	RR	8-1-01
$Mg(NO_3)_2 \cdot 6H_2O$	308.5 g.	308.51 g	RR	8-1-01

Chemical	Target Wt.	Actual Wt.	Initial	Date
$Ru(NO)(NO_3)_3$	84.3 mL	84.5mL	RR	8-1-01
$Sr(NO_3)_2$	143.1 g	143.10g	RR	8-1-01
TiO_2 ~~$TiCl_4$~~	0.46 ~~1.10~~ g	0.46g	RR	8-1-01
$Zn(NO_3)_2 \cdot 6H_2O$	31.21 g	31.22g	RR	8-1-01
KI	2.16 g	2.16g	RR	8-1-01
$CsNO_3$	43.27 g	43.28g	RR	8-1-01
$Hg(NO_3)_2 \cdot 2H_2O$ RR 8-1-01	72.66 ~~69.04~~ g RR 8-1-01	72.66g	RR	8-1-01

2. Add just enough DI water to dissolve 100% of the salts. Stir and heat as needed.

   Actual amount of DI water added: 10 L

3. Add 2.80 g H_3BO_3. Dissolve as much as possible by heating and stirring as necessary.

   Amount of H_3BO_3 added: 2.80g

4. Discontinue heating the simulant. Add 216.74 g of 48 wt% HBF_4 solution. Stir well.

   Amount of 48 wt%HBF_4 solution added: 216.74g

5. In a separate vessel, add about 1800 mL of DI water. Slowly add 387.7 mL of 18M H_2SO_4 to the water and mix thoroughly.

   Amount of DI water added: 30 L Amount of 18M H_2SO_4 added: 388 mL

6. Slowly add the H_2SO_4 solution to the solution from Step 1 while stirring vigorously.

7. Add DI water to the solution to bring the total solution volume to about 80 L.

   Amount of DI water added: N/A

8. In a separate vessel, combine 244.9 mL 12M HCl, 93.8 mL 14.6M H_3PO_4 and 5 L 15.4M HNO_3.

   Amount of HCl added: 245 mL Amount of HNO_3 added: 5 L

Amount of H_3PO_4 added: 94 mL

9. Slowly add this mixture of acids to 10 L of DI water.

   Amount of DI water added: N/A

10. Slowly add the diluted acid mixture to the 80 L solution.

11. Add 3.12 g H_2MoO_4 to the solution.

    Amount of H_2MoO_4 added: 3.12 g

12. Add DI water (approximately 10 L) to the combined solution, bringing the final volume to 100 L.

    Amount of DI water added: Filled to 100 L graduation

    Final slurry volume: 100 L

TEST INSTRUCTION FOR PREPARING 60L OF SODIUM BEARING WASTE SIMULANT

Objective: To prepare a sodium bearing waste (SBW) melter feed simulant to use in PNNL RSM testing for INEEL.

Start Date: August 8, 2001

End Date: August 8, 2001

Laboratory Record Book Used 57721, pp. 676 [illegible]

This test instruction has been reviewed in collaboration with the Responsible Scientist.

Author: Renée Russell

Responsible Scientist: [illegible]

Test Operator(s): Renée Russell

[illegible]

Balance Calibration Record:

Balance Calibration # 743886 Last Cal. 2-21-01 Due 8-01

Balance Calibration # H375375 Last Cal. 8-30-00 Due 8-01

Chemical Inventory List:

Chemical	Manufacturer	Lot #	Amount Used
$NaNO_3$	Noah Tech	37467/3.1	10.5 kg
$Al(NO_3)_3$ soln	Noah Tech	20505/1.1	23.35 kg
$Mn(NO_3)_2$ soln	Chem West	Bar Code # 49243	302.8 g
KNO_3	Noah Tech	22804/1.1	1190.0 g
$Ca(NO_3)_2 \cdot 4H_2O$	Noah Tech	60765/1.1	668.6 g
$Cd(NO_3)_2 \cdot 4H_2O$	Aldrich	09911LN	13.96 g
$Ni(NO_3)_2 \cdot 6H_2O$	Aldrich	00703EF	25.66 g
ZrF_4	Alfa	070886	0.67 g
$Cr(NO_3)_3 \cdot 9H_2O$	Aldrich	07227AU	66.0 g
$Ba(NO_3)_2$	Chem West	Bar Code # 78011	0.87 g
$Ce(NO_3)_3 \cdot 6H_2O$	Aldrich	06824DN	8.75 g
$Co(NO_3)_2 \cdot 6H_2O$	Mallinckrodt	4544	0.35 g
$Cu(NO_3)_2 \cdot 3H_2O$	Alfa Aesar	D05K32	10.1 g
$Gd(NO_3)_3 \cdot 5H_2O$	Ventron Alfa	110674	4.62 g
$Pb(NO_3)_2$	Allied Chem	1838	25.98 g
~~FiO_2~~			AR 8-8-01
$LiNO_3$	not listed	bar code # 49198	1.40 g

Chemical Inventory List:

Chemical	Manufacturer	Lot #	Amount Used
$Sr(NO_3)_2$	Johnson Matthey	C01E	85.84g
$Zn(NO_3)_2 \cdot 6H_2O$	Baker	641084	18.72 g
TiO_2	Spectrum	42018A04	0.28g
$Ru(NO)(NO_3)_3$	Aldrich	12715JF	51ml
$Hg(NO_3)_2 \cdot 2H_2O$	Noah Tech	63009/1.1	43.60 g
$Fe(NO_3)_3 \cdot 9H_2O$	Noah Tech	~~21528/1.2~~	527.6g
$Mg(NO_3)_2 \cdot 6H_2O$	Mallinckrodt	Bar Code# 74415	84.30g
$Mg(NO_3)_2 \cdot 6H_2O$	Fisher	004700	100.80g
KI	Argent Chem	800808	1.29g
$CsNO_3$	Alfa Aesar	L20E08	25.96g
H_3BO_3	Baker	F44704	1.68g
H_2MoO_4	B&A	F043	1.87g

1. Add 302.8 g of 50% $Mn(NO_3)_2$ solution and 18.09 L of 2.2M $Al(NO_3)_3$ to an appropriate sized vessel.

Amount of $Mn(NO_3)_2$ solution added: 302.8g

Amount of 2.2M $Al(NO_3)_3$ added: 18.1L

2. Add the following salts to the solution in the amounts indicated:

Chemical	Target Wt.	Actual Wt.	Initial	Date
$NaNO_3$	10.50 kg	10.5 Kg	RR	8-8-01
KNO_3	1190.0 g	1190.0g	RR	8-8-01
$Ca(NO_3)_2 \cdot 4H_2O$	668.60 g	668.6g	RR	8-8-01
$Cd(NO_3)_2 \cdot 4H_2O$	13.96 g	13.96g	RR	8-8-01
$Ni(NO_3)_2 \cdot 6H_2O$	25.67 g	25.66g	RR	8-8-01
$Fe(NO_3)_3 \cdot 9H_2O$	526.7 g	527.6g	RR	8-8-01
ZrF_4	0.67 g	0.67g	RR	8-8-01
$Cr(NO_3)_3 \cdot 9H_2O$	65.99 g	66.0g	RR	8-8-01
$Ba(NO_3)_2$	0.87 g	0.87g	RR	8-8-01
$Ce(NO_3)_3 \cdot 6H_2O$	8.75 g	8.75g	RR	8-8-01
$Co(NO_3)_2 \cdot 6H_2O$	0.34 g	0.35g	RR	8-8-01
$Cu(NO_3)_2 \cdot 3H_2O$	10.10 g	10.1g	RR	8-8-01
$Gd(NO_3)_3 \cdot 5H_2O$	4.61 g	4.62g	RR	8-8-01
$Pb(NO_3)_2$	25.97 g	25.98g	RR	8-8-01
$LiNO_3$	1.40 g	1.40g	RR	8-8-01
$Mg(NO_3)_2 \cdot 6H_2O$	185.1 g	185.1g	RR	8-8-01

Chemical	Target Wt.	Actual Wt.	Initial	Date
$Ru(NO)(NO_3)_3$	50.6 mL	51mL	RR	8-8-01
$Sr(NO_3)_2$	85.84 g	85.84g	RR	8-8-01
TiO_2	0.28 g	0.28g	RR	8-8-01
$Zn(NO_3)_2 \cdot 6H_2O$	18.72 g	18.72g	RR	8-8-01
KI	1.29 g	1.29g	RR	8-8-01
$CsNO_3$	25.96 g	25.96g	RR	8-8-01
$Hg(NO_3)_2 \cdot 2H_2O$	~~41.42~~ 43.60 g	43.60g	RR	8-8-01

2. Add just enough DI water to dissolve 100% of the salts. Stir and heat as needed.

   Actual amount of DI water added: 10L

3. Add 1.68 g H_3BO_3. Dissolve as much as possible by heating and stirring as necessary.

   Amount of H_3BO_3 added: 1.68g

4. Discontinue heating the simulant. Add 130.04 g of 48 wt% HBF_4 solution. Stir well.

   Amount of 48 wt%HBF_4 solution added: 130.1g

5. In a separate vessel, add about 1800 mL of DI water. Slowly add 232.6 mL of 18M H_2SO_4 to the water and mix thoroughly.

   Amount of DI water added: X RR 8-8-01 Amount of 18M H_2SO_4 added: 233mL

6. Slowly add the H_2SO_4 solution to the solution from Step 1 while stirring vigorously.

7. Add DI water to the solution to bring the total solution volume to about 45 L.

   Amount of DI water added: 10L

8. In a separate vessel, combine 146.9 mL 12M HCl, 56.3 mL 14.6M H_3PO_4 and 3 L 15.4M HNO_3.

   Amount of HCl added: 147mL Amount of HNO_3 added: 3L

Amount of H_3PO_4 added: 56mL

9. Slowly add this mixture of acids to 10 L of DI water.

   Amount of DI water added: X RR 8-8-01

10. Slowly add the diluted acid mixture to the 45 L solution.

11. Add 1.87 g H_2MoO_4 to the solution.

    Amount of H_2MoO_4 added: 1.87g

12. Add DI water (approximately 10 L) to the combined solution, bringing the final volume to 60 L.

    Amount of DI water added: not measured. RR 8-8-01

    Final slurry volume: 60L

Appendix C

RSM Feed Batching and Manual Data Sheets

RSM -01-2 INEEL SBW Run #1 Feed Batching Sheet

Batch Sheet No. RSM-01-2- 1

Prepared by: R.L. Russell

Date: 08/06/2001 Time: 6:00

Reductant (select):	Sugar	x	Glycolic	
Glass Oxidation	Low	x	High	

Note: shaded areas require value to be input

SBW target volume, Liters:	30			
SBW transfer tank gross wt:	39.8	kg	0	kg
SBW transfer tank tare wt:	2.30	kg	2.30	kg
SBW transfer tank net wt:	37.50	kg	0.00	kg
SBW Density:	1.25	kg/L		

Calculated volumed transferred: Net wt. / density = 30 Liters

Target glass waste oxide fraction: 0.2

Glass Former Addition Calculation

Glass former weights: 123.1 gm WO/L * (1 gm glass/ B-gm WO) * (C-gm total GFO/ gm glass) * E-L SBW * (gm GF chemical / gm total GFO)

Waste oxide loading, g/L:	123.1	= A
Fraction waste oxide loading:	0.2	= B
Fraction glass former loading:	0.8	= C
SBW Volume, liters	30	= D
(A/B) * C * D =	14772.00	= E

Glass formers	F		Batch totals: (E * F)	Initial after weighing
gm silica / gm total GFO =	0.680	=>	10037.6 grams	10.04 kg RR
gm LiOH*H2O / gm total GFO =	0.172	=>	2535.3 grams	2535.3 g RR
gm boric acid / gm total GFO =	0.107	=>	1581.6 grams	1581.6 g RR
gm Ca(OH)2 / gm total GFO =	0.066	=>	979.7 grams	979.7 g RR
gm NaOH / gm total GFO =	0.055	=>	817.9 grams	817.9 g RR
gm V2O5/ gm total GFO =	0.049	=>	720.9 grams	720.9 g RR
gm Mg(OH)2 / gm total GFO =	0.025	=>	374.1 grams	374.1 5 g RR
gm ZrO2/ gm total GFO =	0.024	=>	360.4 grams	360.4 g RR
gm Fe2O3 / gm total GFO =	0.015	=>	224.5 grams	224.5 g RR
gm Sugar/L SBW=	180	=>	5400.0 grams	5400.0 g RR
ml HNO3/L SBW=	60	=>	1800.0 cc	1800 mL RR

Initial melter feed tank weight: Ø kg Time: ~ 0930 WCB

Melter feed tank wt. after SBW addition: 34.5 kg Time: ~ 0945 WCB

Melter feed tank wt. after glass formers addition: 58.0 kg Time: 1028 WCB

Completed by (sign & data): WC Budwillen 10/6/01 1033

Reviewed & Approved by: RW Zeh 2/12/02

RSM -01-2 INEEL SBW Run #1 Feed Batching Sheet

Batch Sheet No. RSM-01-2-~~12~~ RR 8-7-01

Prepared by: R.L. Russell

Date: 08/07/2001 Time: 9:00

Reductant (select):	Sugar	x	Glycolic	
Glass Oxidation	Low	x	High	

SBW target volume, Liters: 30

Note: shaded areas require value to be input

SBW transfer tank gross wt:	41.65	kg	0	kg
SBW transfer tank tare wt:	2.30	kg	2.30	kg
SBW transfer tank net wt:	39.35	kg	0.00	kg

SBW Density: 1.25 kg/L

Calculated volumed transferred: Net wt. / density = 31.48 Liters

Target glass waste oxide fraction: 0.2

Glass Former Addition Calculation

Glass former weights: 123.1 gm WO/L * (1 gm glass/ B-gm WO) * (C-gm total GFO/ gm glass) * E-L SBW * (gm GF chemical / gm total GFO)

Waste oxide loading, g/L:	123.1	= A
Fraction waste oxide loading:	0.2	= B
Fraction glass former loading:	0.8	= C
SBW Volume, liters	31.48	= D
(A/B) * C * D =	15500.75	= E

Glass formers	F		Batch totals: (E * F)	Initial after weighing
gm silica / gm total GFO =	0.680	=>	10532.8 grams	10532.7g RR
gm LiOH*H2O / gm total GFO =	0.172	=>	2660.4 grams	2660 kg RR
gm boric acid / gm total GFO =	0.107	=>	1659.6 grams	1659.6g RR
gm Ca(OH)2 / gm total GFO =	0.066	=>	1028.1 grams	1028.1g RR
gm NaOH / gm total GFO =	0.055	=>	858.3 grams	858.3g RR
gm V2O5/ gm total GFO =	0.049	=>	756.4 grams	756.3g RR
gm Mg(OH)2 / gm total GFO =	0.025	=>	392.5 grams	392.5g RR
gm ZrO2/ gm total GFO =	0.024	=>	378.2 grams	378.3g RR
gm Fe2O3 / gm total GFO =	0.015	=>	235.6 grams	235.6g RR
gm Sugar/L SBW=	199	=>	6264.5 grams	6.265kg RR
ml HNO3/L SBW=	100	=>	3148.0 cc	3.15L RR

Initial melter feed tank weight: 22.5 kg Time: 0838

Melter feed tank wt. after SBW addition: 64.0 kg Time: 0838

Melter feed tank wt. after glass formers addition: kg Time:

Completed by (sign & data): WC Buchwalter 2/12/02

Reviewed & Approved by: RW Zeh 2/12/02

RSM -01-2 INEEL SBW Run #1 Feed Batching Sheet

Batch Sheet No. RSM-01-2- 3

Prepared by: R.L. Russell

Date: 08/08/2001 Time: ~~0:00~~ 14:30

Reductant (select): Sugar [ ] Glycolic [ ]
Glass Oxidation Low [ ] High [ ]

SBW target volume, Liters: 30

Note: shaded areas require value to be input

SBW transfer tank gross wt:	39.15	kg	0	kg
SBW transfer tank tare wt:	5.65	kg	2.30	kg
SBW transfer tank net wt:	33.50	kg	0.00	kg

SBW Density: 1.25 kg/L

Calculated volumed transferred: Net wt. / density = 26.8 Liters

Target glass waste oxide fraction: 0.2

Glass Former Addition Calculation

Glass former weights: 123.1 gm WO/L * (1 gm glass/ B-gm WO) * (C-gm total GFO/ gm glass) * E-L SBW * (gm GF chemical / gm total GFO)

Waste oxide loading, g/L:	123.1	= A
Fraction waste oxide loading:	0.2	= B
Fraction glass former loading:	0.8	= C
SBW Volume, liters	26.8	= D
(A/B) * C * D =	13196.32	= E

Glass formers	F		Batch totals: (E * F)	Initial after weighing
gm silica / gm total GFO =	0.680	=>	8966.9 grams	8.965 kg RR
gm LiOH*H2O / gm total GFO =	0.172	=>	2264.9 grams	2265 kg RR
gm boric acid / gm total GFO =	0.107	=>	1412.9 grams	1412.9 g RR
gm Ca(OH)2 / gm total GFO =	0.066	=>	875.2 grams	875.2 g RR
gm NaOH / gm total GFO =	0.055	=>	730.7 grams	730.7 g RR
gm V2O5/ gm total GFO =	0.049	=>	644.0 grams	644.0 g RR
gm Mg(OH)2 / gm total GFO =	0.025	=>	334.2 grams	334.3 g RR
gm ZrO2/ gm total GFO =	0.024	=>	322.0 grams	322.1 g RR
gm Fe2O3 / gm total GFO =	0.015	=>	200.6 grams	200.6 g RR
gm Glycolic/L SBW=	0	=>	0.0 grams	RR
ml HNO3/L SBW=	~~60~~ 100 RR	=>	~~1608.0~~ 2680 cc	2680 mL

Initial melter feed tank weight: _____ kg Time: _____

Melter feed tank wt. after SBW addition: _____ kg Time: _____

Melter feed tank wt. after glass formers addition: _____ kg Time: _____

Completed by (sign & data): W C Buchmiller 2/12/02

Reviewed & Approved by: R W Zeh 2/12/02

RSM -01-2 INEEL SBW Run #1 Feed Batching Sheet

Batch Sheet No. RSM-01-2- 4

Prepared by: R.L. Russell

Date: 08/09/2001 Time: 18:15 0:00

Reductant (select): Sugar Glycolic
Glass Oxidation Low High

SBW target volume, Liters: 30

Note: shaded areas require value to be input

SBW transfer tank gross wt:	39.2	kg	0	kg
SBW transfer tank tare wt:	5.55	kg	2.30	kg
SBW transfer tank net wt:	33.65	kg	0.00	kg

SBW Density: 1.25 kg/L

Calculated volumed transferred: Net wt. / density = 26.92 Liters

Target glass waste oxide fraction: 0.2

Glass Former Addition Calculation

Glass former weights: 123.1 gm WO/L * (1 gm glass/ B-gm WO) * (C-gm total GFO/ gm glass) * E-L SBW * (gm GF chemical / gm total GFO)

Waste oxide loading, g/L:	123.1	= A
Fraction waste oxide loading:	0.2	= B
Fraction glass former loading:	0.8	= C
SBW Volume, liters	26.92	= D
(A/B) * C * D =	13255.41	= E

Glass formers	F		Batch totals: (E * F)		Initial after weighing
gm silica / gm total GFO =	0.680	=>	9007.0	grams	9.005 Kg RR
gm LiOH*H2O / gm total GFO =	0.172	=>	2275.0	grams	2.275 Kg RR
gm boric acid / gm total GFO =	0.107	=>	1419.2	grams	1419.2 g RR
gm Ca(OH)2 / gm total GFO =	0.066	=>	879.1	grams	879.0 g RR
gm NaOH / gm total GFO =	0.055	=>	733.9	grams	734.0 g RR
gm V2O5/ gm total GFO =	0.049	=>	646.9	grams	646.9 g RR
gm Mg(OH)2 / gm total GFO =	0.025	=>	335.7	grams	335.7 g RR
gm ZrO2/ gm total GFO =	0.024	=>	323.4	grams	323.5 g RR
gm Fe2O3 / gm total GFO =	0.015	=>	201.5	grams	201.5 g RR
gm Glycolic/L SBW=	0	=>	0.0	grams	RR
ml HNO3/L SBW=	100	=>	2692.0	cc	2692 mL RR

Initial melter feed tank weight: _____ kg Time: _____

Melter feed tank wt. after SBW addition: _____ kg Time: _____

Melter feed tank wt. after glass formers addition: _____ kg Time: _____

Completed by (sign & data): WC Buchmelly 2/12/02

Reviewed & Approved by: RW [illegible] 2/12/02

Data Sheet #1: Priority & Electrical Data

RSM-01-2										
	By:		[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	Date:		8/6/01	8/6/01	8/6/01	8/6/01	8/6/01	8/6/01	8/6/01	8/7/01
	Time		16:21	18:57	20:04	21:57	22:56	23:09	23:57	0119
DESCRIPTION	UNITS	Range								
Priority Data										
Melt Temperature (T1, control)	° C	1125 - 1175	1098	1149	1151	1149	1148	1152	1149	1149
Melt Temperature (T2)	° C	1125 - 1175	1091	1142	1145	1147	1148	1156	1154	1159
Plenum Temperature	° C	400 - 600	723	726	657	726	657	623	596	605
Feed pump setting	%		20	15 *	15	15	15	15	14	14
Cold Cap Coverage	%	> 75	50	10	90	50	80	80	90	90
Slurry Pool Coverage of Cap	%		50	10	20	20	—	—	—	—
Cold Cap Flexibility Visible	y or n		y	y	y	y	y	y	y	y
Number of Vents	#		N/A	N/A	3	N/A	—	—	—	3
Cold Cap Thickness	inch		0.2	0.2	1	½	—	—	—	0.5-1"
Phase Separation (Note in LRB)	y or n		n	n	n	n	n	n	n	n
Glass Pouring	y or n		n	n	n	n	n	n	n	Y
Electrical Data										
Electrode Potential	Volts		36.7	30.9	24.8	28.6	25.4	24.3	25.9	24.5
Electrode Current	Amps		141.7	123.1	112.0	120.6	117.3	113.1	116	113
Electrode Power	kW		5.4	3.9	2.8	3.4	3.0	2.8	2.9	2.8
Melt Resistance	Ω		0.259	0.251	0.221	0.237	0.217	0.215		0.222
Melt (Electrode) Setpoint Temp	° C		1120	1150	1149	1150	1150	1150	1150	1150
(Electrode Power) Output	%		71	62	62	61	58	57	59	57
(Electrode) Control Mode	A or M		A	A	A	A	A	A	A	A
Kiln Power	kW		0.1	0.3	0.3	0.1	0.2	0.2	0.3	0.3
Kiln Temp Setpoint	° C		850	850	850	850	850	850	850	850
Kiln Actual (Middle) Temp	° C		805	852	851	853	850	850	849	851
Kiln Power Output	%		17	33	33	23	29	31	33	33
Kiln Control Mode	A or M		A	A	A	A	A	A	A	A
Discharge Can Power	kW		0.2	0.2	0.1	0.2	0.2	0.2	0.1	0.2
Discharge Can Temp Setpoint	° C		800	801	800	800	800	800	800	800
Discharge Can Actual Temp	° C		800	801	815	799	801	807	801	802
Discharge Can Power Output	%		72	68	45	75	70	68	56	63
Overflow Heater Power	kW		2.0	1.7	1.3	1.7	1.7	1.6	1.6	1.5
Overflow Heater Setpoint	° C		1050	1050	1050	1050	1050	1050	1050	1050
Overflow Heater Power Output	%		70	66	56	65	70	62	62	61
Time	hh:mm		16:28	19:09	20:16	21:01	22:10	23:12	0001	0122

*feed restarted at 18:55

Reviewed and Approved: [signature]

Date: 8/7/01

sheet 1 of 10

Data Sheet #1: Priority & Electrical Data

RSM-01-2										
	By:			RJJ	[illegible]	[illegible]	RJJ	RJJ	[illegible]	[illegible]
	Date:			8/7/01	8/7/01	8/7/01	8/7/01	8/7/01	8/7/01	8/7/01
	Time			0212	0304	0417	0515	0613	0701	0845
DESCRIPTION	UNITS	Range								
Priority Data										
Melt Temperature (T1, control)	° C	1125 - 1175		1147	1154	1152	1151	1149	1151	1155
Melt Temperature (T2)	° C	1125 - 1175		1142	1155	1156	1157	1154	1166	1182
Plenum Temperature	° C	400 - 600		632	625	637	611	601	596	684
Feed pump setting	%			15	16	16	16	16	16	16
Cold Cap Coverage	%	> 75		95	95	80	95	95	95	75
Slurry Pool Coverage of Cap	%			?	?	?	100	100	100	80
Cold Cap Flexibility Visible	y or n			n	N	Y	Y	Y	Y	Y
Number of Vents	#			0	1	1	lots	lots	1	2
Cold Cap Thickness	inch			?	?	1/2	1/2+	1?	?	1/4"
Phase Separation (Note in LRB)	y or n			n	NO	NO	?	?	?	NO
Glass Pouring	y or n			Y	Y	Y	Y	Y	Y	~~N~~ Y
Electrical Data										
Electrode Potential	Volts			25	25	25	25	26	84.4	25.9
Electrode Current	Amps			108	106	115	110	110	98.4	118
Electrode Power	kW			2.6	2.6	2.9	2.7	2.8	2.4	3.0
Melt Resistance	Ω			.236	0.233	0.222	.226	0.235	0.248	0.222
Melt (Electrode) Setpoint Temp	° C			1150	1150	1150	1150	1150	1150	1150
(Electrode Power) Output	%			54	53	57	54	54	49	59
(Electrode) Control Mode	A or M			A	A	A	A	A	A	A
Kiln Power	kW			0.3	0.3	0.2	0.2	0.3	0.2	0.1
Kiln Temp Setpoint	° C			850	850	850	850	850	850	850
Kiln Actual (Middle) Temp	° C			850	850	851	850	850	850	858
Kiln Power Output	%			35	33	27	31	32	31	18
Kiln Control Mode	A or M			A	A	A	A	A	A	A
Discharge Can Power	kW			0.2	0.2	0.2	0.2	0.2	0.2	0.3
Discharge Can Temp Setpoint	° C			800	800	800	800	800	800	800
Discharge Can Actual Temp	° C			800	800	801	800	800	800	782
Discharge Can Power Output	%			65	65	66	65	63	60	85
Overflow Heater Power	kW			1.6	1.7	1.5	1.6	1.5	1.4	1.9
Overflow Heater Setpoint	° C			1050	1050	1050	1050	1050	1050	1050
Overflow Heater Power Output	%			62	64	60	62	59	59	69
Time	hh:mm			0218	0309	0421	0519	0616	0708	0850

Reviewed and Approved: [signature]

Date: 8/7/01

sheet 2 of 10

Data Sheet #1: Priority & Electrical Data

RSM-01-2										
	By:		TDP	TDP	TDP	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	Date:		8-7-01	8-7-01	8-7-01	8/7/01	8/7/01	8/7/01	8/7/01	8/7/01
	Time		1015	1200	1500	1600	1704	17:59	2100	2159
DESCRIPTION	UNITS	Range								
Priority Data										
Melt Temperature (T1, control)	° C	1125 - 1175	1149	1096	1156	1161	1142	1149	1065	1084
Melt Temperature (T2)	° C	1125 - 1175	1171	1107	1159	1198	1163	1173	1065	1077
Plenum Temperature	° C	400 - 600	703	804	804	636	591	610	634	630
Feed pump setting	%		14	0	13	15	14*	14	13	9.0
Cold Cap Coverage	%	> 75	50	0	10	100	100	Foam	80%	50%
Slurry Pool Coverage of Cap	%		10	0	—	60	—	—	60%	50%
Cold Cap Flexibility Visible	y or n		Y	N	—	Y?	N	Foam	NO	NO
Number of Vents	#		2	0	—	2	0	—	0	0
Cold Cap Thickness	inch		Thin	0	—	1/2?	>1	—	1"	1/2-1"
Phase Separation (Note in LRB)	y or n		N	N	—	?	—	N	N	N
Glass Pouring	y or n		N	N	N	N	N	N	N	Y
Electrical Data										
Electrode Potential	Volts		26.7	8.9	26.3	20.6	22.7	24.2	22.7	29.0
Electrode Current	Amps		119	31	117	91.9	86.7	82.3	73.3	82.6
Electrode Power	kW		3.2	0.2	2.9	1.9	2.0	2.0	1.7	2.4
Melt Resistance	Ω		0.223	0.266	0.223	.229	.262	.294	.310	.350
Melt (Electrode) Setpoint Temp	° C		1150	1050	1150	1140	1140	1145	—	—
(Electrode Power) Output	%		60	15	58	45	43	41	37	42
(Electrode) Control Mode	A or M		A	A	A	A	A	A	M	M
Kiln Power	kW		0	0	0	0	0.1	0.1	0.4	0.3
Kiln Temp Setpoint	° C		850	850	850	850	850	850	850	850
Kiln Actual (Middle) Temp	° C		850	850	850	853	846	852	847	851
Kiln Power Output	%		0	0	0	0	15	21	41	34
Kiln Control Mode	A or M		A	A	A	A	A	A	A	A
Discharge Can Power	kW		0.4	0.3	0.3	.3	0.3	0.3	0.3	0.2
Discharge Can Temp Setpoint	° C		800	800	800	800	800	800	800	800
Discharge Can Actual Temp	° C		773	799	792	794	796	792	800	802
Discharge Can Power Output	%		90	90	90	90	90	90	89%	73
Overflow Heater Power	kW		1.9	2.0	1.7	1.8	1.9	1.9	1.9	1.8
Overflow Heater Setpoint	° C		1050	1050	1050	1050	1050	1050	1050	1050
Overflow Heater Power Output	%		69	69	69	68	69	68	69	65
Time	hh:mm		1015	1157	1501		17:10	18:07	2105	2205

*Turned down to 14% at 17:04

Reviewed and Approved: [signature]

Date: 7/12/02

sheet 3 of 10

Data Sheet #1: Priority & Electrical Data

RSM-01-2										
	By:		[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	Date:		8/7/01	8/8/01	8/8/01	8/8/01	8/8/01	8/8/01	8/8/01	8/8/01
	Time		23:02	0200	0310	0405	0501	0600	0719	0813
DESCRIPTION	UNITS	Range								
Priority Data										
Melt Temperature (T1, control)	° C	1125 - 1175	1113	1149	1150	1147	1149	1148	1150	1149
Melt Temperature (T2)	° C	1125 - 1175	1130	1164	1171	1142	1161	1156	1152	1157
Plenum Temperature	° C	400 - 600	679	813	709	634	595	583	562	581
Feed pump setting	%		11	0	18	18%	15%	16%	16%	16%
Cold Cap Coverage	%	> 75	50	0	60	50	75	75	90%	80%
Slurry Pool Coverage of Cap	%		—	—	50	50	50	50	50	50
Cold Cap Flexibility Visible	y or n		N	—	Y	Y	Y	Y	Y	Y
Number of Vents	#		—	—	3-5	5	3	3-5	1-2	3
Cold Cap Thickness	inch		1	—	1/2	1/2	1/2	1/2	1/2	1/2
Phase Separation (Note in LRB)	y or n		—	NO	NO	N	N	N	N	N
Glass Pouring	y or n		N	NO	NO	Y	Y	N	Y	Y
Electrical Data										
Electrode Potential	Volts		27.4	25.0	25	25.5	24.7	25	25	25.7
Electrode Current	Amps		102.3	122.0	116	125	123	123	123	124
Electrode Power	kW		2.7	3.1	2.9	3.2	3.0	3.2	3.0	3.1
Melt Resistance	Ω		0.277	0.201	0.214	0.203	0.202	0.204	0.201	0.203
Melt (Electrode) Setpoint Temp	° C		1110	1150	1150	1150	1150	1150	1150	1150
(Electrode Power) Output	%		50	62	57	62	61	62	61	61
(Electrode) Control Mode	A or M		A	A	A	A	A	A	A	A
Kiln Power	kW		0.5	0.0	0.0	0.1	0.2	0.2	0.2	0.2
Kiln Temp Setpoint	° C		850	850	850	850	850	850	850	850
Kiln Actual (Middle) Temp	° C		853	860	853	846	851	850	850	850
Kiln Power Output	%		43	0	0	25	30	30	30	25
Kiln Control Mode	A or M		A	A	A	A	A	A	A	A
Discharge Can Power	kW		0.3	0.3	0.3	0.3	0.3	0.3	0.4	0.4
Discharge Can Temp Setpoint	° C		800	800	800	800	800	800	800	800
Discharge Can Actual Temp	° C		800	799	801	800	801	801	735	775
Discharge Can Power Output	%		88	88	84	85	85	78	90	90
Overflow Heater Power	kW		2.0	1.8	1.9	1.8	1.6	1.9	1.8	1.6
Overflow Heater Setpoint	° C		1050	1050	1050	1050	1050	1050	1050	1050
Overflow Heater Power Output	%		70	67	64	67	61	67	66	63
Time	hh:mm		2308	0205	0313	0409	0503	0601	0722	0815

Reviewed and Approved: [signature] Date: 2/12/02 sheet 4 of 10

Data Sheet #1: Priority & Electrical Data

RSM-01-2										
	By:		TDP	LXB	TDP	TDP	TDP	LDJ	LDJ	LDJ
	Date:		8-8-01	8/8/01	8/8/01	8/8/01	8/8/01	8/8/01	8/8/01	8/8/01
	Time		1012	1110	1215	1352	1523	1600	1726	2000
DESCRIPTION	UNITS	Range								
Priority Data										
Melt Temperature (T1, control)	° C	1125 - 1175	1154	1149	1143	1157	1149	1153	1150	1159
Melt Temperature (T2)	° C	1125 - 1175	1156	1157	1147	1156	1155	1157	1150	1164
Plenum Temperature	° C	400 - 600	578	568	636	599	606	597	696	716
Feed pump setting	%		15	15	15	15	15	15	0	15
Cold Cap Coverage	%	> 75	90	80	70%	80%	80%	60%	0	25%
Slurry Pool Coverage of Cap	%		20	80	80	50%	40	—	0	25%
Cold Cap Flexibility Visible	y or n		Y	N	N	N	N	—		Y
Number of Vents	#		2	0	0	0	0	0	Feed off	0
Cold Cap Thickness	inch		1	1/2	1/2	1	1/2	1/2		1/2
Phase Separation (Note in LRB)	y or n		N	N	N	N	N	N		N
Glass Pouring	y or n		Y	Y	N	Y	N	N		N
Electrical Data										
Electrode Potential	Volts		24.3	24.8	26	25.3	24.5	24.6	23.9	26.0
Electrode Current	Amps		121	123.7	132	132	124	128	129.5	135.0
Electrode Power	kW		2.9	3.1	3.4	3.4	3.2	3.2	3.0	3.4
Melt Resistance	Ω		0.200	0.200	0.197	0.188	0.196	0.194	0.191	0.193
Melt (Electrode) Setpoint Temp	° C		1150	1150	1150	1150	1150	1150	1150	1150
(Electrode Power) Output	%		60	61	65	66	63	64	61	65
(Electrode) Control Mode	A or M		A	A	A	A	A	A	A	A
Kiln Power	kW		0.2	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Kiln Temp Setpoint	° C		850	850	850	850	850	850	850	850
Kiln Actual (Middle) Temp	° C		851	848	853	849	849	851	851	855
Kiln Power Output	%		25	21	17	22	23	22	22	17
Kiln Control Mode	A or M		A	A	A	A	A	A	A	A
Discharge Can Power	kW		0.3	0.4	0.3	0.3	0.3	0.3	0.3	0.3
Discharge Can Temp Setpoint	° C		800	800	800	800	800	800	800	800
Discharge Can Actual Temp	° C		790	790	766	801	801	801	801	725
Discharge Can Power Output	%		90	90	90	90	86	85	84	90
Overflow Heater Power	kW		1.7	1.8	1.7	1.7	1.8	1.7	1.7	2.0
Overflow Heater Setpoint	° C		1050	1050	1050	1050	1050	1050	1050	1050
Overflow Heater Power Output	%		64	66	64	65	66	1051	1049	1044
Time	hh:mm		1015	1113	1215	1400	1504	1614	1730	2007

Reviewed and Approved: [signature] Date: 8/22/02

sheet 5 of 10

Data Sheet #1: Priority & Electrical Data

RSM-01-2										
	By:		TDP	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	Date:		8-8-01	8/8/01	8/8/01	8/9/01	8/9/01	8/9/01	8/9/01	8/9/01
	Time		2110	2158	2303	0006	0103	0207	0311	0414
DESCRIPTION	UNITS	Range								
Priority Data										
Melt Temperature (T1, control)	° C	1125 - 1175	1150	1150	1150	1156	1150	1149	1152	1149
Melt Temperature (T2)	° C	1125 - 1175	1156	1158	1157	1159	1154	1159	1164	1164
Plenum Temperature	° C	400 - 600	587	561	570	563	549	577	589	607
Feed pump setting	%		15	15	15	13	13	13	13.	12
Cold Cap Coverage	%	> 75	85	85	85	90	95	90	90	60
Slurry Pool Coverage of Cap	%		60	65	60	60	50	50	50	100
Cold Cap Flexibility Visible	y or n		N	N	N	N	N	N	N	N
Number of Vents	#		0	1	0	0	0	0	0	0
Cold Cap Thickness	inch		65	1"	1.25	3"	1-3"	1-3"	1-3"	1"
Phase Separation (Note in LRB)	y or n		N	N	N	N	N	N	N	N
Glass Pouring	y or n		Y	Y	drips	drips	Y	N	N	N
Electrical Data										
Electrode Potential	Volts		23	22.9	22.7	21.8	22	22.6	22.5	22
Electrode Current	Amps		128	127.2	125.0	122.6	120	120.2	125.3	126
Electrode Power	kW		3.1	3.0	2.8	2.7	2.6	2.8	2.8	2.8
Melt Resistance	Ω		0.181	.181	.183	.182	.184	0.186	0.180	0.178
Melt (Electrode) Setpoint Temp	° C		1150	1150	1150	1150	1150	1150	1150	1150
(Electrode Power) Output	%		64	63	63	61	61	60	62	62
(Electrode) Control Mode	A or M		A	A	A	A	A	A	A	A
Kiln Power	kW		0.1	0.1	0.1	0.1	0.1	0.1	0.0	0.1
Kiln Temp Setpoint	° C		850	850	850	850	850	850	850	850
Kiln Actual (Middle) Temp	° C		849	851	850	850	850	851	852	853
Kiln Power Output	%		21	21	21	17	20	20	4	21
Kiln Control Mode	A or M		A	A	A	A	A	A	A	A
Discharge Can Power	kW		0.3	0.4	0.4	0.3	0.4	0.3	0.4	0.3
Discharge Can Temp Setpoint	° C		800	800	800	800	800	800	800	800
Discharge Can Actual Temp	° C		768	772	778	780	782	782	780	786
Discharge Can Power Output	%		90	90	90	90	90	90	90	90
Overflow Heater Power	kW		1.7	1.8	1.8	1.8	1.7	1.8	1.8	1.8
Overflow Heater Setpoint	° C		1050	1050	1050	1050	1050	1050	1050	1050
Overflow Heater Power Output	%		63	66	67	66	65	67	67	67
Time	hh:mm		2112	2201	2306	0009	0105	0215	0314	0416

Reviewed and Approved: [illegible]

Date: 8/9/01

sheet 6 of 16

Data Sheet #1: Priority & Electrical Data

RSM-01-2										
	By:		[illegible]	[illegible]	[illegible]	WCB	WA	[illegible]	[illegible]	[illegible]
	Date:		8/9/01	8/9/01	8/9/01	8/9/01	8/9/01	8/9/01	8/9/01	8/9/01
	Time		0504	0623	958	1233	1331	1458	1605	2000
DESCRIPTION	UNITS	Range								
Priority Data										
Melt Temperature (T1, control)	° C	1125 - 1175	1149	1150	1150	1149	1149	1150	1150	1147
Melt Temperature (T2)	° C	1125 - 1175	1158	1149	1155	1153	1147	1155	1159	1150
Plenum Temperature	° C	400 - 600	585	639	665	575	575	591	618	675
Feed pump setting	%		12%	12	15	12	12	12	13	14
Cold Cap Coverage	%	> 75	90	40	50	90	95%	70	60	70
Slurry Pool Coverage of Cap	%		40	80	85	30	40%	80	40	40
Cold Cap Flexibility Visible	y or n		N	Y	Y	N	N	N	N	Y
Number of Vents	#		0	0	1	0	0	0	0	1
Cold Cap Thickness	inch		1"	1"	1.5"	1.5	1.25	1.5	1.3	1.25
Phase Separation (Note in LRB)	y or n		n	N	N	N	N	N	N	N
Glass Pouring	y or n		n	N	N	N	N	Y	N	N
Electrical Data										
Electrode Potential	Volts		21	23	23	21.9	21.7	22	22.4	24.4
Electrode Current	Amps		120	110	124	123	118	124	127.0	122.0
Electrode Power	kW		2.7	2.6	2.5	2.7	2.7	2.7	2.8	3.0
Melt Resistance	Ω		0.178	0.206	0.187	0.18	.18	0.179	0.175	.195
Melt (Electrode) Setpoint Temp	° C		1150	1150	1150	1150	1150	1150	1150	1150
(Electrode Power) Output	%		63	55	61	61	60	62	63	62
(Electrode) Control Mode	A or M		A	A	A	A	A	A	A	A
Kiln Power	kW		0.0	0.2	0	0.1	.1	0.1	0.1	0.0
Kiln Temp Setpoint	° C		850	850	850	850	850	850	850	850
Kiln Actual (Middle) Temp	° C		850	850	850	851	849	851	851	851
Kiln Power Output	%		10	27	0	18	19	19	22	0
Kiln Control Mode	A or M		A	A	A	A	A	A	A	A
Discharge Can Power	kW		0.3	0.3	.4	0.3	.3	.3	0.3	0.3
Discharge Can Temp Setpoint	° C		800	800	800	800	800	800	800	800
Discharge Can Actual Temp	° C		788	789	780	800	799	800	800	777
Discharge Can Power Output	%		90	90	90	88	97	85	85	90
Overflow Heater Power	kW		1.8	2.0	1.8	1.8	1.7	1.7	1.8	1.8
Overflow Heater Setpoint	° C		1050	1050	1050	1050	1050	1050	1050	1050
Overflow Heater Power Output	%		66	70	67	[illegible]	65	64	66	67
Time	hh:mm		0506	0626	1001	1239	1335	1501	1608	2004

Reviewed and Approved: [signature] Date: 2/12/02 sheet 7 of 10

Data Sheet #1: Priority & Electrical Data

RSM-01-2										
	By:									
	Date:		8/9/01	8/9/01	8/9/01	8/9/01	8/10/01	8/10/01	8/10/01	8/10/01
	Time		2102	2200	2302	2358	0113	0206	0304	0400
DESCRIPTION	UNITS	Range								
Priority Data										
Melt Temperature (T1, control)	° C	1125 - 1175	1150	1152	1147	1147	1154	1149	1152	1149
Melt Temperature (T2)	° C	1125 - 1175	1152	1140	1136	1143	1158	1152	1147	1147
Plenum Temperature	° C	400 - 600	612	580	583	589	596	609	618	610
Feed pump setting	%		14%	11	11	11	12	12	13	12.5
Cold Cap Coverage	%	> 75	95	95	85	75	90	70	70	100
Slurry Pool Coverage of Cap	%		20	20	30	30	30	25	60	40
Cold Cap Flexibility Visible	y or n		N	N	N	N	N	N	Y	Y
Number of Vents	#		0	0	0	0	0	0	2	1
Cold Cap Thickness	inch		2	3"	1"	3/4	1"	1/2 - 1	1/2	1+
Phase Separation (Note in LRB)	y or n		N	N	N	N	NO	NO	NO	NO
Glass Pouring	y or n		Y	Y	Y	N	NO	Y	NO	NO
Electrical Data										
Electrode Potential	Volts		24.2	21.9	23.5	23.1	23.8	25	22.7	24
Electrode Current	Amps		122.8	111.7	116.4	117.0	116	114	112	114
Electrode Power	kW		3.0	2.5	2.7	2.6	2.7	2.9	2.6	2.7
Melt Resistance	Ω		.198	.201	.198	.197	0.211	0.215	0.204	0.210
Melt (Electrode) Setpoint Temp	° C		1150	1150	1150	1150	1150	1150	1152	1150
(Electrode Power) Output	%		61	56	58	58	57	57	56	56
(Electrode) Control Mode	A or M		A	A	A	A	A	A	A	A
Kiln Power	kW		0.1	0.1	0.1	0.0	0.1	0.2	0.1	0.1
Kiln Temp Setpoint	° C		850	850	850	850	850	850	850	850
Kiln Actual (Middle) Temp	° C		849	850	851	851	850	851	851	851
Kiln Power Output	%		20	19	22	12	19	27	18	21
Kiln Control Mode	A or M		A	A	A	A	A	A	A	A
Discharge Can Power	kW		0.4	0.3	0.3	0.3	0.2	0.3	0.3	0.3
Discharge Can Temp Setpoint	° C		800	800	800	800	800	800	800	800
Discharge Can Actual Temp	° C		784	799	801	800	802	800	799	801
Discharge Can Power Output	%		90	90	89	89	74	86	90	88
Overflow Heater Power	kW		1.7	1.7	1.7	1.7	1.6	1.7	1.8	1.7
Overflow Heater Setpoint	° C		1050	1050	1050	1050	1050	1050	1050	1050
Overflow Heater Power Output	%		66	64	65	65	62	65	67	65
Time	hh:mm		2107	2201	2305	0000	0117	0208	0308	0401

Reviewed and Approved: _____ Date: 8/10/01 sheet 8 of 10

Data Sheet #1: Priority & Electrical Data

RSM-01-2										
	By:		[illegible]	[illegible]	WCD	TDP	[illegible]	[illegible]	[illegible]	JAM
	Date:		8/10/01	8/10/01	8/10/01	8/10/01	8/10/01	8/10/01	8/10/01	8/10/01
	Time		0506	0704	1103	1305	1655	1907	2000	2220
DESCRIPTION	UNITS	Range								
Priority Data										
Melt Temperature (T1, control)	° C	1125 - 1175	1150	1157	1159	1151	1151	1152	1148	1150
Melt Temperature (T2)	° C	1125 - 1175	1151	1148	1147	1147	1155	1142	1142	1145
Plenum Temperature	° C	400 - 600	587	663	740	594	742	601	625	668
Feed pump setting	%		11		12	12	10	8.5	8	18
Cold Cap Coverage	%	> 75	98	50	30	80	50	85	30	30
Slurry Pool Coverage of Cap	%		40	50	80	50	80	30	75	80
Cold Cap Flexibility Visible	y or n		N	Y	Y	Y	Y	N	Y	Y
Number of Vents	#		0	Several	0	2	3	2	1	
Cold Cap Thickness	inch		?	< 1/2	1/2	1/2 - 1	1/2	1"	1/2	
Phase Separation (Note in LRB)	y or n		?	N	N	N	N	N	N	
Glass Pouring	y or n		Y	N	N	N	N	~~N~~ dripping	N	N
Electrical Data										
Electrode Potential	Volts		23	22	22.5	25	27.5	24.7	22.4	
Electrode Current	Amps		113	101	123.6	100	124.4	93.4	94.4	
Electrode Power	kW		2.6	2.3	3.6	2.6	3.4	2.4	2.1	
Melt Resistance	Ω		0.206	0.227	0.240	0.255	.222	.267	.236	
Melt (Electrode) Setpoint Temp	° C		1150	1150	1150	1150	1150	1150	1150	
(Electrode Power) Output	%		56	49	61	50	62	48	48	
(Electrode) Control Mode	A or M		A	A	A	A	A	A	A	
Kiln Power	kW		0	0.1	0.1	0.2	0.0	0.2	0.1	0.0
Kiln Temp Setpoint	° C		850	850	850	850	850	850	850	850
Kiln Actual (Middle) Temp	° C		850	844	855	851	855	850	851	850
Kiln Power Output	%		11	22	18	30	0	26	22	10
Kiln Control Mode	A or M		A	A	A	A	A	A	A	A
Discharge Can Power	kW		0.3	0.4	0.3	0.3	0.3	0.3	0.3	0.4
Discharge Can Temp Setpoint	° C		800	800	800	800	800	800	800	800
Discharge Can Actual Temp	° C		801	797	718	787	747	795	797	787
Discharge Can Power Output	%		81	90	90	90	90	90	90	90
Overflow Heater Power	kW		1.7	1.9	2.0	1.9	2.1	1.7	2.0	1.8
Overflow Heater Setpoint	° C		1050	1050	1050	1050	1049	1052	1049	1050
Overflow Heater Power Output	%		64	69	70	68	72	65	70	66
Time	hh:mm		0510	0708	1109	1307	1700	1912	2001	2230

Reviewed and Approved: [signature] Date: 2/12/02

sheet 9 of 10

Data Sheet #1: Priority & Electrical Data

RSM-01-2										
	By:									
	Date:		8/10/01	8/10/01						
	Time		1757	2208						
DESCRIPTION	UNITS	Range								
Priority Data										
Melt Temperature (T1, control)	° C	1125 - 1175	1149	1152						
Melt Temperature (T2)	° C	1125 - 1175	1147	1153						
Plenum Temperature	° C	400 - 600	646	669						
Feed pump setting	%		11	16						
Cold Cap Coverage	%	> 75	85							
Slurry Pool Coverage of Cap	%		50							
Cold Cap Flexibility Visible	y or n		Y							
Number of Vents	#		3							
Cold Cap Thickness	inch		3/4							
Phase Separation (Note in LRB)	y or n		N							
Glass Pouring	y or n		N							
Electrical Data										
Electrode Potential	Volts		23.3	22.6						
Electrode Current	Amps		103.0	99.1						
Electrode Power	kW		2.3	2.3						
Melt Resistance	Ω		.226	.224						
Melt (Electrode) Setpoint Temp	° C		1150	1150						
(Electrode Power) Output	%		52	50						
(Electrode) Control Mode	A or M		A	A						
Kiln Power	kW		0	0.1						
Kiln Temp Setpoint	° C		850	850						
Kiln Actual (Middle) Temp	° C		846	850						
Kiln Power Output	%		7	24						
Kiln Control Mode	A or M		A	A						
Discharge Can Power	kW		0.3	0.3						
Discharge Can Temp Setpoint	° C		800	800						
Discharge Can Actual Temp	° C		757	798						
Discharge Can Power Output	%		~~0.057~~	90						
Overflow Heater Power	kW		1.9	2.0						
Overflow Heater Setpoint	° C		1050	1050						
Overflow Heater Power Output	%		69	70						
Time	hh:mm		1800	2210						

Reviewed and Approved: [signature] Date: 2/12/02

sheet 10 of 10

Data Sheet #2: Routine Status Sheet

RSM-01-2

DESCRIPTION	units	range								
		By:	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
		Date:	8/6/01	8/6/01	8/6/01	8/6/01	8/6/01	8/6/01	8/6/01	8/6/01
		Time:	16:30	17:40	18:50	19:53	21:01	22:10	23:04	0001
Feed Nozzle Cooling Flow	gpm	.5 - 1(reg FNT)	0.6	0.6	0.6	0.6	0.6	0.6	0.65	0.65
Film Cooler Air Supply	scfm	1-10 (reg OGT)	6.5	6.5	6.5	6.5	6.9	7.0	7.0	7.0
Melter Vacuum-Magnehelic	in. H_2O	0.1 - 2.0	-1.95	-2.0	-1.93	-1.8	-1.95	-1.97	-1.88	-1.91
EVS ΔP	in. H_2O		-.1517	-.20	-0.218	-0.207	-0.18	-0.21	-0.21	-0.21
Film Cooler ΔP	in. H_2O		-0.023	0.13	0.203	0.76	-0.086	0.07	-0.047	-0.035
HEME ΔP	in. H_2O		0.6328	0.58	0.60	0.538	0.652	0.605	0.609	0.613
System ΔP	in. H_2O		0.6710	0.95	0.996	1.59	0.79	0.879	0.766	0.703
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	3.2	3.2	3.1	3.1	3.1	3.1	3.3	3.7
EVS Scrub Tank Volume	Liters ~~gallon~~	35 - 50	60	61	62	62	62	62	62	63
EVS Nozzle Pressure	psi	50 - 55	50	50	50	50	50	50	50	50
~~EVS Scrub Solution pH~~	~~pH~~	~~> 9~~								
OG Control Valve position	%		50	50	50	42	50	50	56	50
OG Control Valve mode	A or M		A	A	A	A	A	A	A	A
Feed Pump (tubing) Condition			OK	OK	OK	OK	OK	OK	OK	OK
Agitator Setting	%		7	6	6	6	6	6	6	6
Blower Cooling Flow	gpm	1 - 1.5	2.2	2	1.8	1.9	1.9	1.9	2.0	2.3
Feed Nozzle Temp (FNT)	° C	< 40	29	29	30	30	29	30	29	29
Off-Gas Temp (OGT)	° C	< 250	199	164	168	184	190	143	138	141
Post EVS Off-Gas Temp	° C	< 50	39	37	33	37	36	35	35	34
Scrub Liquid Temp (SLT)	° C	<40	35	33	30	34	33	33	32	32
Heat Xer Temp	° C	< 30	37	36	32	35	35	34	34	34
Feed Pump Setting	%		20	15	15	15	15	15	15	14
Feed Pump Control Mode	A or M		A	Ext	Ext	Ext	Ext	Ext	Ext	Ext
Feed Pressure	psi	~~4.0~~ 2.0	1.0	0.8	1.3	1.1	1.1	1.1	1.3	1.0
Feed Tank Wt	Kg	decreasing	66.7	62.6	61.5	57.4	56.9	53.1	50.4	47.4
Overflow Temp	° C	1000 - 1100	1051	1050	1051	1050	1050	1050	1050	1051
Discharge Can Temp	° C	750 - 850	800	800	801	799	802	800	805	801
Glass Scale	Kg	< 10	0.8	0	1.55	1.56	2.29	2.26	2.55	3.53
Alarm Condition	On/Off	off	off	off	off	off	off	off	off	off
time	hh:mm				18:57	20:04	21:10	22:17	23:09	0018

deleted per RWC [illegible]

* small tank

** Shut restarted feed 18:55

* Maintenance activity on feed system may be skewing cup weight reading

Reviewed and Approved: [signature] Date: 8/7/01

sheet 1 of 10

Data Sheet #2: Routine Status Sheet

RSM-01-2

		By:	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
		Date:	8/7/01	8/7/01	8/7/01	8/7/01	8/7/01	8/7/01	8/7/01	8/7/01
		Time:	0126	0210	0306	0421	0511	0603	0709	0835
DESCRIPTION	**units**	**range**								
Feed Nozzle Cooling Flow	gpm	.5 - 1(reg FNT)	0.7	0.55	0.53	.52	0.53	0.48	0.48	0.5
Film Cooler Air Supply	scfm	1-10 (reg OGT)	7.1	7.1	7.1	7.1	7.2	7.2	7.2	7.2
Melter Vacuum-Magnehelic	in. H_2O	0.1 - 2.0	2.1	2.1	2.1	2.2	2.1	2.1	1.4	2.1
EVS ΔP	in. H_2O		0.2187	-0.21	-0.20	0.21	0.20	-.20	0.21	0.21
Film Cooler ΔP	in. H_2O		0.14	0.35	-0.01	0.22	0.26	0.29	1.31	0.05
HEME ΔP	in. H_2O		0.62	0.62	0.65	0.64	0.65	0.66	0.63	0.65
System ΔP	in. H_2O		0.98	1.15	0.85	1.06	1.15	1.18	2.1	0.79
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	3.7	4.3	4.3	4.2	4.1	3.8	3.6	3.6
EVS Scrub Tank Volume	gallon	35 - 50	65	65	66	66	66	66	70	70
EVS Nozzle Pressure	psi	50 - 55	50	50	50	50	50	50	50	50
EVS Scrub Solution pH	pH	> 9	—	—	—	—	—	—	—	—
OG Control Valve position	%		60	65	60	60	60	70	95	60
OG Control Valve mode	A or M		A	A	A	A	A	A	A	A
Feed Pump (tubing) Condition			OK	OK	OK	OK	OK	OK	OK	OK
Agitator Setting	~~1-10%~~		6	6	5.8	5.8	5.8	5.8	5.8	5.8
Blower Cooling Flow	gpm	1 - 1.5	2	1.9	1.9	1.8	1.8	1.6	1.6	1.6
Feed Nozzle Temp (FNT)	° C	< 40	29	29	29	29	29	29	29	29
Off-Gas Temp (OGT)	° C	< 250	138	142	146	147	137	137	118	145
Post EVS Off-Gas Temp	° C	< 50	34	34	34	34	34	34	34	33
Scrub Liquid Temp (SLT)	° C	<40	31	31	31	30	30	31	31	30
Heat Xer Temp	° C	< 30	33	34	34	33	32	34	33	33
Feed Pump Setting	%		14	15	16	16	16	16	16	16
Feed Pump Control Mode	A or M		A	A	A	A	A	A	A	A
Feed Pressure	psi	~~<1.0~~ 2.0	1.3	1.2	1.2	0.8	0.8	0.8	0.9	1.3
Feed Tank Wt	Kg	decreasing	43.5	42.0	39.0	32.9	31.0	30.4	27.5	45
Overflow Temp	° C	1000 - 1100	1050	1050	1050	1049	1050	1051	1050	1051
Discharge Can Temp	° C	750 - 850	801	801	799	800	800	800	801	719
Glass Scale	Kg	< 10	4.89	5.32	5.91	6.53	7.05	7.72	9.12	0.32
Alarm Condition	On/Off	off	off	off	off	off	off	off	off	T.2
time	hh:mm		0146	0218	0314	0428	0517	0613	0715	0844

Reviewed and Approved: [signature]

Date: 8/7/01

sheet 2 of 10

Data Sheet #2: Routine Status Sheet

RSM-01-2

		By:	TDP	TDP	TDP	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
		Date:	8-7-01	8-7-01	8-7-01	8-7-01	8/7/01	8/7/01	8/7/01	8/7/01
		Time:	1006	1200	1500	16:15	1709	1759	21:01	21:56
DESCRIPTION	**units**	**range**								
Feed Nozzle Cooling Flow	gpm	.5 - 1(reg FNT)	.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Film Cooler Air Supply	scfm	1-10 (reg OGT)	7.2	7.2	7.2	7.1	7.1	7.1	7.1	7.1
Melter Vacuum-Magnehelic	in. H_2O	0.1 - 2.0	2	2	2	2	2	2.1	2.1	2.2
EVS ΔP	in. H_2O		-0.2	-2.4	-0.2	-0.2	0.0	0.0	0.19	-0.20
Film Cooler ΔP	in. H_2O		0.1	-0.1	-0.05	-0.08	0.0	0.2	0.13	0.238
HEME ΔP	in. H_2O		0.7	0.64	0.65	0.64	0.6	0.7	0.63	0.637
System ΔP	in. H_2O		0.8	0.7	0.7	0.78	0.7	1.0	0.97	1.10
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	3.75	3.75	3.75	3.9	3.8	3.8	3.9	3.6
EVS Scrub Tank Volume	liters ~~gallon~~	35 - 50	70L	70L	68L	68	68	68	68	68
EVS Nozzle Pressure	psi	50 - 55	50	50	50	50	50	50	50	50
EVS Scrub Solution pH	pH	> 9	—	—	—	—	—	—	—	—
OG Control Valve position	%		40	50	50	58		52	55	66
OG Control Valve mode	A or M		A	A	A	A	A	A	A	A
Feed Pump (tubing) Condition			OK	OK	OK	OK	OK	OK	OK	OK
Agitator Setting	%		4.5	OFF	5.5	5	5.5	5.5	5.5	5
Blower Cooling Flow	gpm	1 - 1.5	1.75	1.75	1.75	1.7	1.7	1.7	1.7	1.6
Feed Nozzle Temp (FNT)	° C	< 40	30	31	30	30	30	29	29	30
Off-Gas Temp (OGT)	° C	< 250	159	120	162	131	128	126	114	115
Post EVS Off-Gas Temp	° C	< 50	35	31	34	35	36	36	34	33
Scrub Liquid Temp (SLT)	° C	<40	32	29	30	32	32	32	31	31
Heat Xer Temp	° C	< 30	29	29	33	33	33	34	32	32
Feed Pump Setting	%		14	0	15	15	14	14	13	9
Feed Pump Control Mode	A or M		A	A	A	A	A	A	A	A
Feed Pressure	psi	<1.0	1.3	1.3	1.4	1.3	1.2	1.0	0.9	1.0
Feed Tank Wt	Kg	decreasing	57.8	78.5	77.8	74.4	72.4	70.0	64.0	62.8
Overflow Temp	° C	1000 - 1100	1048	1049	1050	1051	1050	1150	1052	1050
Discharge Can Temp	° C	750 - 850	773	782	794	796	796	792	800	800
Glass Scale	Kg	< 10	—	1.69	1.96	2.55	3.18	3.41	4.58	6.47
Alarm Condition	On/Off	off	OFF	OFF	OFF	OFF	None/off	None/off	None	None
time	hh:mm		1015		1507	16:26	1716	1615	21:07	22:43

Reviewed and Approved: [signature] Date: 8-8-01 sheet 3 of 10

Data Sheet #2: Routine Status Sheet

RSM-01-2

		By:								
		Date:	8/7/01	8/8/01	8/8/01	8/8/01	8/8/01	8/8/1	8/8/1	8/8/01
		Time:	2304	0206	0300	0401	0505	0602	0713	0816
DESCRIPTION	**units**	**range**								
Feed Nozzle Cooling Flow	gpm	.5 - 1(reg FNT)	0.5	0.5	0.55	0.52	0.52	0.50	0.50	0.5
Film Cooler Air Supply	scfm	1-10 (reg OGT)	7.2	7.2	7.2	7.1	7.1	7.2	7.2	7.2
Melter Vacuum-Magnehelic	in. H_2O	0.1 - 2.0	2.0	2.1	2.1	2.1	1.9	2.1	2.1	2.2
EVS ΔP	in. H_2O		0	0.19	0.14	0.16	0.13	-0.16	-0.14	-0.15
Film Cooler ΔP	in. H_2O		0.4	0.07	0.05	0.06	0.36	-0.2	-0.08	0.34
HEME ΔP	in. H_2O		0.7	0.63	0.69	0.67	0.75	0.77	0.77	0.76
System ΔP	in. H_2O		1.3	0.76	0.88	0.97	1.37	1.00	1.00	1.3
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	4.0	3.9	4.1	4.0	3.8	3.7	3.6	3.7
EVS Scrub Tank Volume L	~~gallon~~	60 ~~25~~ - ~~50~~ 200	68	68	68	68	68	68	68	68
EVS Nozzle Pressure	psi	50 - 55	50	50	50	50	50	50	50	50
EVS Scrub Solution ~~pH~~ Flow	~~gpph~~	~~.8~~ 1.5-2	—	—	—	1.8	1.8	1.9	1.8	1.8
OG Control Valve position	%		40	50	60	60	90	55	50	60
OG Control Valve mode	A or M		A	A	A	A	A	A	A	A
Feed Pump (tubing) Condition			OK	OK	ok	ok	ok	ok	ok	OK
Agitator Setting	%		5	6.5	7.5	7.0	5.0	4.6	4.6	4.6
Blower Cooling Flow	gpm	1 - 1.5	1.7	1.7	1.8	1.7	1.7	1.6	1.6	1.6
Feed Nozzle Temp (FNT)	° C	< 40	30	31	31	30	29	29	29	29
Off-Gas Temp (OGT)	° C	< 250	161	149	182	157	147	153	149	146
Post EVS Off-Gas Temp	° C	< 50	34	30	34	34	34	34	34	34
Scrub Liquid Temp (SLT)	° C	<40	32	29	31	32	31	32	31	32
Heat Xer Temp	° C	< 30	32	29	34	34	34	34	34	34
Feed Pump Setting	%		11.0	0	18	18	15	16	16	16
Feed Pump Control Mode	A or M		A	A	A	A	A	A	A	A
Feed Pressure	psi	<1.0	1.0	2.8	1.4	0.9	1.1	1.2	1.2	1.2
Feed Tank Wt	Kg	decreasing	60.1	(working)	54.7	51.5	48.5	45.8	42.4	27.0
Overflow Temp	° C	1000 - 1100	1029	1050	1051	1051	1048	1049	1050	1050
Discharge Can Temp	° C	750 - 850	800	800	800	799	801	797	724 *	775
Glass Scale	Kg	< 10	6.7	6.70	7.47	7.68	8.85	9.16	0.00	1.56
Alarm Condition	On/Off	off	Move/off	on x	off	off	off	off	off	off
time	hh:mm		2315	0213	0307	0413	0515	0608	0718	0824

* PLENUM TEMP HIGH (813) due to no cold cap while feed is shut down.

x 0713 forgot to turn can temp all the way back to 800 after changeout

Reviewed and Approved: [signature] Date: 2/12/02

sheet 4 of 10

Data Sheet #2: Routine Status Sheet

RSM-01-2

		By:	TDO	WCB	TDO	WCB	TDO	TDO	TDO	TDO
		Date:	8-8-01	8/8/01	8/8/01	8/8/01	8/8/01	8/8/01	8/8/01	8/8/01
		Time:	1005	1100	1218	1354	1509	1608	1714	1917
DESCRIPTION	**units**	**range**								
Feed Nozzle Cooling Flow	gpm	.5 - 1(reg FNT)	.5	.5	0.5	.5	.5	.5	0.5	0.5
Film Cooler Air Supply	scfm	1-10 (reg OGT)	7.2	7.2	7.2	7.2	7.2	7.2	7.1	7
Melter Vacuum-Magnehelic	in. H_2O	0.1 - 2.0	2	2.2	2.0	1.89	2	2	2	2.2
EVS ΔP	in. H_2O		-0.14	-0.14	-0.14	-.13	-0.14	-0.16	-0.15	-0.17
Film Cooler ΔP	in. H_2O		0.1	0.9	-.008	+.1601	0.367	0.485	0.328	+0.07
HEME ΔP	in. H_2O		0.75	0.77	0.75	.76	0.77	0.72	0.78	0.76
System ΔP	in. H_2O		1	1.12	0.98	1.18	1.4	1.4	1.34	0.99
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	3.75	3.8	3.5	4	3.75	3.8	3.9	3.7
EVS Scrub Tank Volume	~~Liters~~	60 ~~25~~ - ~~50~~ 200	70	68	70	70	72	72	72	60
EVS Nozzle Pressure	psi	50 - 55	50	50	50	50	50	50	49	50
EVS Scrub Solution ~~pH~~ Flow	~~Gpm~~	1.5-2 ~~2-9~~	1.75	1.75	1.75	1.75	1.75	1.75	1.75	1.8
OG Control Valve position	%		43	41	50	50	42	50	50	50
OG Control Valve mode	A or M		A	A	A	A	A	A	A	A
Feed Pump (tubing) Condition			OK	OK	OK	OK	OK	OK	OK	OK
Agitator Setting	%		4		4	6	6	6	2.5	6
Blower Cooling Flow	gpm	1 - 1.5	1.65	1.7	1.75	1.75	1.75	1.75	1.7	1.6
Feed Nozzle Temp (FNT)	° C	< 40	29	29	29	30	30	30	29	30
Off-Gas Temp (OGT)	° C	< 250	144	147	159	159	157	159	150	267
Post EVS Off-Gas Temp	° C	< 50	35	36	35	35	36	36	36	34
Scrub Liquid Temp (SLT)	° C	<40	32	33	30	32	32	32	32	30
Heat Xer Temp	° C	< 30	34	34	34	34	36	35	32	32
Feed Pump Setting	%		15	15	15	15	15	15	0	0
Feed Pump Control Mode	A or M		A	A	A	A	A	A	OFF	OFF
Feed Pressure	psi	<1.0	1	1.2	1.0	1.1	1.2	1.1	1.3	1.5
Feed Tank Wt	Kg	decreasing	34.9	32.4	29.5	23.4	53.1	50.6	68*	64.0
Overflow Temp	° C	1000 - 1100	1051	1050	1050	1051	1050	1050	1049	1029
Discharge Can Temp	° C	750 - 850	786	793	797	8020	799	800	799	796
Glass Scale	Kg	< 10	2.83	3.87	4.38	5.61	6.47	7.17	8.04	8.95+
Alarm Condition	On/Off	off	off	off	off	off	off	off	off	off
time	hh:mm			11:10		1401	1514	1617	1722	1930

*New Feed Tank

+ Glass poured while probing salt due to no vacuum in plenum

Reviewed and Approved: R W Geles

Date: 2/12/02

sheet 5 of 10

Data Sheet #2: Routine Status Sheet

RSM-01-2

		By:	TDS	TDS	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
		Date:	8-8-01	8-8-01	8/8/01	8/8/01	8/9/01	8/9/01	8/9/01	8/9/01
		Time:	2002	2117	2202	2257	0001	0106	0207	0304
DESCRIPTION	**units**	**range**								
Feed Nozzle Cooling Flow	gpm	.5 - 1(reg FNT)	0.5	0.5	0.5	0.55	.55	0.72	0.71	0.71
Film Cooler Air Supply	scfm	1-10 (reg OGT)	7	7	7.0	7.0	7.0	7.1	7.1	7.1
Melter Vacuum-Magnehelic	in. H_2O	0.1 - 2.0	2	2	2	2	2	2.2	2.1	2.1
EVS ΔP	in. H_2O		-0.15	-0.17	0.0	0.0	0.0	.25	-.21	-2.3
Film Cooler ΔP	in. H_2O		-0.01	-0.04	0.0	0.0	0.0	0.11	.30	0.58
HEME ΔP	in. H_2O		0.76	0.74	0.6	0.6	0.6	0.6	.63	0.59
System ΔP	in. H_2O		1.0	0.9	0.6	0.7	0.8	0.89	1.14	1.31
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	3.75	3.75	3.6	4.4	4.4 3.5	3.5	3.4	3.4
EVS Scrub Tank Volume	~~L gallon~~	~~60 35 - 80~~ 200	62	62	63	62	62	60	60	60
EVS Nozzle Pressure	psi	50 - 55	50	50	50	50	50	51	50	50
EVS Scrub Solution ~~pH~~ Flow	~~L/min~~	1.5-2 ~~>8~~	1.75	1.75	1.75	1.8	1.8	1.9	1.8	1.8
OG Control Valve position	%		50	50	49	47		50	50	50
OG Control Valve mode	A or M		A	A	A	A	A	A	A	A
Feed Pump (tubing) Condition			OK	OK	OK	OK	OK	OK	OK	OK
Agitator Setting	%				6	6	6	4.5	4.5	4.5
Blower Cooling Flow	gpm	1 - 1.5	1.5	1.6	1.6	1.9	1.9	1.8	1.8	1.8
Feed Nozzle Temp (FNT)	° C	< 40	31	29	29	29	28	29	29	30
Off-Gas Temp (OGT)	° C	< 250	208	157	131	135	124	124	121	116
Post EVS Off-Gas Temp	° C	< 50	36	35	34	34	34	32	32	32
Scrub Liquid Temp (SLT)	° C	<40	32	32	32	32	32	31	29	32
Heat Xer Temp	° C	< 30	34	34	34	32	32	32	32	31
Feed Pump Setting	%		15	15	15	15	13	13	13	13
Feed Pump Control Mode	A or M		A	A	A	A	A	A	A	A
Feed Pressure	psi	<1.0	0.9	0.8	1.1	0.9	1.1	1.2	0.9	1.0
Feed Tank Wt	Kg	decreasing	67.8	65.1	62.6	60.1	56.8	54.0	51.9	50.0
Overflow Temp	° C	1000 - 1100	1045	1050	1048	1049	1052	1050	1052	1046
Discharge Can Temp	° C	750 - 850	726	770	773	777	780	781	782	780
Glass Scale	Kg	< 10	0.	0.18	0.73	1.63	2.12	2.67	3.35	3.51
Alarm Condition	On/Off	off	off	off	off	off	off	off	off	off
time	hh:mm			2117	2206	2302	0005	0103	0219	0310

Reviewed and Approved: [signature]

Date: 8/9/01

sheet 6 of 10

Data Sheet #2: Routine Status Sheet

RSM-01-2

		By:								
		Date:	8/9/01	8/9/01	8/9/01	8/9/01	8/9/01	8/9/01	8/9/01	8/9/01
		Time:	0420	0506	0616	1000	1226	1331	1502	1608
DESCRIPTION	**units**	**range**								
Feed Nozzle Cooling Flow	gpm	.5 - 1(reg FNT)	0.67	0.66	0.65	0.65	0.66	0.6	0.67	0.65
Film Cooler Air Supply	scfm	1-10 (reg OGT)	7.2	7.1	7.0	7.1	7.1	7.1	7.0	7.0
Melter Vacuum-Magnehelic	in. H_2O	0.1 - 2.0	2.2	2.2	2.4	2.1	2.3	2	2	2.2
EVS ΔP	in. H_2O		-.23	-.22	-.199	-.26	-.28	-0.24	-0.25	-0.30
Film Cooler ΔP	in. H_2O		1.0	1.5	-0.06	-.16	-.04	0.02	0.03	0.02
HEME ΔP	in. H_2O		.59	.62	0.656	.66	.68	0.7	0.64	0.62
System ΔP	in. H_2O		1.78	2.33	0.80	.64	.69	0.89	0.8	0.78
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	3.3	3.3	3.1	3.1	3.1	3.25	3.25	3.3
EVS Scrub Tank Volume	~~gallon~~ L	60 ~~35~~ ~~50~~ 200	60	60	60	59	59	59	59	59
EVS Nozzle Pressure	psi	50 - 55	50	51	51	49.5	50	50	50	50
EVS Scrub Solution ~~pH~~ Flow	~~GPM~~ L	1.5-2 ~~>0~~	1.8	1.8	1.8	1.6	1.8	1.25	1.25	1.75
OG Control Valve position	%		55	60	40	53	51	40	40	46
OG Control Valve mode	A or M		A	A	A	A	A	A	A	A
Feed Pump (tubing) Condition			ok	replace soon	OK	OK	OK	OK	OK	OK
Agitator Setting	%		5	5	5	5	5	5	5	3.6
Blower Cooling Flow	gpm	1 - 1.5	1.8	1.7	1.7	1.75	1.75	1.75	1.8	1.8
Feed Nozzle Temp (FNT)	° C	< 40	28	29	29	30	29	30	30	30
Off-Gas Temp (OGT)	° C	< 250	128	121	138	149	128	135	139	143
Post EVS Off-Gas Temp	° C	< 50	32	32	32	34	34	34	34	37
Scrub Liquid Temp (SLT)	° C	<40	30	30	31	32	32	32	32	33
Heat Xer Temp	° C	< 30	32	32	32	34	32	34	34	34
Feed Pump Setting	%		12	12	12	15	12	12	12	13
Feed Pump Control Mode	A or M		A	A	A	A	A	A	A	A
Feed Pressure	psi	<1.0	1.1	0.8	1.0	1.1	0.8	1.1	0.8	1.1
Feed Tank Wt	Kg	decreasing	45.8	43.8	41.3	35.6	42.0	40.1	37	35.5
Overflow Temp	° C	1000 - 1100	1050	1051	1044	1050	1051	1050	1050	1050
Discharge Can Temp	° C	750 - 850	785	789	790	782	800	799	799	800
Glass Scale	Kg	< 10	4.37	4.63	6.08	6.29	7.15	7.61	8.4	9.1
Alarm Condition	On/Off	off	off	off	off	off	off	off	off	off
time	hh:mm		0426	0512	0622	1009	1233	1335	1501	1614

Reviewed and Approved: RW Gale

Date: 2/12/02

sheet 7 of 10

Data Sheet #2: Routine Status Sheet

RSM-01-2

		By:	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
		Date:	8/9/01	8/9/01	8/9/01	8/9/01	8/9/01	8/9/01	8/10/01	8/10/01
		Time:	1915	2003	2100	2202	2257	2358	0115	0209
DESCRIPTION	**units**	**range**								
Feed Nozzle Cooling Flow	gpm	.5 - 1(reg FNT)	0.6	0.6	0.6	0.6	0.65	0.65	0.65	0.66
Film Cooler Air Supply	scfm	1-10 (reg OGT)	7.0	7.0	7.0	7.0	7.0	7.0	7.0	7.1
Melter Vacuum-Magnehelic	in. H_2O	0.1 - 2.0	2.2	2.2	2.2	2.1	2.1	2.2	2.2	2.2
EVS ΔP	in. H_2O		-0.17	-0.20	-0.19	0.0	0.0	-0.26	-0.246	-0.28
Film Cooler ΔP	in. H_2O		-0.14	-0.18	-0.14	0.0	0.0	-0.08	-0.148	-0.13
HEME ΔP	in. H_2O		0.66	0.62	0.62	0.6	0.6	0.65	0.66	0.61
System ΔP	in. H_2O		0.76	0.67	0.67	0.6	0.7	0.64	0.65	0.59
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	3.1	3.0	3.1	3.1	3.1	3.2	3.2	3.2
EVS Scrub Tank Volume	~~gallon~~ L	60 ~~98~~ - ~~90~~ 200	59	59	60	61	62	62	63	61
EVS Nozzle Pressure	psi	50 - 55	49	49	50	49	49	49	49	50
EVS Scrub Solution ~~pH~~ Flow	~~SNpH~~	1.5-2 ~~>8~~	1.7	1.7	1.7	1.8	1.8	1.8	1.8	1.8
OG Control Valve position	%		50	50	50	51	52	45	50	50
OG Control Valve mode	A or M		A	A	A	A	A	A	A	A
Feed Pump (tubing) Condition			ok	ok	ok	ok	ok	ok	ok	ok
Agitator Setting	%		6	6	6	6	6	6	6	6 (10L)
Blower Cooling Flow	gpm	1 - 1.5	1.8	1.75	1.75	1.7	1.8	1.8	1.8	1.8
Feed Nozzle Temp (FNT)	° C	< 40	30	30	29	29	29	29	29	29
Off-Gas Temp (OGT)	° C	< 250	195	159	138	131	131	134	136	135
Post EVS Off-Gas Temp	° C	< 50	37	38	37	35	36	36	36	36
Scrub Liquid Temp (SLT)	° C	<40	32	32	33	32	32	31	32	32
Heat Xer Temp	° C	< 30	34	34	34	32	32	32	32	32
Feed Pump Setting	%		13	14	13	11	11	11	12	12
Feed Pump Control Mode	A or M		A	A	A	A	A	A	A	A
Feed Pressure	psi	<1.0	0.9	1.1	1.1	0.7	1.0	1.0	0.7	1.4
Feed Tank Wt	Kg	decreasing	86.4	84.4	81.9	78.9	76.4	73.9	59.9	70.5
Overflow Temp	° C	1000 - 1100	1050	1050	1049	1051	1051	1049	1051	1050
Discharge Can Temp	° C	750 - 850	744	781	783	798	800	800	797	800
Glass Scale	Kg	< 10	0.06	0.07	0.41	1.16	1.69	2.33	3.63	4.36
Alarm Condition	On/Off	off	OFF	OFF	OFF	OFF	OFF	OFF	off	off
time	hh:mm		19:22	20:09	21:07	2207	2301	00:10	01:25	0218

Reviewed and Approved: [illegible]

Date: 8/10/01

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Data Sheet #2: Routine Status Sheet

RSM-01-2

		By:								
		Date:	8/10/01	8/10/01	8/10/01	8/10/01	8/10/01	8/10/01	8/10/01	8/10/01
		Time:	0858	0402	0500	0706	0910	1109	1255	1700
DESCRIPTION	**units**	**range**								
Feed Nozzle Cooling Flow	gpm	.5 - 1(reg FNT)	0.65	0.63	0.63	0.60	0.62	0.64	0.64	.65
Film Cooler Air Supply	scfm	1-10 (reg OGT)	7.0	7.1	7.1	7.1	7.1	7.0	7.0	7.0
Melter Vacuum-Magnehelic	in. H_2O	0.1 - 2.0	2.2	2.1	2.2	2.2	2.2	1.1	2.0	2
EVS ΔP	in. H_2O		-0.27	-0.25	-0.25	-0.21	-0.25	-0.21	-0.26	0.0
Film Cooler ΔP	in. H_2O		-0.15	-0.11	-0.11	-0.09	0.332	-.19	0.05	0.4
HEME ΔP	in. H_2O		0.67	0.64	0.65	0.69	0.65	0.67	0.57	0.7
System ΔP	in. H_2O		0.64	0.65	0.69	0.80	1.19	0.68	0.777	1.3
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	3.2	3.1	3.1	3.0	3.2	3.2	3.2	3.2
EVS Scrub Tank Volume	~~gallon~~ L	60 ~~35~~ - ~~50~~ 200	64	61	61	61	63	60	62	57
EVS Nozzle Pressure	psi	50 - 55	50	50	50	50	50	50	50	50
EVS Scrub Solution ~~pH~~ Flow	~~GPM~~	1.5-2 ~~>8~~	1.8	1.8	1.7	1.7	1.6	1.5	1.5	1.4
OG Control Valve position	%		45	50	55	50	48	53	50	45
OG Control Valve mode	A or M		A	A	A	A	A	A	A	A
Feed Pump (tubing) Condition			OK	OK	OK	OK	OK	OK	OK	OK
Agitator Setting	%		6	6 (64)	6	6 (8.24)	6	6	6	6
Blower Cooling Flow	gpm	1 - 1.5	1.8	1.8	1.7	1.7	1.7	1.75	1.75	1.7
Feed Nozzle Temp (FNT)	° C	< 40	29	29	29	29	29	31	30	32
Off-Gas Temp (OGT)	° C	< 250	133	126	120	137	107	175	116	192
Post EVS Off-Gas Temp	° C	< 50	34	34	34	36	33	37	37	37
Scrub Liquid Temp (SLT)	° C	<40	31	30	30	32	29	32	31	33
Heat Xer Temp	° C	< 30	32	32	32	32	29	34	32	34
Feed Pump Setting	%		13	12.5	11	12	[illegible]	12	12	11
Feed Pump Control Mode	A or M		A	A	A	A	A	A	A	A
Feed Pressure	psi	<1.0	1.5	1.4	1.2	2.0	1.2	2.3	2.6	2.3
Feed Tank Wt	Kg	decreasing	68.5	65.6	63.5	55.3	51.0	49.9	45.4	41.5
Overflow Temp	° C	1000 - 1100	1051	1051	1051	1051	1051	1051	1050	1050
Discharge Can Temp	° C	750 - 850	798	799	801	798	800	723	785	749
Glass Scale	Kg	< 10	4.45	4.85	5.26	6.41	9.03	0.11	0.3	2.03
Alarm Condition	On/Off	off	off	off	off	off	off	off	off	off
time	hh:mm		0303	0411	0504	0712	944	1115	1305	

Reviewed and Approved: R W Gelb Date: 2/12/02 sheet 9 of 10

Data Sheet #2: Routine Status Sheet

RSM-01-2

		By:	RW	[illegible]						
		Date:	8/10/01	8/10/01	8/10/01	8/10/01				
		Time:	1800	1900	2000	2310				
DESCRIPTION	**units**	**range**								
Feed Nozzle Cooling Flow	gpm	.5 - 1(reg FNT)	.65	.65	.65					
Film Cooler Air Supply	scfm	1-10 (reg OGT)	7	6.9	7.0					
Melter Vacuum-Magnehelic	in. H_2O	0.1 - 2.0	2.2	2	2.2	2.0				
EVS ΔP	in. H_2O		0.0	0.0	0.0	Ø				
Film Cooler ΔP	in. H_2O		0.0	0.1	0.3	0.2				
HEME ΔP	in. H_2O		0.6	0.5	0.6	0.6				
System ΔP	in. H_2O		0.6	0.7	1.2	0.7				
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	3.1	3.1	3.0					
EVS Scrub Tank Volume	~~L gallons~~	60 ~~35~~ - ~~50~~ 200	59	60	60					
EVS Nozzle Pressure	psi	50 - 55	50	50	50					
EVS Scrub Solution ~~pH~~ Flow	~~L/min~~	1.5-2 ~~≤8~~	1.5	1.5	1.5					
OG Control Valve position	%		53	49	47					
OG Control Valve mode	A or M		A	A	A					
Feed Pump (tubing) Condition			OK	OK	OK	—				
Agitator Setting	%		6	6	60	—				
Blower Cooling Flow	gpm	1 - 1.5	1.8	1.8	1.7					
Feed Nozzle Temp (FNT)	° C	< 40	30	30	30					
Off-Gas Temp (OGT)	° C	< 250	119	110	134					
Post EVS Off-Gas Temp	° C	< 50	37	36	36	39				
Scrub Liquid Temp (SLT)	° C	<40	32	31	32	32				
Heat Xer Temp	° C	< 30	33	32	32					
Feed Pump Setting	%		10.5	8.5	8.0	15				
Feed Pump Control Mode	A or M		A	A	A					
Feed Pressure	psi	<1.0	2.8	2.4	2.2	2.5				
Feed Tank Wt	Kg	decreasing	39.0	37.7	37.7	—				
Overflow Temp	° C	1000 - 1100	1050	1051	1051	1050				
Discharge Can Temp	° C	750 - 850	768	788	791	789				
Glass Scale	Kg	< 10	2.05	2.54	3.07					
Alarm Condition	On/Off	off	off	off	off	off				
time	hh:mm		1821	1907						

Reviewed and Approved: RW [illegible] Date: 2/12/02 sheet 10 of 10

Data Sheet #3: Off-Gas Analyzers

RSM-01-2	By:	MKS	RW/MM						
	Date:	6 Aug 01	7 Aug 01						
	Time:	1708	00:23						
DESCRIPTION	**units**								
Gas Chromatograph									
He	ppm	5,941	6,300						
H_2	ppm	325	297						
O_2	ppm	174,000	180,000						
N_2	ppm	653,000	694,000						
NO	ppm	4,285	6,070	MS					
CO	ppm		3,964						
CO_2	ppm	13,400	9,309						
N_2O	ppm	598	516						
NOx	ppm								
NOx Analyzer Computer									
Time	hh:mm	1708	0013:34						
NOx	ppm	1783	1,366						
NO	ppm	1,196	839						
NOx Voltage	Volts	3.56	2,321						
NOx Instrument									
Range selector	value	auto	a						
Mode selector	value	NOx							
Sample Pressure	psig	3.6							
Sample bypass flow	sccm	8							
Ozone Pressure	psig	32							
SO_2 Instrument									
Mode	1st Fld								
Range	ppm								
Reading	ppm	11.12							
MKS Mass Flow									
Ar Carrier, Chan-1	lpm	2.01							
He Tracer, Chan-2	lpm	4.01							
Total Sample , Chan-3	lpm	5.00							
Sample Pump*									
Vac Gauge	"Hg	-4-3							
Pres Gauge	psig	3.5							
Gas Cylinders Tank Pres#									
O_2	psig	1,000							
Ar GC	psig	~~2,000~~ 1,850							
Ar Sweep	psig	400							
He GC	psig	2,600							
He Tracer	psig	2,000							
Fid Analyzer (in Hood)									
Conc. (multi-digit display)	value	10							
Range (single digit display)	value	~~44~~							
Pump Outlet Pres	psig	~~7~~ 2.5							
Air Pres (reg next to pump)	psig	40							
Smple Pres, (Internal reg)	psig	7.2							
Fuel Pres, (internal reg)	psig	50							
time	hh:mm	1720							

*Located on computer table beside the GC/NOx analyzer rack connected to MKS mass flowmeter

Multiple tanks (Ar Sweep #s 1&2 and He Tracer #s 1,2) can be switched by manual valving if need be.

Reviewed and Approved: RW Hale

Date: 2/12/02

sheet 1 of 12

1

Off-Gas Sampling Data Sheet

No Cold Cap Feed off

Exposed Sample

Filter cooler changed from 2.5-2.0

RSM-02-1

DESCRIPTION	units								
	By:	MKS		MKS	MK	[illegible]		[illegible]	[illegible]
	Date:	6/4/01		6/4/01	6/6/01	3/6/01		3/6/01	3/6/01
	Time:	1737		1842	20:06	21:00		22:00	23:05
Gas Cyl. Line/Tank Pres									
O_2	psig	600		580	525	525		500	400
Ar GC	psig	1,850		1800	1800	1780		1750	1700
Ar Sweep#1	psig	2,000		2000	1920	1750		1750	1700
Ar Sweep#2	psig	200		150	150	150		150	150
He GC	psig	2,600		2600	2600	2600		2600	2600
He Tracer #1	psig	2,000		2000	2000	2000		2000	2000
He Tracer #2	psig	1,000		950	800	850		800	750
NOx Analyzer Computer									
Program Time	HH:MM	1742		18:42:49	20:01:09	21:01		22:11	23:08
NOx	ppm	1,575	HG 2,170	6.41	1378		1640	1578	1549
NO	ppm	974	1,525	4.10	1006	Feed off	857	1065	980
NOx Instrument				2.24V					
Sample Loop Pressure NOx	psig	3.6		3.6	3.5	3.4		3.5	3.5
Sample bypass flow	sccm	8		8	7	7		8	8
Ozone Pressure	psig	32		32	32	32		32	30
MKS Mass Flow Controller									
He Tracer, Chan-1 (SP=2.00)	lpm	2.00		2.01	2.01	2.01	~~2.01~~	2.01	2.01
Ar Sweep, Chan-2 (SP=4.00)	lpm	4.01		4.00	4.01	4.01	~~4.01~~	4.01	4.01
Total Sample Ch.3 (SP=5.00)	lpm	4.84	5.0	5.00	5.00	5.00	~~5.00~~	5.00	5.00
Main Sample Pump*									
Vac Gauge (<5 "Hg)	"Hg	-3	-3	-4	-7	-7	-7.0	-7.0	-9
Pres Gauge (~ 5 psig) 5-10	psig	3.0	5.0	7.0		5.0	4.5	4.5	7.0
Gas Chromatograph									
Pres Gauge (~8 to 9 psig)	psig	8.5		7.7		8.6		8.5	8.5
He	ppm	6460		7775	[illegible]	5433		6410	6573
H_2	ppm	271		0	253	93.2		254	266
O_2	ppm	181,000		210,349	184,000	184336		184077	187497
N_2	ppm	686,000		735,000	645,000	642736		694130	706925
NO	ppm	11,800		0	8500	9148		9667	10,807
CO	ppm	770		0	3850	4462		4374	4890
CO_2	ppm	13,400		4.62	10700	10212		10932	9200
N_2O	ppm	464		324	650	368		417	513
Total Hydrocarbon Analyzer									
Conc. (LED display)	ppm	13.0		1.6	4.10	4.9		9.0	8.5
PLC % Output for THA	%FS	3.5		0.5					
Air Pres (internal reg)	psig	38		39	39	39		39	39
Sample Pres (set @ 7 psi)	psig	7.2		7.2	7.2	7.2		7.1	7.1
Fuel Pres, (internal reg)	psig	49		50	50	50		50	50
SO_2 Instrument (in hood)									
Sampling Pressure (~ 2psi)	psig	2.5		2.5	2.5	2.5	~~2.0~~	2.5	2.5
Concentration Reading	ppm	11.3		3.7	10.7	10.22	~~10.1~~	10.1	8.3
Completion Time	hh:mm	1800	1800	18:53	20:14	21:08		22:15	23:13

*Located on table between the computer table and the GC/NOx analyzer rack connected to MKS mass flowmeter

Multiple tanks (Ar Sweep #s 1&2 and He Tracer #s 1&2) can be switched by manual valving if pressure in one tank is critically low

check drierite

FS=300 ppm

raised pump P, MFC 3 flow from 4.8 to 5.0

1818 - feed shut off to burn off cold top

Feed off 20:11

2) 2300 - Ron opened filter housing - to clean front half - sample flow & pres weak back up. White filter was dirty. $O_2 \rightarrow 21.0\%$ H_2 78.5%

Reviewed and Approved: RW [illegible]

Date: 2/12/02

sheet 2 of 12

RSM-02-1

3/

	By:	MR							
	Date:	7 Aug 01							
	Time:	0003							
DESCRIPTION	**units**								
Gas Cyl. Line/Tank Pres									
O_2	psig	400							
Ar GC	psig	1,700							
Ar Sweep#1	psig	1,600							
Ar Sweep#2	psig	150 closed							
He GC	psig	2,600							
He Tracer #1	psig	2,000 closed							
He Tracer #2	psig	700							
NOx Analyzer Computer									
Program Time	HH:MM	0023							
NOx	ppm	1,510							
NO	ppm	1,070							
NOx Instrument									
Sample Loop Pressure NOx	psig	3.685							
Sample bypass flow	sccm	8							
Ozone Pressure	psig	32							
MKS Mass Flow Controller									
He Tracer, Chan-1 (SP=2.00)	lpm	2.01							
Ar Sweep, Chan-2 (SP=4.00)	lpm	4.01							
Total Sample Ch.3 (SP=5.00)	lpm	5.00							
Main Sample Pump*									
Vac Gauge (<5 "Hg)	"Hg	7.1							
Pres Gauge (~ 5 psig) (Adj?)	psig	6							
Gas Chromatograph									
Pres Gauge (~8 to 9 psig)	psig								
He	ppm	6,300							
H_2	ppm	397							
O_2	ppm	180,000							
N_2	ppm	694,000							
NO	ppm	6,770							
CO	ppm	3,064							
CO_2	ppm	9,300							
N_2O	ppm	516							
Total Hydrocarbon Analyzer									
Conc. (LED display)	ppm	10.5							
PLC % Output for THA X3	%FS	3							
Air Pres (internal reg)	psig	40							
Sample Pres (set @ 7 psi)	psig	7.2							
Fuel Pres, (internal reg)	psig	50							
SO_2 Instrument (in hood)									
Sampling Pressure (~ 2psi)	psig	2.5							
Concentration Reading	ppm	9.4							
Completion Time	hh:mm	0024							

*Located on table between the computer table and the GC/NOx analyzer rack connected to MKS mass flowmeter

Multiple tanks (Ar Sweep #s 1&2 and He Tracer #s 1&2) can be switched by manual valving if pressure in one tank is critically low

Reviewed and Approved: [signature] Date: 2/12/02

sheet 3 of 12

RSM-02-1										
	By:	NRS								
	Date:	0120 Aug 7								
	Time:	0120								
GC & NOx analyzer										
He	ppm	6,544								
H_2	ppm	258								
O_2	ppm	188,000								
N_2	ppm	711,000								
NO	ppm	9,701								
CO	ppm	4,390								
CO_2	ppm	10,800								
N_2O	ppm	470								
Program Time	hhmm	0122								
NOx	ppm	1,047								
NO	ppm	850								
MKS Mass Flow Controller										
He Tracer, Chan-1 (SP=2.00)	lpm	2.01								
Ar Sweep, Chan-2 (SP=4.00)	lpm	4.01								
Total Sample Ch.3 (SP=5.00)	lpm	5.00								
Main Sample Pump										
Vac Gauge (<5 "Hg)	"Hg	-1								
Pres Gauge (~ 5 psig) (adj?)	psig	6								
Gas Cyl. Line/Tank Pres										
O_2	psig	350								
Ar GC	psig	1,650								
Ar Sweep 1 (switch?)	psig	1,500								
Ar Sweep#2 (switch?)	psig	150 closed —								
He GC	psig	2,600								
He Tracer 1 (switch?)	psig	2,000 closed —								
He Tracer 2 (switch?)	psig	650								
NOx Instrument										
Drierite color (blue)		blue/pink								
GC/NOx Press (~8 to 9 psig)	psig	8.5								
Sample Loop Pressure NOx	psig	3.6/4.4								
Sample bypass flow	sccm	8								
Ozone Pressure	psig	32								
SO_2 Instrument (in hood)										
Sampling Pressure (~ 2psi)	psig	2.5								
Concentration Reading	ppm	8.3								
Total Hydrocarbon Analyzer										
Conc. (LED display)	ppm	10.5								
Air Pres (internal reg)	psig	40								
Sample Pres (set @ 7 psi)	psig	7.2								
Fuel Pres, (internal reg)	psig	6.9								
PLC THA (x3=ppm)	%FS	3x3=9 ppm								
PLC feedrate	%FS	140 ppm								
PLC Completion Time	hhmm	0126								

0122 – Most of NOx is NO – is this ok? / 0128 – Need to change Drierite column to vertical to better use. Drierite

Reviewed and Approved: [signature] Date: 2/12/02 sheet 4 of 12

RSM-02-1	By:	NRS	NRS	ARB	BT	BT	MJ	UG	UG
	Date:	7Aug01	7Aug01	7Aug01	7Aug01	7Aug01	7Aug01	8/7/01	8/7/01
	Time:	0153	0345	0400	0457	0614	0719	0937	1029
GC & NOx: He	ppm	6,489	0	7106	0	317	293	289	6294
H_2	ppm	256	0	0	271	0	0	0	271
O_2	ppm	187,000	46,800	726,000	659000	584000	538000	672368	209002
N_2	ppm	705,000	175,000	843,000	766,000	678,000	624000	780610	789915
NO	ppm	8,413	0	0	8700	7491	6654	9170	9197
CO	ppm	3,807	0	0	522	384	102	8141	4162
CO_2	ppm	10,900	3.5	277	11800	7807	6551	10403	12153
N_2O	ppm	689, 112	0	0	406	276	387	395	442
Program Time	hhmm	0156		0400	0458	0616	0721	0934	1015
NOx	ppm	1,423	1.4	11.7	1535	979	1437	-.5471	1278
NO	ppm	894	0.5	6.9	1029	640	799	-.4926	888
MFC & Main Pump									
He Tracer, Chan-1 (SP=2.00)	lpm	2.00	2.01	2.01	2.01	2.01	2.01	2	2.01
Ar Sweep, Chan-2 (SP=4.00)	lpm	4.01	4.01	4.01	4.02	4.02	4.02		4.02
Total Sample Ch.3 (SP=5.00)	lpm	5.00	3.2	5.00	5.00	4.74	5.00		5.00
Pump Vac (<5 "Hg)	"Hg	2	-1	-2	-5	-4	9		10" 0 Clogged filter
Pump P (~ 5 psig) (adjust?)	psig	6	2	6	5	6	5		6
Gas Cyl. Line/Tank Pres									
O_2	psig	450		450	400	400	400		1900
Ar GC	psig	1,650		1600	1600	1550	1500		1500
Ar Sweep 1 (switch?)	psig	1,450		1300	1200	1150	1050		900
Ar Sweep#2 (switch?)	psig	150 (off)			off	off	off		950
He GC	psig	2,600		2,600	2600	2600	2600		2600
He Tracer 1 (switch?)	psig	2,000 (off)			OFF	off	off		2000
He Tracer 2 (switch?)	psig	600		550	500	500	400		450
NOx instrument									
Drierite color (blue)	---	Blue/pink		Blue	Blue/Pink	Blue/Pink	75% blue	60% blue	60% blue
GC/NOx Press (~8 to 9 psig)	psig	8.5		8.5	8.5	8.6	8.5	8.5	8
Sample Loop Pressure NOx	psig	3.6/4.4		3.6/4.4	3.6/4.4	3.6/4.4	3.6/4.4	3.5/4.4	3.6/4.5
Sample bypass flow	sccm	8	Sampling Hrly	9	8	8	8	9	9
Ozone Pressure	psig	32		32	32	32	30	0	30
SO_2 & THC (in hood)									
SO2 Sample P (~ 2psi)	psig	2.5		2.5	2.5	2.5	2.5	~~2.5~~ 2.5	2.5
SO2 Conc	ppm	8.6		0.3	7.2	8.4	7.5	7.5	7.1
THC Conc	ppm	9.8		4.9	10.5	9.8	11.1	7.5	5.6
THC Air P (internal reg, 40)	psig	40		40	40	40	40	40	40
THC Sample P (7 psi)	psig	7.2		7.2	7.2	7	7	7	7
THC Fuel P (internal reg, 50)	psig	50		50	50	50	49	50	50
PLC THA (x3=ppm)	%FS	3x3=9		2x3=6	4x3=12	4x3=12	4x3=12		6
PLC feedrate	%FS	14.0		0.0	16	16	16		14
PLC Completion Time	hhmm	0207		0403	0510	0622	0720		1054

0155 N_2O varied from 112–689 – RT? / 0351 – Back sampling 0351 – stopped feeding – feed plug?
0719 MFC ch3 on 4.63, pump P = 3.5, brought P to 5 The increased MFC ch3 to 5.00
1030 Leak was stopped for 1' long

Reviewed and Approved: [signature] Date: 2/12/02 sheet 5 of 12

RSM-02-1	By:	WRS	WRS	WRS	RLK	WRS		RLK	RLK
	Date:	7 Aug 01			→			→	→
	Time:	1335	1300	1432	16:30	1731		19:29	21:29
GC & NOx: He	ppm	0		6,672	6034	5,548		6868	6090
H_2	ppm	0		0,0	271	774		235	0
O_2	ppm	0	30,000	212,000	192,946	176,000		184,000	208,890
N_2	ppm	0	112,000	785,000	717,749	664,000		692,000	740,183
NO	ppm	0		0	5009 ~~2432~~	10,900		7242	8067
CO	ppm	0		0	~~2032~~ 2202	4,019		2365	3128
CO_2	ppm	0		401	1032	22,400 - 11,700		13952	4459
N_2O	ppm	0		0	120	726		323	322
Program Time	hhmm	—		1434	16:33	1734		19:30	21:31
NOx	ppm	—		2.2	731-09	1,100 -790		917	1256
NO	ppm	—		0.8	806.5	846 -600		828	903
MFC & Main Pump									
He Tracer, Chan-1 (SP=2.00)	lpm	2.01	2.01	2.00	2.0	2.00	2.00	2.00	2.00
Ar Sweep, Chan-2 (SP=4.00)	lpm	4.01	4.01	4.00	4.0	4.00	4.00	4.00	4.00
Total Sample Ch.3 (SP=5.00)	lpm	4. ~~5.00~~ 3.2	3.2	5.00	4 energy only	5.00	5.00	5.00	5.00
Pump Vac (<5 "Hg)	"Hg			-1	-2 Sealing Charge	-3	-4.5	-4.5	-6.0
Pump P (~ 5 psig) (adjust?)	psig			6	5	7	5	5	5
Gas Cyl. Line/Tank Pres									
O_2	psig			1900	1900	1900		1725	1800?
Ar GC	psig			1400	1300	1,300		1250	1200
Ar Sweep 1 (switch?)	psig			500 2,000 (off)	3 00	600		off Sweep	off
Ar Sweep#2 (switch?)	psig			2,100 500 (off)	2200	2200		2200	2200
He GC	psig			2,600	2600	2,600		2600	2600
He Tracer 1 (switch?)	psig			2,000 (off)	1450	1,900		1810	1750
He Tracer 2 (switch?)	psig			500	MT	0 closed		closed	closed
NOx Instrument									
Drierite color (blue)	---			1/2 pink	1/2 Pink	2/3 pink		2/3 Pink	3/4 Pink
GC/NOx Press (~8 to 9 psig)	psig			8.5	8-2	8.2		8-2	8-3
Sample Loop Pressure NOx	psig			14.5	4-6	4.0/4.6 no signal		4-6	4.6-4.6
Sample bypass flow	sccm			8	8	8		8	8
Ozone Pressure	psig			32	32	32 5		32	32
SO_2 & THC (in hood)									
SO2 Sample P (~ 2psi)	psig			2.6	~~2.6~~ 2.6	2.5		2-5	2-5
SO2 Conc	ppm			1.0	8.6	12.1		10.0	10-9
THC Conc	ppm			1.0	21.8	32.0 no match		4-9	4-2
THC Air P (internal reg, 40)	psig			40	40	40		40	40
THC Sample P (7 psi)	psig			7.2	7.2	7.2		7-0	7-0
THC Fuel P (internal reg, 50)	psig			49	49	49		50	50
PLC THA (x3=ppm)	%FS			0x3=0		2x3=6		(2x3) 60	(1.3x3) 3.9
PLC feedrate	%FS			0.0	15.0	14.0		13	10.0
PLC Completion Time	hhmm			1438	16:52	1740		19:38	21:38

1335 - Brett did leak checks using Ar direct to GC; no O_2 & H_2 detected; when sampling thru drierite, O_2 was 30,000 ppm, N_2 = 112,000 ppm. O_2 balance suggests 30,000/5.2 6000 ppm unacc.; 6,000/210,000 = 2.9% dil from air

1731 - The NOx Sample Loop P did not change when the unit switched; the local THC did not match the DAS output

Reviewed and Approved: RW Lee Date: 2/12/02 sheet 6 of 12

Main Comp

Bay 11 Bx

Tanks

Cabinet

Hood

RSM-02-1	By:	AK	BT	TDD	BT	BT	BT	TDD	BT
	Date:	8/7/01	8/8/01	8/8/01	8/8/01	8/8/01	8/8/01	8/8/1	8/8/01
	Time:	23:40	0216	0316	0412	0508	0600	0723	908
GC & NOx: He	ppm	5670	6309	5313	5206	4925	4505	5139	4919
H_2	ppm	0	0	178	277	175	209	139	152
O_2	~~ppm~~	20.7%	223 000 ppm	193000	191000	206000	203000	21.9%	212630
N_2	~~ppm~~	76.7%	82400 ppm	724000	718000	767000	755000	81.5%	790457
NO	ppm	7301	0	6883	9564	5892	5090	1830	6452
CO	ppm	2884	0	3360	2667	2436	2467	175	7614
CO_2	ppm	3586	345	10755	7534	6937	7408	6142	9178
N_2O	ppm	126	0	78	410	189	356	586	562
Program Time	hhmm	23:41	0218	0318	0415	0509	0601	0724	909
NOx	ppm	1352	4.59	1499	1895	1187	1119	1558	1290
NO	ppm	1035	2.33	1335	1954	926	1052	996	1262
MFC & Main Pump									
He Tracer, Chan-1 (SP=2.00)	lpm	2.0	2.0	2.00	2.00	2.00	2.00	2.00	2.00
Ar Sweep, Chan-2 (SP=4.00)	lpm	4.0	4.0	4.01	4.01	4.01	4.01	4.01	4.01
Total Sample Ch.3 (SP=5.00)	lpm	5.0	5.0	4.94	5.00	4.92	5.06	5.02	4.98
Pump Vac (<5 "Hg)	"Hg	6	6	6	8	8	9	10	10/10 changed filter
Pump P (~ 5 psig) (adjust?)	psig	5	4	3.3	3	3	3	3.5	2.5
Gas Cyl. Line/Tank Pres									
O_2	psig	1750	1700	1700	1700	1650	1650	1620	1600
Ar GC	psig	1150	1100	1050	1050	1000	1000	950	900
Ar Sweep 1 (switch?)	psig	off	off	0 / off	off	off	off	off	off
Ar Sweep#2 (switch?)	psig	1400	1800	1720	1600	1500	1450	1360	1400
He GC	psig	2600	2600	2600	2600	2600	2600	2600	2600
He Tracer 1 (switch?)	psig	1700	1600	1500	1500	1500	1400	1360	900
He Tracer 2 (switch?)	psig	off	off	0 / off	off	off	off	off	off
NOx Instrument									
Drierite color (blue)	---	80% Blue	Blue	95% blue	95% Blue	90% Blue	80% Blue	80% Blue	90% Blue
GC/~~NOx~~ Press (~8 to 9 psig)	psig	8.3	8.5	8.5	8.5	8.5	8.5	8.5	8.1
Sample Loop Pressure NOx	psig	4.6	4.6	4.55	4.6	4.6	4.6	4.5	4.5
Sample bypass flow	sccm	8.0	8	8	8	8	8	8.5	6
Ozone Pressure	psig	32	30	30	30	30	30	30	30
SO_2 & THC (in hood)									
SO2 Sample P (~ 2psi)	psig	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
SO2 Conc	ppm	8.1	0.64	6.27	7.56	5.8	6.8	6.9	5.08
THC Conc	ppm	3.4	3.9	7.8	9.1	7.8	9.8	6.5	12.5
THC Air P (internal reg, 40)	psig	40	40	40	40	40	40	40	40
THC Sample P (7 psi)	psig	7	7	7	7	7	7	7	7
THC Fuel P (internal reg, 50)	psig	50	50	50	50	50	50	50	50
PLC THA (x3=ppm)	%FS	1.1(3) = 3.3	1.1(3) = 3.3	2.65 → 7.95	3.6(3) = 10.8	3.1(3) = 9.3	2.7(3) = 8.1	2.35 → 7.05	3.15 9.45
PLC feedrate	%FS	12	0	18	18	15	16	16	16
PLC Completion Time	hhmm	23:47	0224	0325	0420	0511	0604	0728	925

00:30 - 00:40 charged Decarbonator

0316 - adjusted pump flow valve to bring MFC ch 3 back up to 5.0

Reviewed and Approved: RW [illegible] Date: 2/12/02

sheet 2 of 12

RSM-02-1	By:	nRS	nRS	nRS	nRS	nRS	nRS	nRS	nRS
	Date:	8 Aug 01	8 Aug 01	8 Aug 01	8 Aug 01	8 Aug 01	8 Aug 01	8 Aug	9 Aug
	Time:	1330	1430	1533	1640	1808	2228	2342	0039
GC & NOx: He	ppm	5,176	5,145	5,156	4,840	3,585	6,897	5,945	5,972
H_2	ppm	154	177	105	183	0.0	333	207	326
O_2	ppm	215,000	209,000	214,000	207,600	226,000	213,000	216,000	220,000
N_2	ppm	796,000	774,000	792,000	772,000	840,000	779,000	795,000	810,000
NO	ppm	6,630	7,124	5,539	9,884	0.0	7,830	5,867	5,327
CO	ppm	2,511	2,527	2,670	3,380	0.0	3052	2,428	2,256
CO_2	ppm	6,261	10,500	8,854	9,600	422	10,000	8,405	8,802
N_2O	ppm	228	638	260	591	0.0	739	795	330
Program Time	hhmm	1332	1432	1536	1642	1808	2231	2345	0041
NOx	ppm	1251	1,532	1,461	1,320	4.8	1195	1,200	1,017
NO	ppm	1087	1,541	1,211	838	2.2	1063	1,127	851
MFC & Main Pump									
He Tracer, Chan-1 (SP=2.00)	lpm	2.00	1.99	2.00	2.00	2.00	2.00	2.00	2.00
Ar Sweep, Chan-2 (SP=4.00)	lpm	4.00	3.99	3.99	3.99	3.99	4.00	4.00	4.00
Total Sample Ch.3 (SP=5.00)	lpm	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00
Pump Vac (<5 "Hg)	"Hg	-2	-2	-3	-3	-3	-4	-5	-1
Pump P (~ 5 psig) (adjust?)	psig	5	6	6	6	5	-6	5	6
Gas Cyl. Line/Tank Pres									
O_2	psig	1500	1,500	1,500	1,600	1,500	1,400	1,400	1350
Ar GC	psig	550	500	500	500	500	600	500	500
Ar Sweep 1 (switch?)	psig	0	2,100	2,150	2,200	2,200	2,200	2,200	2,100
Ar Sweep#2 (switch?)	psig	900	900	850	700	600	350	100	0
He GC	psig	2,600	2,600	2,600	2,600	2,600	2,600	2,600	2,600
He Tracer 1 (switch?)	psig	1,100 ~~400~~	1,100	1,100	1,000	950	800	700	700
He Tracer 2 (switch?)	psig	400	350	300	2,150	2,150	2150	2,150	2,150
NOx Instrument									
Drierite color (blue)	---	1/3 pink	1/3 pink	1/3 pink	1/3 pink	1/2 pink	1/2 pink	1/2 pink	1/2 pink
GC/NOx Press (~8 to 9 psig)	psig	8.5	8.5	8.5	8.5	8.5	8.5	8.5	8.5
Sample Loop Pressure NOx	psig	4.5	4.5	4.5	4.5	4.6	4.6	4.6	4.6
Sample bypass flow	sccm	8	8	8	8	8	8	8	8
Ozone Pressure	psig	32	32	32	32	32	32	32	32
SO_2 & THC (in hood)									
SO2 Sample P (~ 2psi)	psig	2.5	2.5	2.5	~~8.6~~ 2.5	2.5	2.5	3.0	~~4~~ 2.5
SO2 Conc	ppm	6.8	8.2	7.7	8.6	1.5	4.6	4.9	4.6
THC Conc	ppm	3.3	3.6	3.3	11.9	0.3	6.2	7.2	6.9
THC Air P (internal reg, 40)	psig	40	40	40	40	39	39	40	40
THC Sample P (7 psi)	psig	7.0	7.0	7.0	7.0	7.0	7.0	7.0	7.0
THC Fuel P (internal reg, 50)	psig	49	49	49	49	49	49	49	39
PLC THA (x3=ppm)	%FS	1.2x3=3.6	1.3x3=3.9	1.4x3=4.2	4.0x3=12	0.0x3=0.0	2.2x3=6.6	2.3x3=6.9	2.3x3=6.9
PLC feedrate	%FS	15.0	15.0	15.0	15.0	0.0	15.0	14.0	13.0
PLC Completion Time	hhmm	1338	1434	1538	1645	1812	2233	2347	0042

1806 – Feed off since 1712; no cold trap, port open for probing, baseline on O_2 & mid air leakage, Film layer @ 7.0 scfm, 11 psig, T ~ 90F

1149 – switched Ar 1 & 2

0158 – replaced CEMS filter, pump vac & P ok

Reviewed and Approved: R W Gale Date: 2/12/02

sheet 8 of 12

RSM-02-1	By:	WRS	WRS	WRS	WRS	WRS	WRS	WRS	
	Date:	9Aug01	9Aug01	9Aug	9Aug	8/9/01	8/9/01	9Aug	
	Time:	0137	0232	0333	0438	0514	0723	1628	1646
GC & NOx: He	ppm	5,990	6,000	6,062	6039	6112	5540	6,079	6,058
H_2	ppm	327	304	292	291	296	0.0	458	458
O_2	ppm	219,000	219,000	225,000	227,000	22.9%	23.3	180,000	180,000
N_2	ppm	806,000	806,000	825,000	834,000	84.3%	85.9	677,000	677,000
NO	ppm	6,020	4769	6,721	4746	5103	218	0	0
CO	ppm	2,476	747	2,700	2,200	2185	55	520	636
CO_2	ppm	9,043	9,900	10,200	8,230	8631	528	12,900	14,500
N_2O	ppm	452	408	330	485	530	0.0	698	638
Program Time	hhmm	0137	0233	0333	0440	5:16	7:30	1628	1647
NOx	ppm	6,029	914	1,287	739	1095	11.2	472 -1,000	478 -1,000
NO	ppm	841	745	1,069	865	940	3.6	507	365
MFC & Main Pump									
He Tracer, Chan-1 (SP=2.00)	lpm	2.00	2.00	2.00	2.00	2.00	2.00	2.00	
Ar Sweep, Chan-2 (SP=4.00)	lpm	4.00	4.00	4.00	4.00	4.00	4.00	4.00	
Total Sample Ch.3 (SP=5.00)	lpm	5.00	5.00	5.00	5.00	5.01	5.01	5.00	
Pump Vac (<5 "Hg)	"Hg	1	1	1	1	1	1	2	
Pump P (~ 5 psig) (adjust?)	psig	7	6	6	7	6	6	7	
Gas Cyl. Line/Tank Pres									
O_2	psig	1,300	1,300	1,300	1,300	1280	1220	1,100	
Ar GC	psig	500	500	450	400	400	350	350	
Ar Sweep 1 (switch?)	psig	2,000	1,900	1,850	1,800	1710	1560	900	
Ar Sweep#2 (switch?)	psig	0	0	0	0	0	0	2,100	
He GC	psig	2,550	2,550	2,550	2,550	2520	2510	2,600	
He Tracer 1 (switch?)	psig	650	600	600	500	500	420	300	
He Tracer 2 (switch?)	psig	2,150	2,100	2,100	2,100	2100	2100	2,000	
NOx Instrument									
Drierite color (blue)	---	1/2 pink	1/2 pink	2/3 pink	2/3 pink	2/3 pink	2/3 pink	3/4 pink	
GC/NOx Press (~8 to 9 psig)	psig	8.5	8.5	8.5	8.5	8.5	8.5	8.5	
Sample Loop Pressure NOx	psig	4.6	4.6	4.6	4.6	4.6	4.6	4.6	
Sample bypass flow	sccm	8	8	8	8	8.5	9	8	
Ozone Pressure	psig	32	32	32	32	32	32	32	
SO_2 & THC (in hood)									
SO2 Sample P (~ 2psi)	psig	3	3	3	3	3	3	3	
SO2 Conc	ppm	4.0	3.4	5.9	3.1	2.7	-2.2	4.7	
THC Conc	ppm	7.5	7.2	5.3	6.5	6.2	3.2	7.6	
THC Air P (internal reg, 40)	psig	40	40	40	40	40	40	39	
THC Sample P (7 psi)	psig	7.0	7.0	7.0	7.0	7	7	7.0	
THC Fuel P (internal reg, 50)	psig	49	39	39	39	50	50	48	
PLC THA (x3=ppm)	%FS	2.6x3=7.8	2.3x3=6.9	1.8x3=5.4	2.3x3=6.9	2.25x3=6.75	0.9 → 2.7	2.5x3=7.5	
PLC feedrate	%FS	13.0	13.0	12.0	12.0	12	0	13.0	
PLC Completion Time	hhmm	0141	0236	0339	0442	0521	0733	1649	

0333 just knocked down cell tops. NOx shot up, now back down some.
0723 feed still off (since 0623)
1647 - feed off

Reviewed and Approved: [signature] Date: 2/12/02 sheet 9 of 12

RSM-02-1	By:	MRS	MRS	MRS	MRS	MRS	MRS		MRS
	Date:	9 Aug 01	9 Aug 01	9 Aug 01	10 Aug	10 Aug	10 Aug		10 Aug 01
	Time:	1718	2236	2342	0031	0156	0209	0256	0304
GC & NOx: He	ppm	6,552	5,696	5,590	5,716	3,000	5,933	2375	6,037
H_2	ppm	0.0	68	150	162		105		0
O_2	ppm	196,000	194,000	190,000	195,000	160,000	197,000	137,000	204,000
N_2	ppm	728,000	725,000	714,000	731,000		738,000	511,000	760,000
NO	ppm	0.0	8,397	6,859	6,200		6,888		784
CO	ppm	0.0	3,000	2,775	2,500		2,437		200
CO_2	ppm	465	9,000	7,500	7,400		6,750		3,800
N_2O	ppm	0.1	215	628	156		275		121
Program Time	hhmm	1721	2239	2344	0033		0205		0306
NOx	ppm	5.3	32	4	-0.3		-0.4		-0.4
NO	ppm	1.9	34	4	-0.3		-0.3		-0.4
MFC & Main Pump									
He Tracer, Chan-1 (SP=2.00)	lpm	2.00	2.00	2.00	2.00		2.00	2.00	2.00
Ar Sweep, Chan-2 (SP=4.00)	lpm	4.00	4.00	4.00	4.00		4.00	4.00	4.00
Total Sample Ch.3 (SP=5.00)	lpm	5.00	5.00	5.00	5.00	4.4	5.00	4.4	5.00
Pump Vac (<5 "Hg)	"Hg	1	2	2	2		2	1	2
Pump P (~ 5 psig) (adjust?)	psig	6	6	6	6	3	6	3	4
Gas Cyl. Line/Tank Pres									
O_2	psig	1,100	1,000	1,000	1,000		900		900
Ar GC	psig	350	350	350	350		~~350~~ 350		350
Ar Sweep 1 (switch?)	psig	850	400	350	300		100		100
Ar Sweep#2 (switch?)	psig	2,100	2,100	2,100	2,100		2,100		2,000
He GC	psig	2,600	2,600	2,600	2,550		2,550		2,550
He Tracer 1 (switch?)	psig	200	200	200	200		200		200
He Tracer 2 (switch?)	psig	1,900	1,700	1,600	1,600		1,500		1,500
NOx Instrument									
Drierite color (blue)	---	3/4 pink	blue	blue	blue		1/4 ~~three~~ pink		1/4 pink
GC/NOx Press (~8 to 9 psig)	psig	8.5	8.5	8.5	8.5		8.5		8.5
Sample Loop Pressure NOx	psig	4.6	4.8	4.9	4.9		5.8 pegged		5+
Sample bypass flow	sccm	8	8	8	8		8		8
Ozone Pressure	psig	32	32	32	32		32		32
SO_2 & THC (in hood)									
SO2 Sample P (~ 2psi)	psig	~~0.8~~ 3	3	3	3		3		3
SO2 Conc	ppm	0.8	4.5	3.6	5.4		2.5		3.2
THC Conc	ppm	4.6	10.1 -20	12.1	11.4		11.1		11.4
THC Air P (internal reg, 40)	psig	39	40	40	40		40		40
THC Sample P (7 psi)	psig	7.0	7.0	7.0	7.0		6.8		6.8
THC Fuel P (internal reg, 50)	psig	48	39	48	48		48		48
PLC THA (x3=ppm)	%FS	1.4x3=4.2	3.4x3=10.2	3.7x3=11.1	4.1x3=12.3		4.2x3=12.6		3.7
PLC feedrate	%FS	0.0	11.0	11.0	11.0		12.0		13.0
PLC Completion Time	hhmm	1723	2244	2347	0035		0207		0310

1718- Feed has been off since 1647; this is an air condition
2145- CEMS was off from dryer change, about 1745 until now
2240- NOx was ~1,000 ppm from Beckman, now only 50; something appears wrong with the Beckman NO
0200- Pump pressure was too low; increased P, and the MFC 3 went from 4.5 to 3 and concentrations went to normal.
0256- Observed insufficient flow again; changed flow; filter ΔP changed

Reviewed and Approved: [signature] Date: 2/12/02

sheet 10 of 12

RSM-02-1	By:	TW	TW	TW	TW	MRS	MRS	MRS	MRS
	Date:	10 Aug 01	10 Aug 01	10 Aug 01	10 Aug 01	10 Aug 01	10 Aug 01	10 Aug 01	10 Aug 01
	Time:	0410	0603	0700	0800	1228	1356	1727	1907
GC & NOx: He	ppm	5764	6261	5447	5696	6,505	6,400	6,760	6,481
H_2	ppm	67	0	0	0	0.0	0.0	0.0	0.0
O_2	ppm	197686	212029	203730	201862	204,000	205,000	197,000	199,000
N_2	ppm	78358	78806	749349	743674	751,000	752,000	719,000	730,000
NO	ppm	7053	0	5545	1541	4,019	0.0	4,066	4,360
CO	ppm	2805	0	2325	644	2,400	2,824	0.0	0.0
CO_2	ppm	6255	548	679	809	751	1,267	not	—
N_2O	ppm	116	0	5.6	0	0.0	0.0	visible	—
Program Time	hhmm	0416	0609	0702	0812	1229	~~1357~~ 1357	1728	1903
NOx	ppm	-0.32366	-0.18579	-0.46165	-0.19272	1,340	~~1,253~~ 2,035	1,515	1,700
NO	ppm	-0.33079	-0.36334	-0.42003	-0.40229	881	1,252	922	1,000
MFC & Main Pump									
He Tracer, Chan-1 (SP=2.00)	lpm	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00
Ar Sweep, Chan-2 (SP=4.00)	lpm	4.00	4.00	4.00	4.00	4.00	4.00	4.00	4.00
Total Sample Ch.3 (SP=5.00)	lpm	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00
Pump Vac (<5 "Hg)	"Hg	2	2	2	2	2	2	3	3
Pump P (~ 5 psig) (adjust?)	psig	5	5	5	5	7	7	6	7
Gas Cyl. Line/Tank Pres									
O_2	psig		900		850	800	800	700	
Ar GC	psig		13000		12500	350	350	350	
Ar Sweep 1 (switch?)	psig		0		0	2,100	2,100	2,100	
Ar Sweep#2 (switch?)	psig		1850		1725	1,400	1,350	1,000	
He GC	psig		2500		2500	2,500	2,500	2,500	
He Tracer 1 (switch?)	psig		200		100	1,800	1,820	1,800	
He Tracer 2 (switch?)	psig		1375		1280	1,100	1,050	900	
NOx Instrument									
Drierite color (blue)	---	1/4 pink	1/4 pink	1/4 pink	1/4 pink	1/3 pink	1/3 pink	1/3 pink	
GC/NOx Press (~8 to 9 psig)	psig	8.5	8.5	8.5	8.5	8.5	8.5	8.5	
Sample Loop Pressure NOx	psig	5+	5+	5+	5+	~~4.22~~ 4.8	4.8	4.7	
Sample bypass flow	sccm	6.2	6.0	6.0	6.0	~~0~~ 3	3	3	
Ozone Pressure	psig	32	30	30	30	~~0~~ 32	32	32	
SO_2 & THC (in hood)									
SO2 Sample P (~ 2psi)	psig	3	3	3	3	3	3	3	3
SO2 Conc	ppm	.452	2.98	1.662	1.796	3.8	4.7	4.7	5.9
THC Conc	ppm	11.1	9.1	8.8	8.8	1.0	2.0	2.6	1.6
THC Air P (internal reg, 40)	psig	40	40	40	40	40	39	39	
THC Sample P (7 psi)	psig	6.8	6.8	6.8	6.9	6.8	6.8	7.0	
THC Fuel P (internal reg, 50)	psig	48	48	48	48	48	48	48	
PLC THA (x3=ppm)	%FS	3.8	3.15	2.85	2.75	0.0	0.0	0.00	
PLC feedrate	%FS	12.5	0.0	12.09	12.06	12.0	12.0	11.0	
PLC Completion Time	hhmm	0423	0614	0706	0817	1233	1407	1731	

1144 – increased pump P because sample flow too low
1846 – Cleaned pump filter inlet line – flowrate was too low now better.

Reviewed and Approved: [signature] Date: 2/12/02

sheet 11 of 12

RSM-02-1	By:	MDS	MRS						
	Date:	10 Aug	10 Aug						
	Time:	2016	2128						
GC & NOx: He	ppm	5422	5,580						
H_2	ppm	133	137						
O_2	ppm	198,000	184,000						
N_2	ppm	736,000	694,000						
NO	ppm	2,081	2,340						
CO	ppm	264	771						
CO_2	ppm	not visible 7,000	13,100						
N_2O	ppm	100	362						
Program Time	hhmm	~~1437~~ 2013	2125						
NOx	ppm	1137	1340						
NO	ppm	972	1039						
MFC & Main Pump									
He Tracer, Chan-1 (SP=2.00)	lpm	2.00	2.00						
Ar Sweep, Chan-2 (SP=4.00)	lpm	4.00	4.00						
Total Sample Ch.3 (SP=5.00)	lpm	5.00	5.00						
Pump Vac (<5 "Hg)	"Hg	-2	-5						
Pump P (~ 5 psig) (adjust?)	psig	7	5						
Gas Cyl. Line/Tank Pres									
O_2	psig	700	650						
Ar GC	psig	350	350						
Ar Sweep 1 (switch?)	psig	2,100	2,100						
Ar Sweep#2 (switch?)	psig	800	800						
He GC	psig	2,500	2,500						
He Tracer 1 (switch?)	psig	1,800	1,800						
He Tracer 2 (switch?)	psig	800	800						
NOx Instrument									
Drierite color (blue)	---	1/2 blue	1/2 blue						
GC/NOx Press (~8 to 9 psig)	psig	8.5	8.5						
Sample Loop Pressure NOx	psig	4.8	4.8						
Sample bypass flow	sccm	3	3						
Ozone Pressure	psig	32	32						
SO_2 & THC (in hood)									
SO2 Sample P (~ 2psi)	psig	3	3						
SO2 Conc	ppm	4.4	5.2						
THC Conc	ppm	3.3	6.0						
THC Air P (internal reg, 40)	psig	40	40						
THC Sample P (7 psi)	psig	7.0	7.0						
THC Fuel P (internal reg, 50)	psig	48	48						
PLC THA (x3=ppm)	%FS	0.4x3=1.2	2.5x3=7.5						
PLC feedrate	%FS	10.0	18						
PLC Completion Time	hhmm	2016	2129						

2130 – Pressure & flow just starting to drop for CEMS

Reviewed and Approved: RW Sale Date: 2/12/02 sheet 12 of 12

Appendix D

Colorimetric Procedure for Determining Fe^{+2} to Fe_{tot} Ratio

Appendix D: Colorimetric Procedure for Determining Fe^{+2} to Fe_{tot} Ratio

Fe^{+2} and Fe_{tot} Ratio

1.0 APPLICABILITY

This procedure is applicable for the determination of Fe^{+2} by completing the ferrous ion with 1, 10-phenanthroline. The concentration of the orange-red complex id determined spectrophotometrically at 510 nm.

2.0 DEFINITIONS

2.1 1,10-phenanthroline (phenanthroline) - The organic chelating agent that forms an orange-red complex with ferrous ion (Fe^{+2}).

2.2 Hydroquinone - An organic reducing agent that will reduce ferric ion (Fe^{+3}) to ferrous ion Fe^{+2} in an aqueous solution at room temperature.

2.3 Standard blank - The Fe_{tot} (Fe^{+2} and Fe^{+3}) concentration in all the reagents and the water used in this method.

2.4 Reagent blank - The Fe^{+2} concentration in all the reagents and the water used in this method.

3.0 RESPONSIBLE STAFF

3.1 Responsible Scientist.

3.2 Cognizant Staff.

4.0 PROCEDURE

4.1 Summary of the method

Samples are dissolved in a non-oxidizing condition using a mixture of sulfuric and hydrofluoric acid. Boric acid is added to complex the excess fluoride ion. The ferrous ion in the solution is-chelated by three molecules of phenanthroline forming an orange-red complex. The colored solution obeys Beer's law; its intensity is independent of pH from 3 to 9. A pH between 2.9 to 3.5 insures the rapid color development in the presence of an excess of phenanthroline. The total iron in the sample is determined by reducing the ferric ion to a ferrous ion with Hydroquinone at room temperature. The orange-red color of 'the complex is stable for up to six -months.

Technical Reviewer Date	Approval Authority Date (Line Manager)		Project Mgr Date
Author Date	Project Quality Engineer Date		Other Date
Procedure No:	REVISION NO.	EFFECTIVE DATE:	Page of

4.2 *Reagent*

4.2.1 Iron Standard Solution - A l00 ppm certified iron solution is used to prepare the calibration curve.

4.2.2 0.25% Phenanthroline Solution – Weigh 0.25 ± 0.05 gram of phenanthroline and dissolve in 100 mL of iron-free water.

4.2.3 4% Boric, Acid Solution - Weigh 40 ± 4 grams of orthoboric acid and transfer the orthoboric acid into a 1 liter plastic bottle. Fill the bottle with iron-free water.

4.2.4 Potassium Hydrogen Phthalate (KHP) - Weigh 100 + 10 grams of KHP and transfer the KHP into a 1 liter plastic bottle. Fill the solution with iron-free water and shake the solution to form a saturate the solution (Note: Since the solution is a saturated, some undissolved KHP is present in the bottom of the bottle).

4.3 *Equipment*

4.3.1 pH Meter - The pH meter used in this procedure is the-Corning Model 240-pH meter. Refer to APSL-08, CALIBRATION of PH METER, technical procedure to calibrate the pH meter

4.3.2 Spectrophotometer - The spectrophotometer used in this procedure is the Milton Roy, "Spectronic", model 601 spectrophotometer. Refer to APSL-09, USING THE "SPECTRONIC," MODEL 601 SPECTROPHOTOMETER, technical procedure for the set-up of the spectrophotometer.

4.4 *Safety*

4.4.1 Eye protection required in the laboratory.

4.4.2 Rubber or plastic cloves must be worn when working with concentrated acids.

4.5 *Calibration*

The spectrophotometer is calibrated with a certified iron solution at seven different calibration points. A linear regression is performed on the seven calibration points and the estimate of the slope (m), the y intercept (b), and the correlation coefficient (r) are calculated. If the calculated correlation coefficient is greater than 0.999, the calibration curve is closely approximated by a linear function. The closer the correlation coefficient approaches unity, the closer all the points used to establish the calibration curve falls on a straight line.

4.5.1 Dilute the 1000-ppm iron standard solution to a 100-ppm (0.1 mg/mL) solution by pipetting 10 mL of the 1000-ppm solution into a l00-mL volumetric flask. Add about 5 mL of concentrated HCL and dilute to volume with iron-free water. Thoroughly mix the solution in the flask.

4.5.2 Prepare a 100-mL disposable plastic beaker for the standard blank and each of the seven points used to calibrate the Fe^{+2} CALIBRATION CURVE (Figure 1). Mark the 8 plastic beakers as Std Blk, 0.005 mg Fe, 0.01 mg Fe, 0.05 mg Fe, 0.10 mg Fe, 0.20 mg Fe, 0.30 mg Fe, and 0.40 mg Fe.

4.5.3 Pipet the required volume of the 0.10 mg/mL Fe standard solution prepared in step 4.5.1 into the 7 beakers from step 4.5.2. Since no Fe is added to the Std Blk, this beaker will be empty.

4.5.4 Prepare a second set of disposable plastic beakers for the Std Blk and each of the 7 Fe calibration points. Each of the beakers with a capacity greater than 50 mL shall contain;

a) a glass covered magnetic stir-bar,

b) 25 mL of boric acid solution,

c) 7 mL of KHP solution,

d) 6 mL of phenanthroline solution.

4.5.5 Transfer the beakers from 4.5.4 to a fume hood and add 2 mL of concentrated ammonium hydroxide to each of the beakers. Swirl each of the beakers to mix the solution.

4.5.6 Slowly dispense 0.5 mL of concentrated sulfuric acid dropwi8e to each of the beakers containing the Std Blk and the 7 Fe standards prepared in step 4.5.2 using a 500 microliter pipet. After the sulfuric acid has been added to each of the beakers, add 1.5 mL of concentrated hydrofluoric acid to each of the beakers.

4.5.7 Pour one beaker of the buffer solution prepared in step 4.5.5 into each of the beakers from step 4.5.6.

4.5.8 After mixing the two solutions in step 4.5.7, place each of the beakers on a magnetic stirrer. Using a calibrated pH meter, adjust the pH of each of the solutions to a pH between 3.3 and 3.5 with either dilute sulfuric acid or dilute ammonium hydroxide.

4.5.9 Transfer the solution to a 100-mL volumetric flask. Add 20 ± 10 mg of Hydroquinone to each of the flasks. Using a small amount of iron-free water, wash the adhering Hydroquinone from the neck of the volumetric flask. Swirl the solution to dissolve the Hydroquinone and let the solution stand for at least 30 minutes.

4.5.10 After letting the solution stand for at least 30 minutes, dilute the solution to volume with iron-free water. Thoroughly mix the solution in the flask.

4.5.11 Set the absorbance of the spectrophotometer to 510 nm and zero the instrument with iron-free water in the sample cell.

4.5.12 Read the absorbance of the standard blank and each the seven iron standards. Record the absorbance reading from each of the solutions onto a Xerox copy of the Fe^{+2} CALIBRATION CURVE sheet (Figure 1).

4.5.13 Once all the absorbance readings have been taken, using a linear regression calculation, calculate the slope (m), the γ intercept (b), and the correlation coefficient(r) for the calibration data. If the calculated correlation coefficient is less than 0.999, notify the Responsible Scientist. If the correlation coefficient is greater than 0.999, enter the slope, the intercept, and all the absorbance readings onto the Fe^{+2} CALIBRATION CURVE using the Excel program called "FERCAL.XSL". The Excel program will calculate the values for the 7 calibration points using a linear curve with the estimated slope and intercept values determined above. The 0.005 mg Fe/100 mL of solution must have a calculated value between 0.003 and 0.007 mg Fe/100 mL. The mid-range values of the calibration curve must be within ± 5% of the true values. If the calculated values are not within these limits, notify the Responsible Scientist.

4.5.14 This completes the calibration of the spectrophotometer for the Fe^{+2} analyses. A copy of the signed and approved Fe^{+2} CALIBRATION CURVE shall be pasted into the notebook assigned to the Milton Roy, "Spectronic" model 601 spectrophotometer.

4.6 Sample Log-In Procedure

Refer to APSL-01.

4.7 Sample Preparation

The sample taken in the laboratory for the final grinding will depend upon the appearance and the size of the sample submitted by the customer. If a large sample (greater than 100 grams total) is submitted by the customer and the sample is not homogeneous, the sample will be ground to a particle size of less than approximately ¼ inch. This ground sample will then be reduced to a manageable sample size using the quartering method. If the sample is a homogeneous glass sample, between 5 and 10 grams of sample will be broken from the sample for final sample preparation.

4.7.1 Large non-homogeneous sample.

4.7.1.1 If the total sample weight is greater than 100 grams, grind the sample to less than ¼ inch particle size.

4.7.1.2 Place the sample in a pile on a clean sheet of paper. Using a flat spatula, quarter the sample into four equal parts.

4.7.1.3 Using alternate quarters of the sample in step 4.7.1.2, separate sample into two equal parts.

4.7.1.4 If one of the separated samples from step 4.7.1.3 is still greater than 40 grams, repeat steps 4.7.1.2 and 4.7.1.3 until the sample obtained by the quartering method weighs less than 40 grams.

4.7.1.5 When the sample weight has been reduced to less than 40 grams, grind the sample so that the particle size is less than 1/8 inch. Quarter the sample using steps 4.7.1.2 and 4.7.1.3.

4.7.1.6 Grind the sample from step 4.7.1.5 so that the particle size is less than 1/16 inch.

4.7.1.7 Quarter the sample two times using step 4.7.1.2 and 4.7.1.3. At this point, the sample should be reduced to less than 5 grams.

4.7.1.8 Grind the sample from step 4.7.1.7 using an agate or a porcelain mortar and pestle. Sieve the sample through a 140-mesh sieve. Repeat the grinding and sieving until all the sample has been ground and sieved to less than 140 mesh.

4.7.2 Homogeneous sample.

4.7.2.1 Cover the glass sample with a paper towel. Hit an edge of the sample to chip off pieces of glass. Collect the pieces of chipped glass from the larger sample.

4.7.2.2 Continue step 4.7.2.1 until approximately 3-5 grams of glass chips have been collected.

4.7.2.3 Grind the glass chips using an agate or a porcelain mortar and pestle. Sieve the sample through a 140-mesh sieve. Repeat the grinding and sieving until all the sample has been ground and sieved to less than 140 mesh.

4.8 *<u>Fe^{+2} Analyses</u>*

4.8.1 Prepare a disposable plastic beaker (beaker volume of at least 50 mL) for the standard blank and the sample blank and a beaker for each of the samples, the 0.005 mg Fe standard, and the 0.20 mg Fe standard. Add to each of the beakers:

a) a glass stir-bar,

b) 25 mL of boric acid solution,

c) 7 mL of KHP solution,

d) 6 mL of 4% phenanthroline solution and in a fume hood, pipet,

e) 2 mL of concentrated ammonium hydroxide.

4.8.2 To a second set 100-mL disposable plastic beakers, weight 0.025 ± 0.010 gram of ground sample from step 4.7.1.8 or 4.7.2.3 into a disposable plastic beaker. Record the sample weight and sample identification on each of the beakers. Also record the laboratory number, the customer identification, and the sample weights on a Xerox copy of the <u>Fe^{+2} AND Fe_{tot} DATA SHEET</u> (Figure 2).

4.8.3 Prepare two 100-mL disposable plastic beakers for the standard blank and the sample blank as well as the two beakers for the 0.005 mg Fe and the 0.20 mg Fe standards. Pipet 50 microliter of the 0.10 mg/mL Fe standard prepared in step 4.5.1 into the beaker marked 0.005 mg Fe Std and 2.0 mL of the 0.10 mg/mL Fe standard into the beaker marked 0.20 mg of Fe.

4.8.4 Pipet 0.5 mL of concentrated sulfuric acid to each of the beakers from steps 4.8.2 and 4.8.3 in a fume hood. Swirl the beakers with the samples so the samples are mixed with the sulfuric acid.

4.8.5 Tip the beaker so the sulfuric acid/sample mixture moves the slurry to one side of the beaker. Set the beaker on the floor of the fume hood and gently pipet 1.5 mL of concentrated hydrofluoric acid into the beaker on the opposite side of the sulfuric acid/sample mixture. Gently tip the beaker so the two acids are mixed. Once the initial vigorous reaction between the hydrofluoric acid and the silicate in the glass has subsided, swirl the beakers so that the hydrofluoric acid is mixed completely with the glass sample in the beaker. (Note: The total time for hydrofluoric acid to dissolve the glass must be less than about 30 seconds to reduce the amount of air oxidation of the Fe^{+2} in the solution. Once the solution from step 4.8.1 containing the phenanthroline has been added to the solution in 4.8.5, air oxidation of Fe^{+2} is minimized. Each sample is carried through the pH adjustment steps 4.8.6, 4.8.7, and 4.8.8 before adding hydrofluoric acid to the next beaker.)

4.8.6 After the dissolution of the glass by the hydrofluoric acid, immediately transfer the contents of one of the beakers prepared in step 4.8.1 into the beaker containing the dissolved glass in step 4.8.5.

4.8.7 Mix the two solutions together from step 4.8.6 and using a calibrated pH meter, adjust the solution pH to between 3.3 and 3.5 using either dilute sulfuric acid or ammonium hydroxide. (Note: There should be no precipitate formed during the pH adjustment. If a precipitate forms, notify the Responsible Scientist.)

4.8.8 Transfer the pH-adjusted solution into a 100-mL volumetric flask. Use a small volume of iron-free water to rinse all the solution from the beaker into the volumetric flask.

4.8.9 Repeat steps 4.8.5, 4.8.6, 4.8.7, and 4.8.8 with each beaker one at a time until all the blanks, the two standards, and all the samples have been carried through the pH adjustment and solution transfer outlined in steps 4.8.7 and 4.8.8.

4.8.10 Set aside the flasks containing the standard blank, the 0.005 mg Fe and the 0.20 mg Fe standards. These solutions will be completed in the Fe_{tot} analyses portion of this procedure.

4.8.11 Fill the remaining flasks to volume with iron-free water. Cap and thoroughly mix the solution in each of the flasks.

4.8.12 Adjust the spectrophotometer to read zero absorbance with iron-free water in the sample cell.

4.8.13 Transfer the sample blank to the sample cell and record the Fe^{+2} absorbance reading onto the Xerox copy of the Fe^{+2} AND Fe_{tot} DATA SHEET in the column marked Fe^{+2} Abs. Place the next sample from step 4.8.11 into the sample cell. Read the absorbance of the sample and record absorbance reading onto the Xerox copy of the Fe^{+2} AND Fe_{tot} DATA SHEET. Repeat reading and recording of the absorbance until all the samples in step 4.8.11 are completed.

4.9 *Fe_{tot} Analyses*

Soluble iron in an acidic aqueous solution is present either as ferrous or ferric ion. Hydroquinone will reduce all ferric ion (Fe^{+3}) in the solution to a ferrous ion (Fe^{+2}). With the

reduction of all the Fe^{+3} to Fe^{+2}, the analyses of Fe^{+2} in the solution with the phenanthroline are the Fe_{tot} analyses.

4.9.1 Pipet 10 mL (or a suitable aliquot) of each the sample blank and the samples from step 4.8.13 into separate 100-mL volumetric flasks containing 4 mL of phenanthroline solution in each of the flasks.

4.9.2 Add 20 ± 10 mg of Hydroquinone to each of the flasks from step 4.9.1 and the three flasks containing the standard blank, the 0.005 mg Fe and the 0.20 mg Fe standards from step 4.8.10.

4.9.3 Wash down any Hydroquinone adhering to the neck of the flasks with a small volume of iron-free water. Swirl the solution in the flasks to dissolve the Hydroquinone. Let the flasks stand for at least 30 minutes.

4.9.4 After 30 minutes, dilute the solution in the flasks to volume with iron-free water. Cap the flasks and thoroughly mix the solution in each flask.

4.9.5 Adjust the spectrophotometer to read zero absorbance with iron-free water in the sample cell.

4.9.6 Transfer each of the solutions from Step 4.9.4 into the sample cell and read the Fe_{tot} absorbance of the solution. Record the Fe_{tot} absorbance on the Xerox copy of the Fe^{+2} AND Fe_{tot} DATA SHEET in the column labeled Fe_{tot} Abs. Also record the volume of solution pipetted in Step 4.9.1 (divided by 100) onto the Fe^{+2} AND Fe_{tot} DATA SHEET.

4.10 Calculation

The Fe^{+2} and Fe_{tot} are calculated by entering the values from the Fe^{+2} AND Fe_{tot} DATA SHEET onto the first page of the Excel program labeled "FERATIO4.XLS" and titled Fe^{+2} AND Fe_{tot} DATA ENTRY SHEET (Figure 3). The Excel program will perform the required calculations using a linear equation, which is:

$$y = mx + b$$

where:

m = slope

x = mg Fe/100 mL of solution

b = y intercept of the calibration curve

y = sample absorbance – blank absorbance

The third page of the Excel program will generate the ANALYSIS OF Fe^{+2} AND Fe_{tot} report that is shown in Figure 4 and will be submitted to the customer.

For verification of the excel program labeled "FERATIO4.XLS", a set of sample data will be "hand" calculated and compared with the data generated by the "FERATIO4.XLS." This will be pasted in the notebook for the Milton Roy, "Spectronic", model 601 spectrophotometer.

4.11 Calibration Acceptance Criteria

4.11.1 If there is more than 0.005 differences in the absorbance of the sample blank and the standard blank, notify the Responsible Scientist. A significant difference between the two blanks typically indicates a reagent contamination of ferric ion. This must be corrected before continuing with the analysis.

4.11.2 Check the calculated values for the 0.20 mg Fe standard. This value in the mid-range of the calibration curve should read within 0.20 ± 0.01 mg of Fe_{tot}/100 mL of solution. If the calculated value is not within the acceptable range, notify the Responsible Scientist.

4.11.3 Check the calculated value for the 0.005 mg Fe standard. This standard is at the lower end of the calibration curve. If the calculated value is not within 0.005 ± 0.002 mg of Fe/100 mL of solution, notify the Responsible Scientist.

4.12 Archiving of Data

4.12.1 A copy of all the Fe^{+2} CALIBRATION CURVE analyses performed on the Milton Roy, "Spectronic", model 601 spectrophotometer are posted in the notebook for this instrument. The calibration curve used for the Fe^{+2} analyses is identified by the slope (m), the intercept (y), and correlation coefficient (r) recorded on the Fe^{+2} AND Fe_{tot} DATA SHEET, the Fe^{+2} ANAD Fe_{tot} DATA ENTRY SHEET, and the ANALYSIS OF Fe AND Fe_{tot} forms.

4.12.2 The completed Fe^{+2} AND Fe_{tot} DATA SHEET is stored in the sample data file stored under Laboratory Number.

4.12.3 A copy of the completed and approved ANALYSIS OF Fe^{+2} AND Fe_{tot} form sent to the customer shall be stored in the sample data file stored under the Laboratory Number.

Figure 1

Fe^{+2} CALIBRATION CURVE

Analytical and Process Support Laboratory

1.0 Date of Calibration _____

2.0 Spectrophotometer Used _____

3.0 Cell Used _____

4.0 Iron Standard Used _____

5.0 Absorbance Readings:

		Absorbance		Absorbance	Calculated Fe Values
5.1	Standard Blank	_____			
5.2	0.005 mg Fe Std	_____	Minus Blk	_____	_____
5.3	0.01 mg Fe Std	_____	Minus Blk	_____	_____
5.4	0.05 mg Fe Std	_____	Minus Blk	_____	_____
5.5	0.10 mg Fe Std	_____	Minus Blk	_____	_____
5.6	0.20 mg Fe Std	_____	Minus Blk	_____	_____
5.7	0.30 mg Fe Std	_____	Minus Blk	_____	_____
5.8	0.40 mg Fe Std	_____	Minus Blk	_____	_____

6.0 Linear Regression Analysis Calculation:

6.1 Correlation Coefficient (r) _____

6.2 Slope (m) _____

6.3 Intercept (y) _____

7.0 Calibrated by and Date: _____

8.0 Reviewed by and Date: _____

Figure 2

Fe^{+2} AND Fe_{tot} DATA SHEET

Analytical and Process Support Laboratory

Customer _____

Calibration curve used: Slope (m) = _____

Intercept (y) = _____

Correlation coefficient = _____

Low and high standard used to verify calibration curve.

Standard blank Absorbance = _____

0.005 mg Fe Std Absorbance = _____

0.200 mg Fe Std Absorbance = _____

Sample blank (Fe^{+2}) Absorbance = _____

Sample blank (Fe_{tot}) Absorbance = _____

Row	Laboratory Number	Customer's Sample ID	Sample Wt	Fe^{+2} Dil	Fe^{+2} Abs	Fe_{tot} Dil	Fe_{tot} Abs
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							
16							

Balance Used and Date _____

Analyst Signature and Date _____

Figure 3

Fe^{+2} AND Fe_{tot} DATA ENTRY SHEET

Analytical and Process Support Laboratory

Customer _____

Calibration curve used Slope (m) = _____

Intercept (y) = _____

Correlation Coefficient = _____

Low and high standard used to verify calibration curve.

Standard blank Absorbance = _____

0.005 mg Fe Std Absorbance = _____

0.200 mg Fe Std Absorbance = _____

Sample blank (Fell) Absorbance = _____

Sample blank (Fe_{tot}) Absorbance = _____

Row	Laboratory Number	Customer's Sample ID	Sample Wt	Fe^{+2} Dil	Fe^{+2} Abs	Fe_{tot} Dil	Fe_{tot} Abs
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							
16							

Data entered by and date _____

Figure 4

ANALYSIS OF Fe^{+2} AND Fe_{tot}

Analytical and Process Support Laboratory

To:

The Iron II (Fe^{+2}) and Total Iron (Fe_{tot}) have been analyzed in your samples. Two iron standards are analyzed with each set of samples to verify the slope and intercept of the linear calibration curve. The first standard containing 0.005 mg Fe/100 mL is near the detection limit of the colormetric method, while the second standard containing 0.20 mg Fe/100 mL is used to verify the mid-range of the calibration curve. The results of the two iron standards and your samples are tabulated below:

Calibration Curve Used. Slope (m) = _____

Intercept (y) = _____

Correlation Coefficient = _____

Low and high iron standard analysis to verify calibration curve.

0.005 Fe Std = _____ Lower Limit = 0.0025 Upper Limit = 0.0075 mg

0.200 Fe Std = _____ Lower Limit = 0.190 Upper Limit = 0.210 mg

Sample Analyses

Lab No	Customer's Sample ID	Wt % Fe^{+2}	Wt % Fe_{tot}	Fe^{+2}/Fe_{tot}	Precipitate Yes	Precipitate No	If yes, comment

Comments:
1. Dark non-magnetic precipitate.
2. Dark magnetic precipitate.
3. Light colored (whitish) precipitate.
4. Other _____

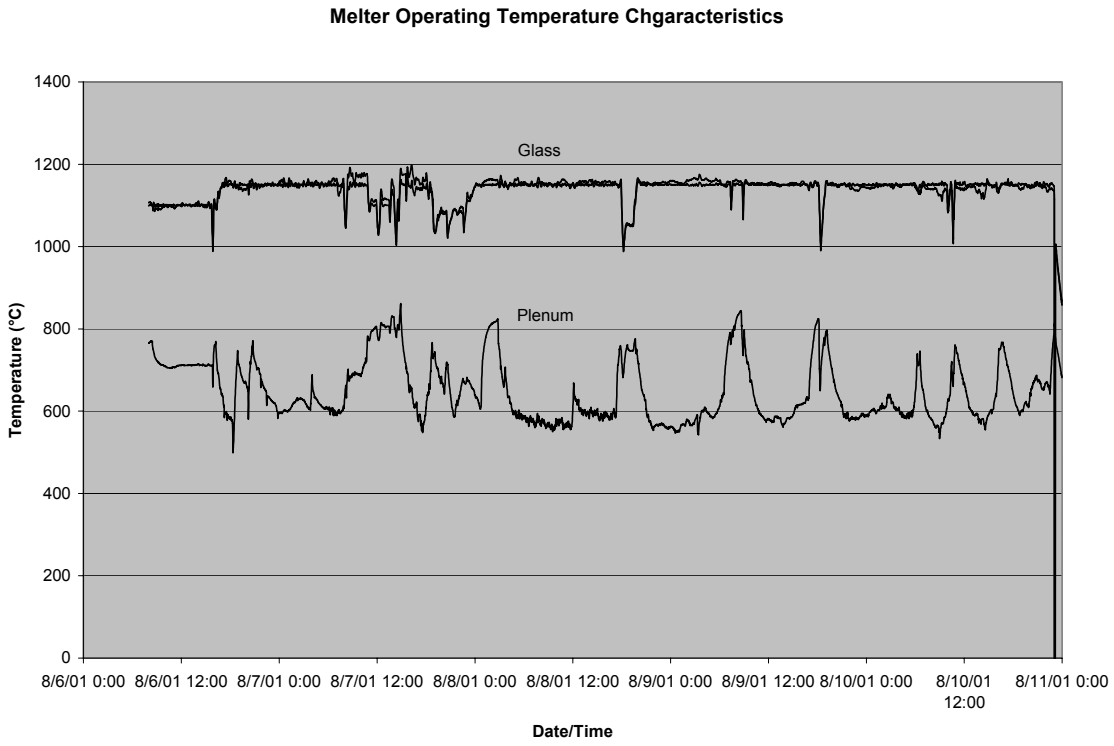
Analyst signature and date _____

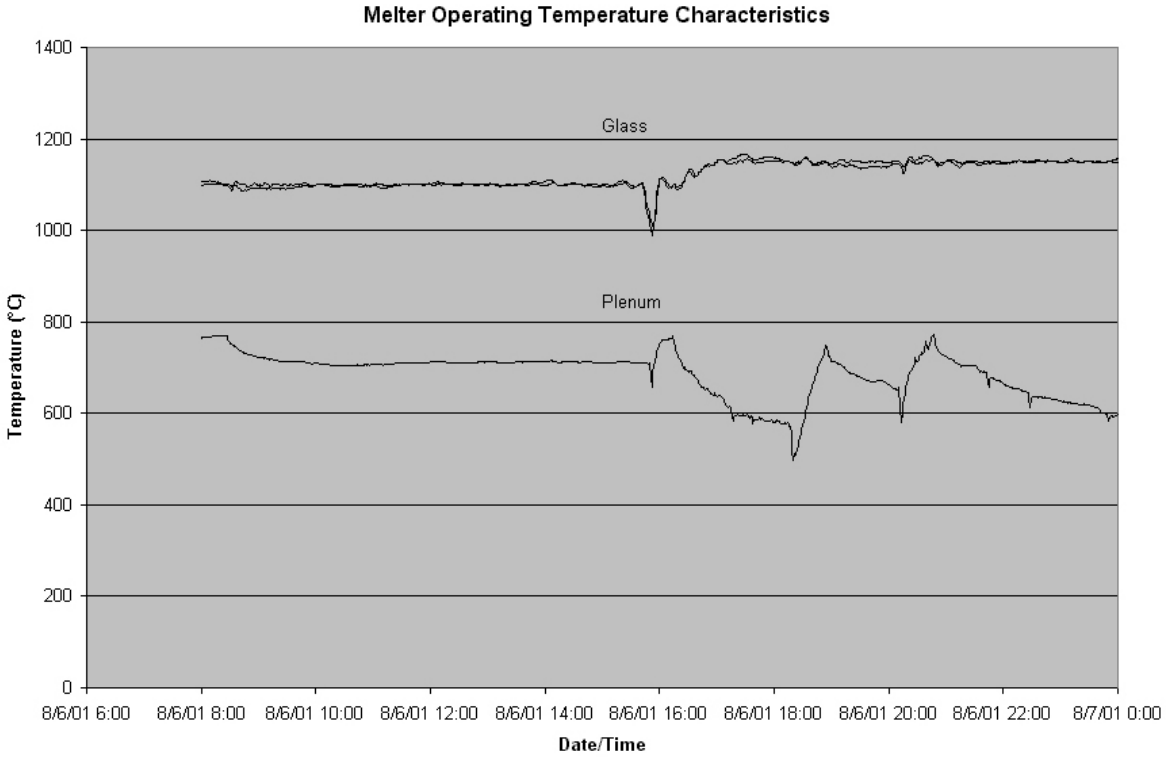
Approved by and date _____

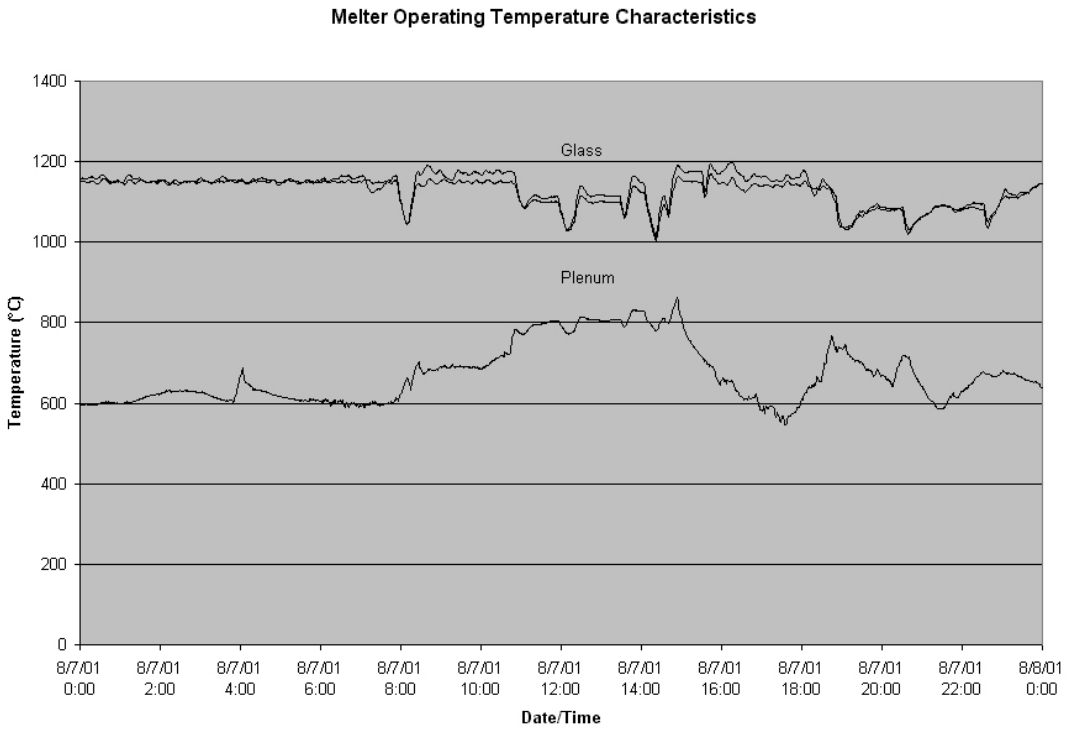
Appendix E

Variable Process-Temperature Data Collected During the SBW Flowsheet Evaluations

Appendix E: Variable Process-Temperature Data Collected During the SBW Flowsheet Evaluations






Melter Operating Temperature Characteristics
Glass
Plenum
Temperature (°C)
1400
1200
1000
800
600
400
200
0
8/7/01 0:00
8/7/01 2:00
8/7/01 4:00
8/7/01 6:00
8/7/01 8:00
8/7/01 10:00
8/7/01 12:00
8/7/01 14:00
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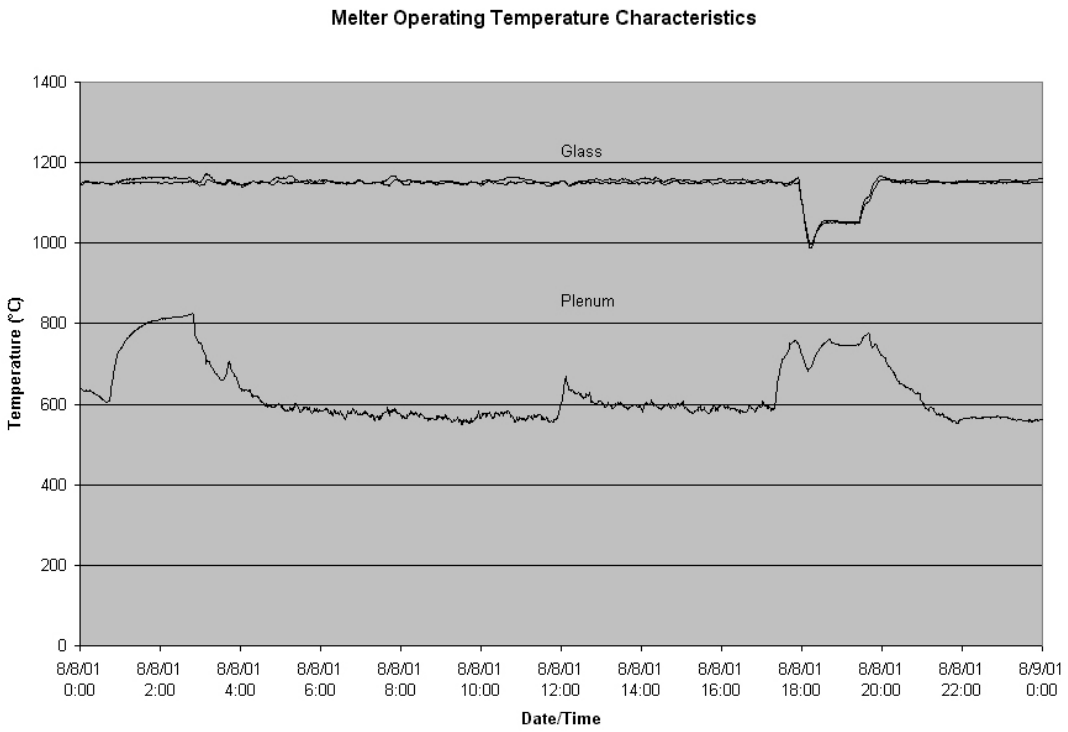
8/8/01 0:00
Date/Time


Melter Operating Temperature Characteristics
Glass
Plenum
Temperature (°C)
1400
1200
1000
800
600
400
200
0
8/8/01 0:00
8/8/01 2:00
8/8/01 4:00
8/8/01 6:00
8/8/01 8:00
8/8/01 10:00
8/8/01 12:00
8/8/01 14:00
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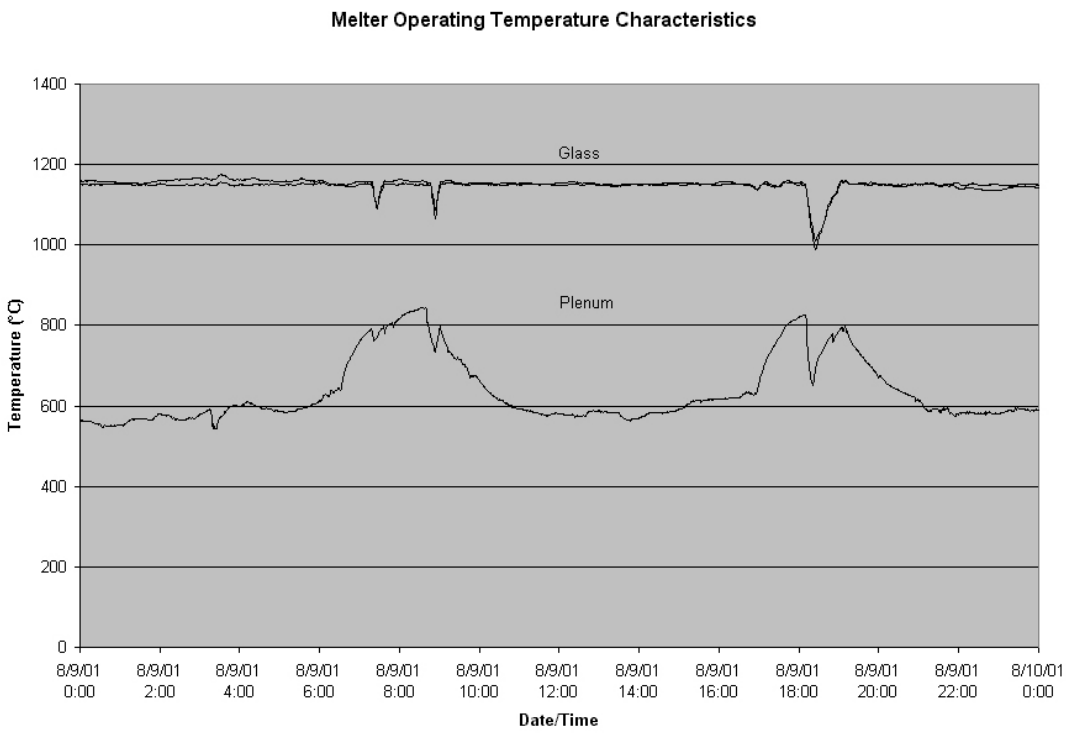
8/9/01 0:00
Date/Time


Melter Operating Temperature Characteristics
Glass
Plenum
Temperature (°C)
1400
1200
1000
800
600
400
200
0
8/9/01 0:00
8/9/01 2:00
8/9/01 4:00
8/9/01 6:00
8/9/01 8:00
8/9/01 10:00
8/9/01 12:00
8/9/01 14:00
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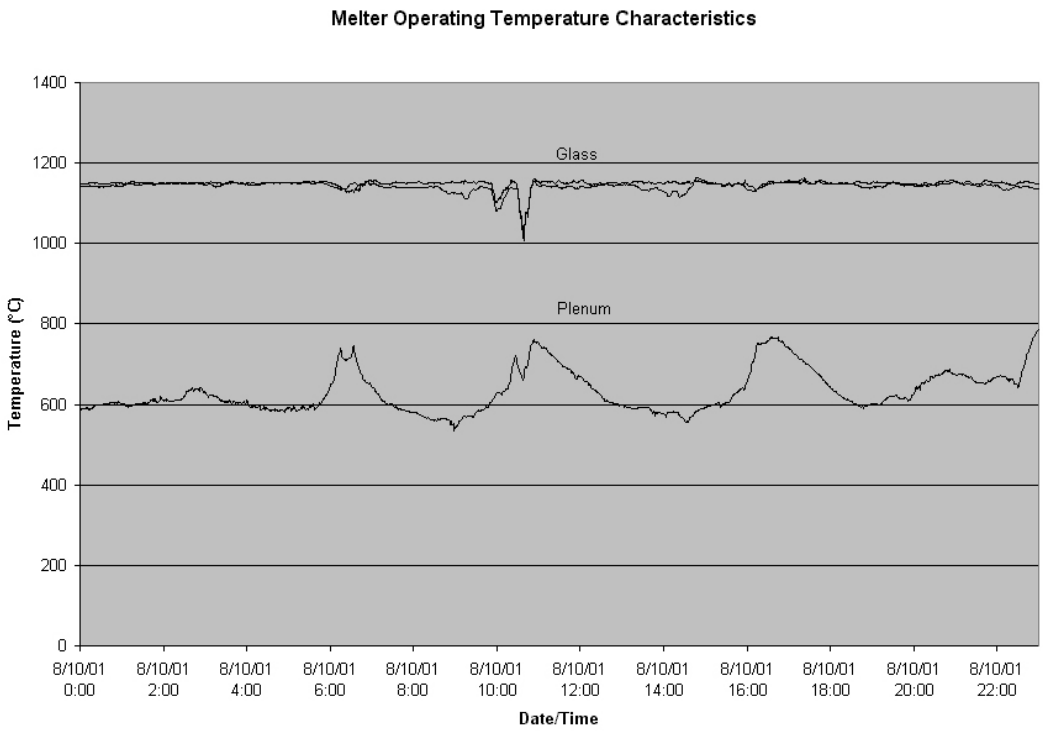
8/10/01 0:00
Date/Time


Melter Operating Temperature Characteristics
Glass
Plenum
Temperature (°C)
1400
1200
1000
800
600
400
200
0
8/10/01 0:00
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8/10/01 4:00
8/10/01 6:00
8/10/01 8:00
8/10/01 10:00
8/10/01 12:00
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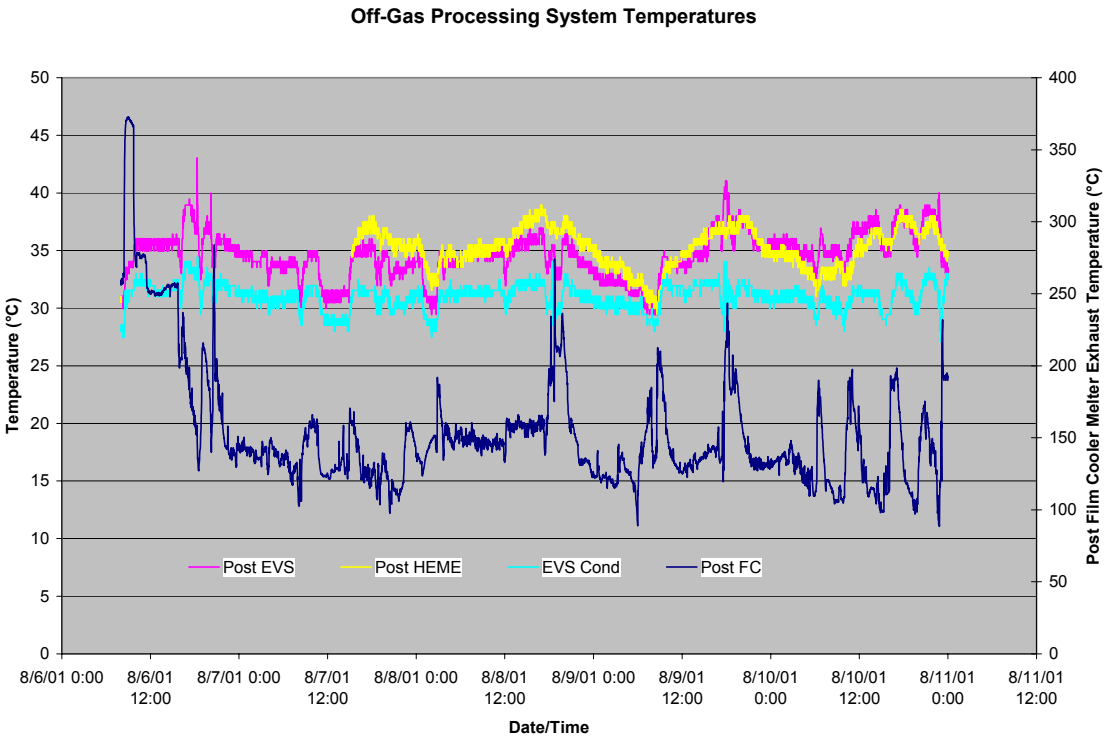
8/10/01 22:00
Date/Time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Temperature (°C)
Post Film Cooler Melter Exhaust Temperature (°C)
Post EVS
Post HEME
EVS Cond
Post FC
Date/Time
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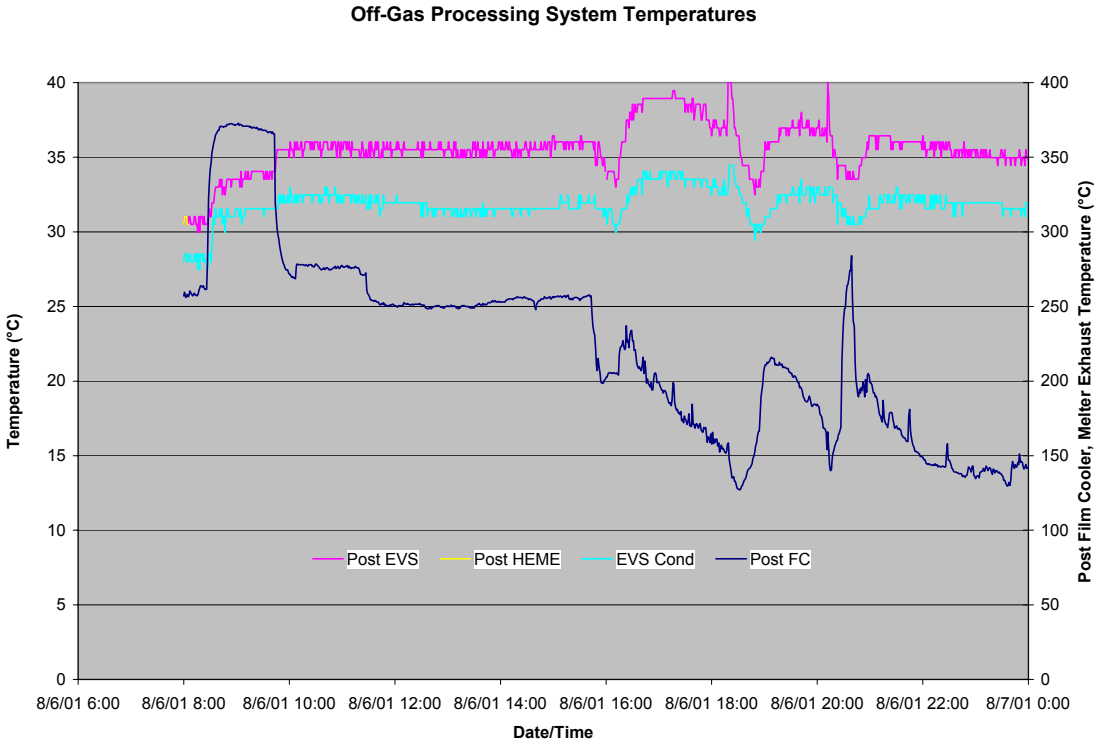
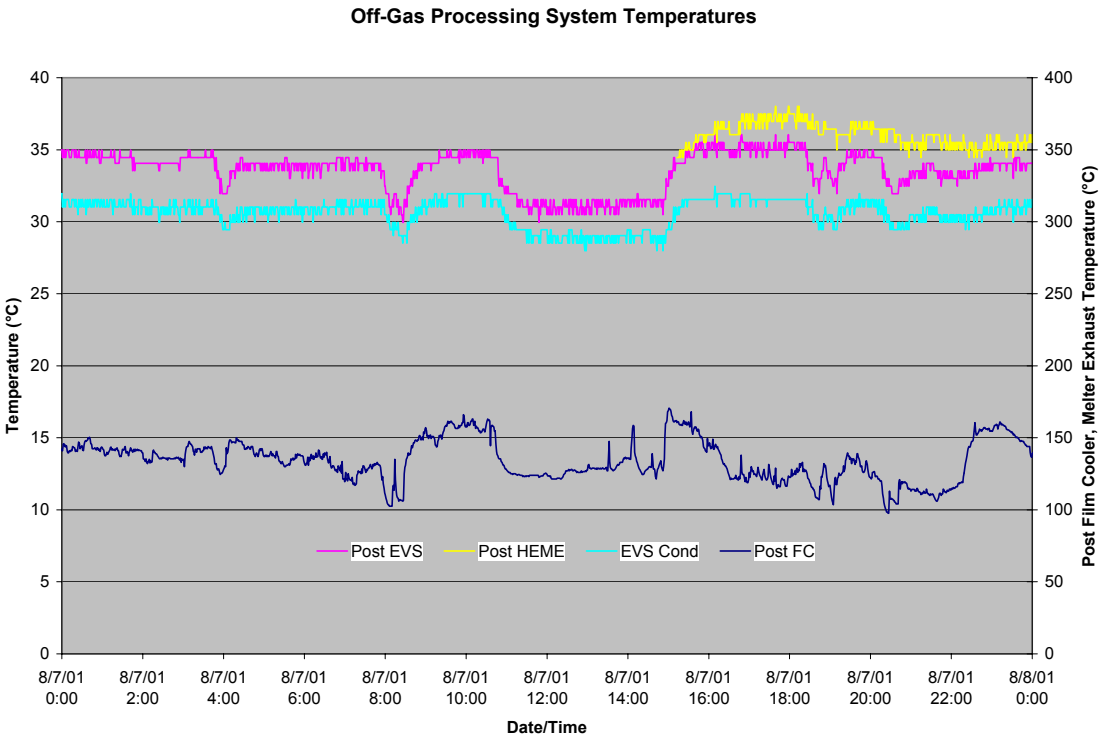
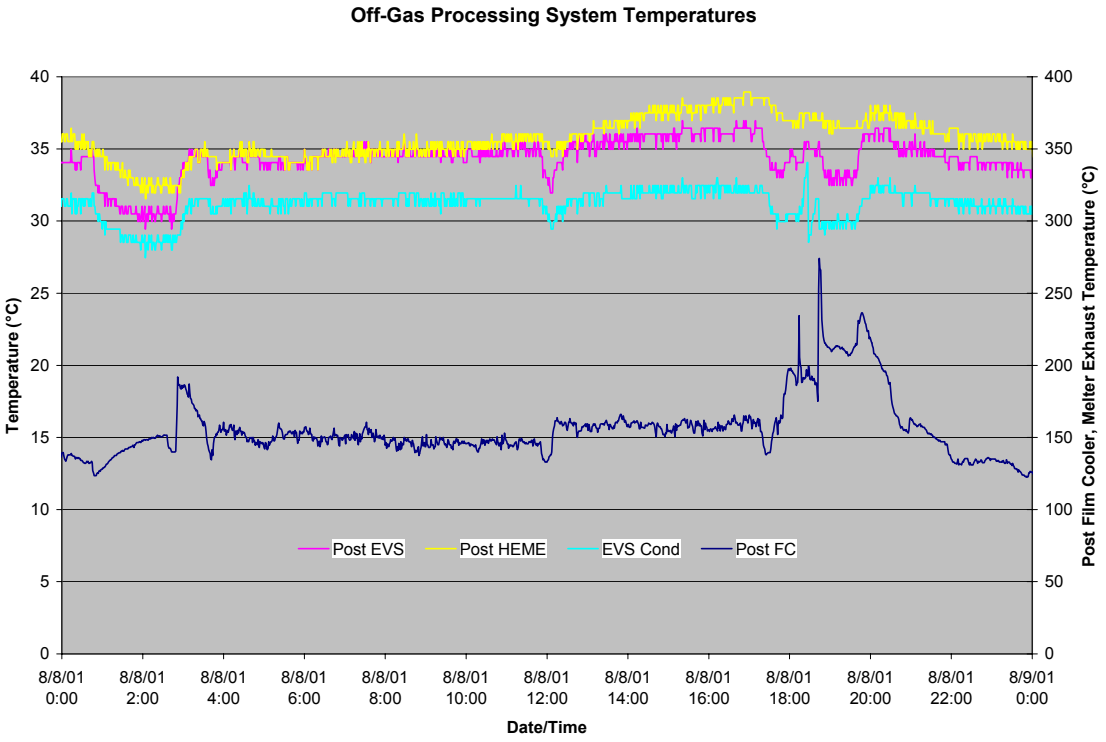
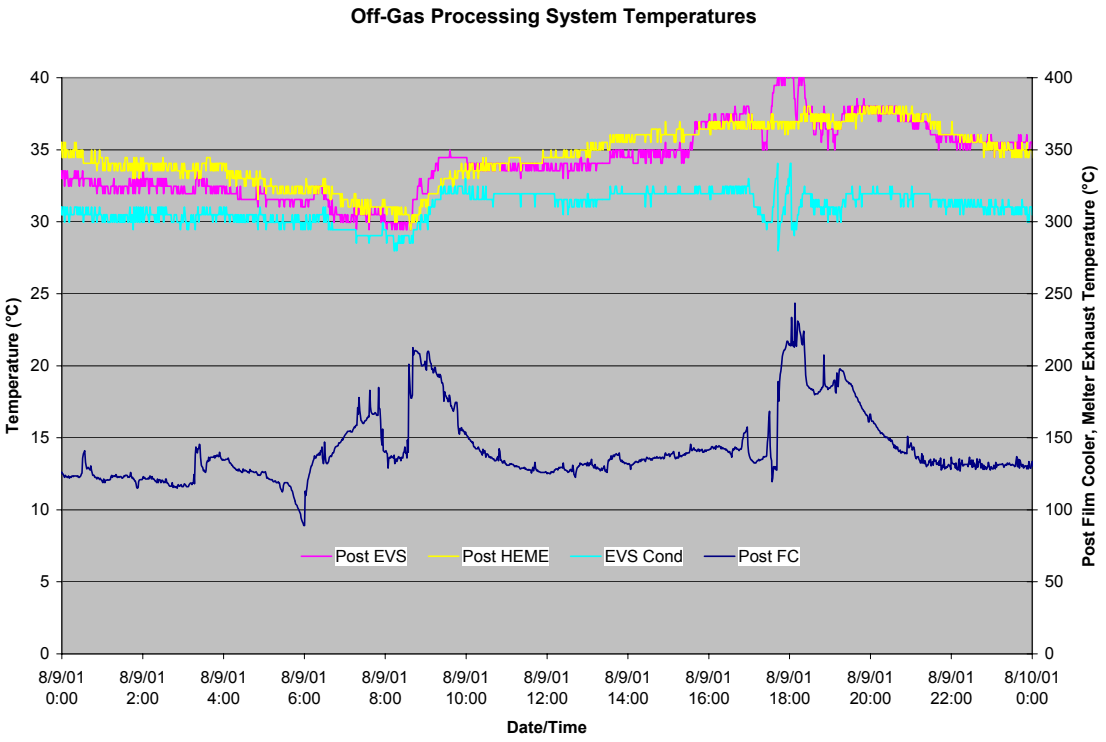
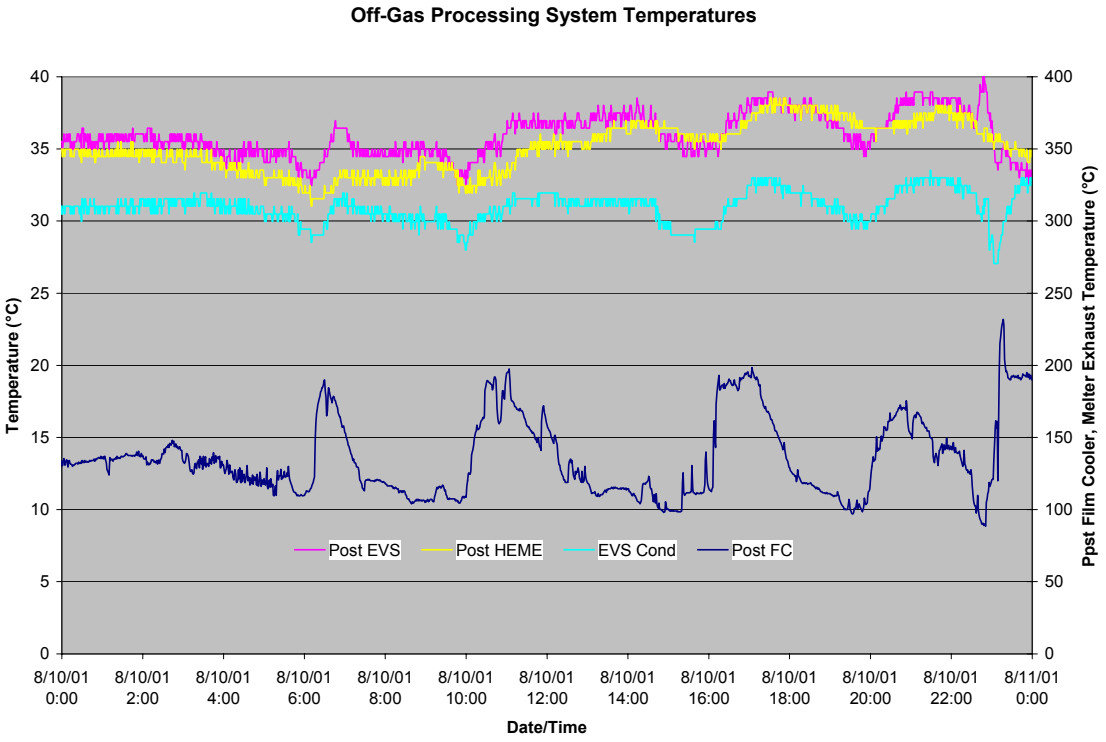
8/8/01 0:00
8/8/01 12:00
8/9/01 0:00
8/9/01 12:00
8/10/01 0:00
8/10/01 12:00
8/11/01 0:00
8/11/01 12:00


Off-Gas Processing System Temperatures
Temperature (°C)
Post Film Cooler, Melter Exhaust Temperature (°C)
Post EVS
Post HEME
EVS Cond
Post FC
Date/Time
8/6/01 6:00
8/6/01 8:00
8/6/01 10:00
8/6/01 12:00
8/6/01 14:00
8/6/01 16:00
8/6/01 18:00
8/6/01 20:00
8/6/01 22:00
8/7/01 0:00


Off-Gas Processing System Temperatures
Temperature (°C)
Post Film Cooler, Melter Exhaust Temperature (°C)
Post EVS
Post HEME
EVS Cond
Post FC
Date/Time
0
5
10
15
20
25
30
35
40
0
50
100
150
200
250
300
350
400
8/7/01 0:00
8/7/01 2:00
8/7/01 4:00
8/7/01 6:00
8/7/01 8:00
8/7/01 10:00
8/7/01 12:00
8/7/01 14:00
8/7/01 16:00
8/7/01 18:00
8/7/01 20:00
8/7/01 22:00
8/8/01 0:00


Off-Gas Processing System Temperatures
Temperature (°C)
Post Film Cooler, Melter Exhaust Temperature (°C)
Post EVS
Post HEME
EVS Cond
Post FC
Date/Time
0
5
10
15
20
25
30
35
40
0
50
100
150
200
250
300
350
400
8/8/01 0:00
8/8/01 2:00
8/8/01 4:00
8/8/01 6:00
8/8/01 8:00
8/8/01 10:00
8/8/01 12:00
8/8/01 14:00
8/8/01 16:00
8/8/01 18:00
8/8/01 20:00
8/8/01 22:00
8/9/01 0:00

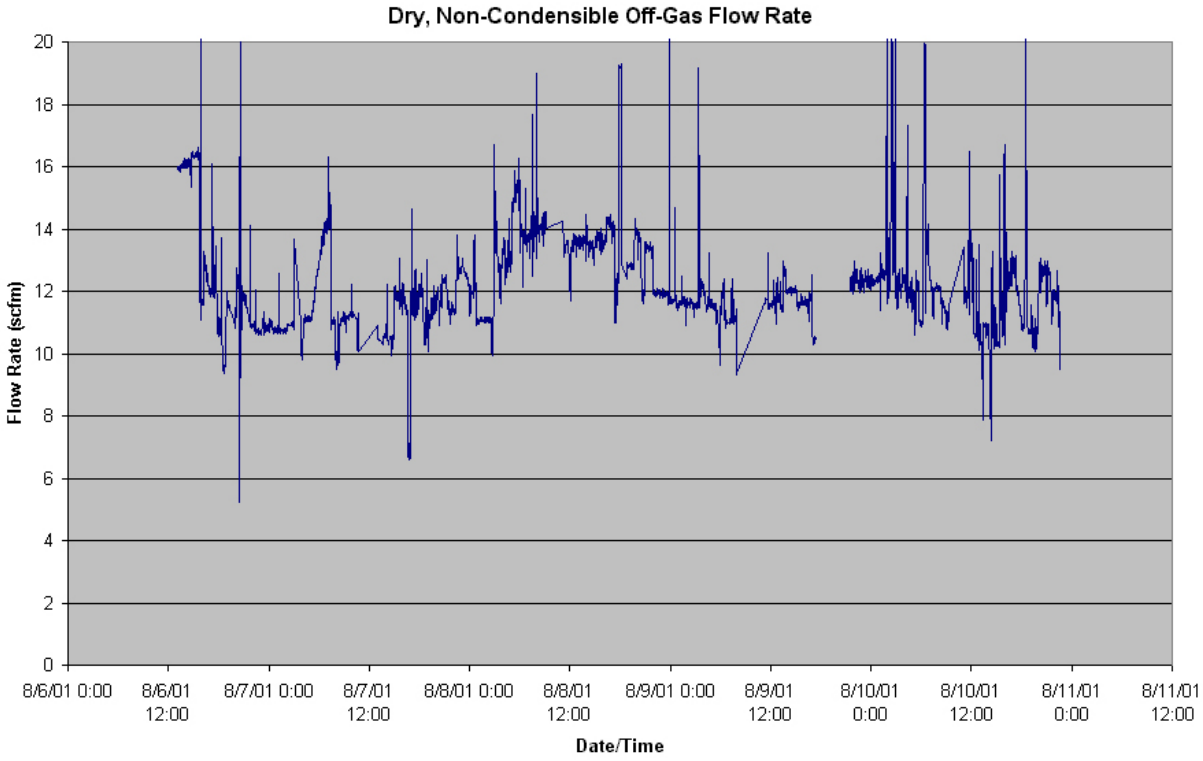

Off-Gas Processing System Temperatures
Temperature (°C)
Post Film Cooler, Melter Exhaust Temperature (°C)
Post EVS
Post HEME
EVS Cond
Post FC
Date/Time
0
5
10
15
20
25
30
35
40
0
50
100
150
200
250
300
350
400
8/9/01 0:00
8/9/01 2:00
8/9/01 4:00
8/9/01 6:00
8/9/01 8:00
8/9/01 10:00
8/9/01 12:00
8/9/01 14:00
8/9/01 16:00
8/9/01 18:00
8/9/01 20:00
8/9/01 22:00
8/10/01 0:00

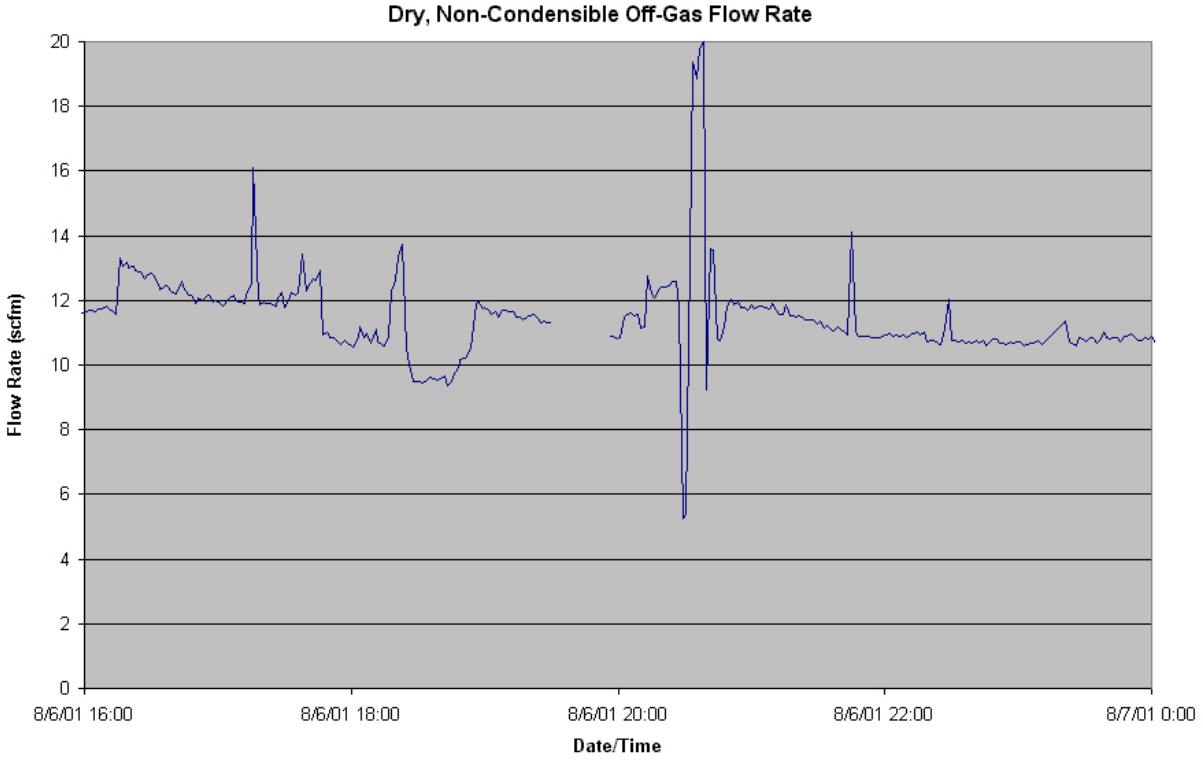

Off-Gas Processing System Temperatures
Temperature (°C)
Ppst Film Cooler, Melter Exhaust Temperature (°C)
Post EVS
Post HEME
EVS Cond
Post FC
Date/Time
0
5
10
15
20
25
30
35
40
0
50
100
150
200
250
300
350
400
8/10/01 0:00
8/10/01 2:00
8/10/01 4:00
8/10/01 6:00
8/10/01 8:00
8/10/01 10:00
8/10/01 12:00
8/10/01 14:00
8/10/01 16:00
8/10/01 18:00
8/10/01 20:00
8/10/01 22:00
8/11/01 0:00

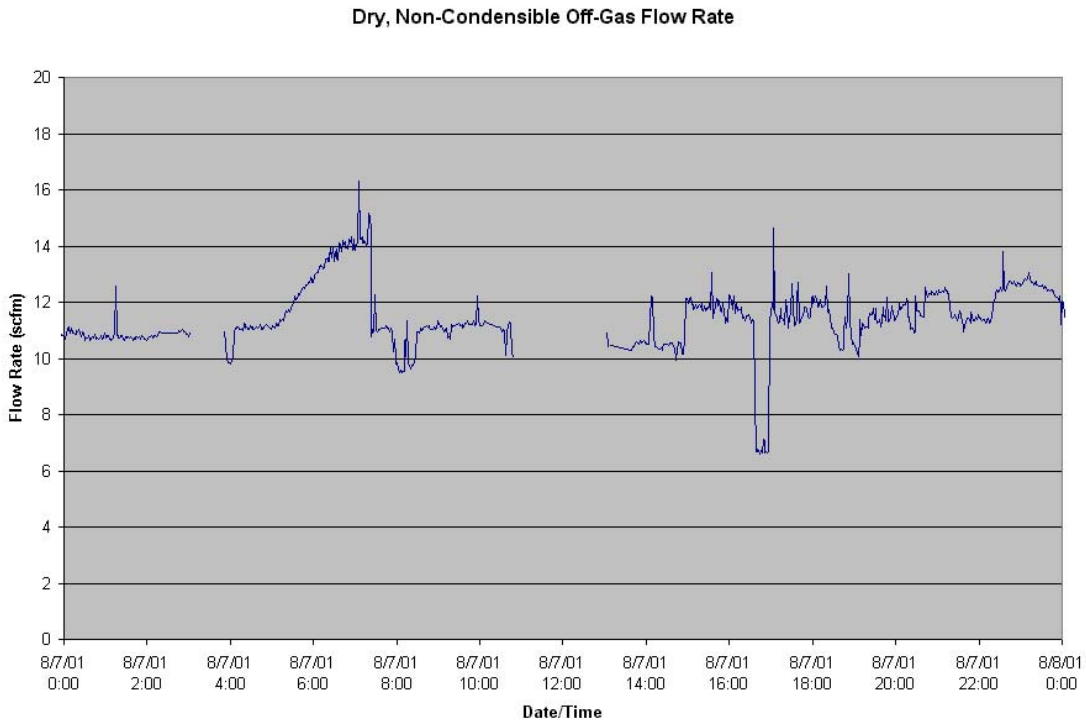
Appendix F

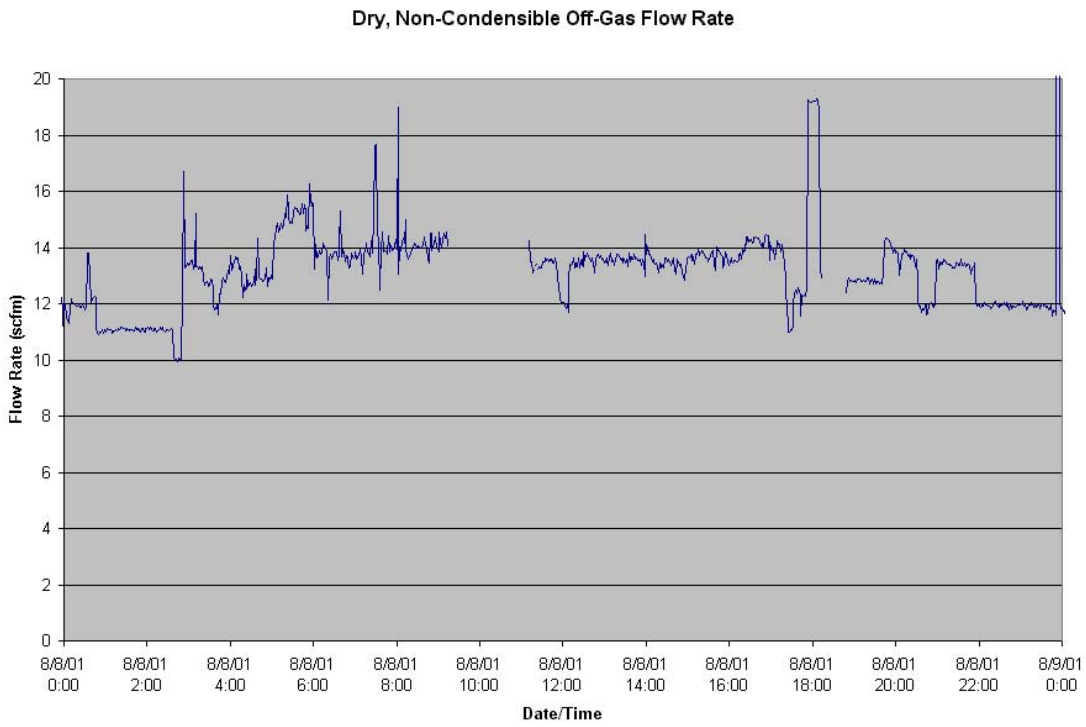
Melter Off-Gas Flow Rates (Dry Basis)

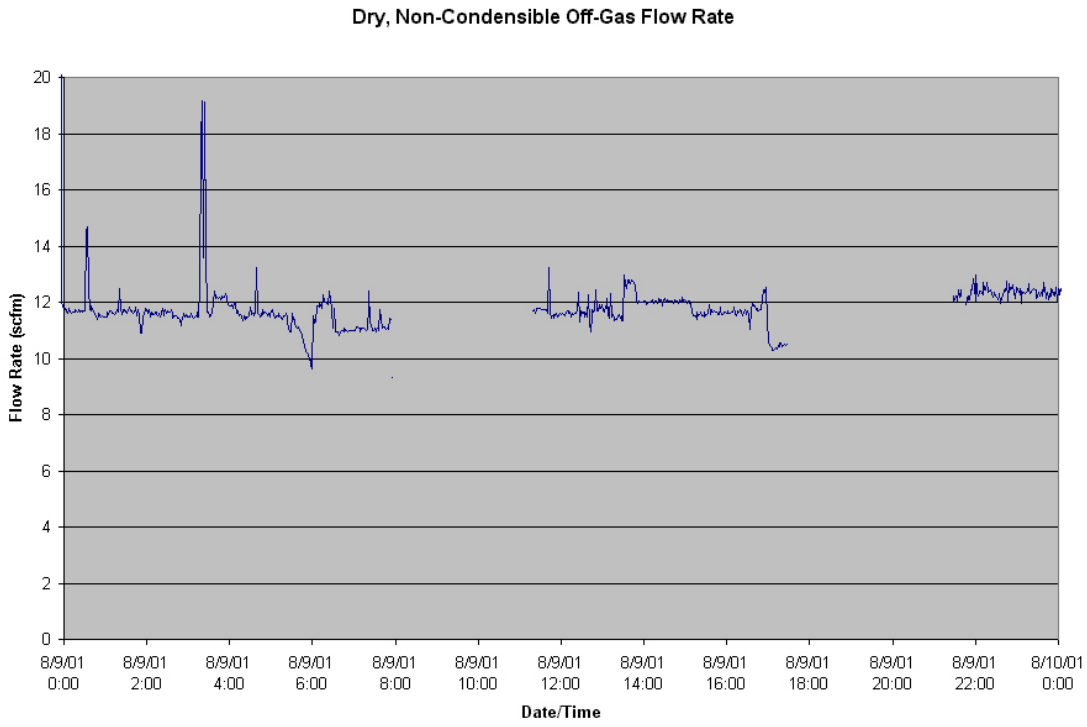
Appendix F: Melter Off-Gas Flow Rates (Dry Basis)

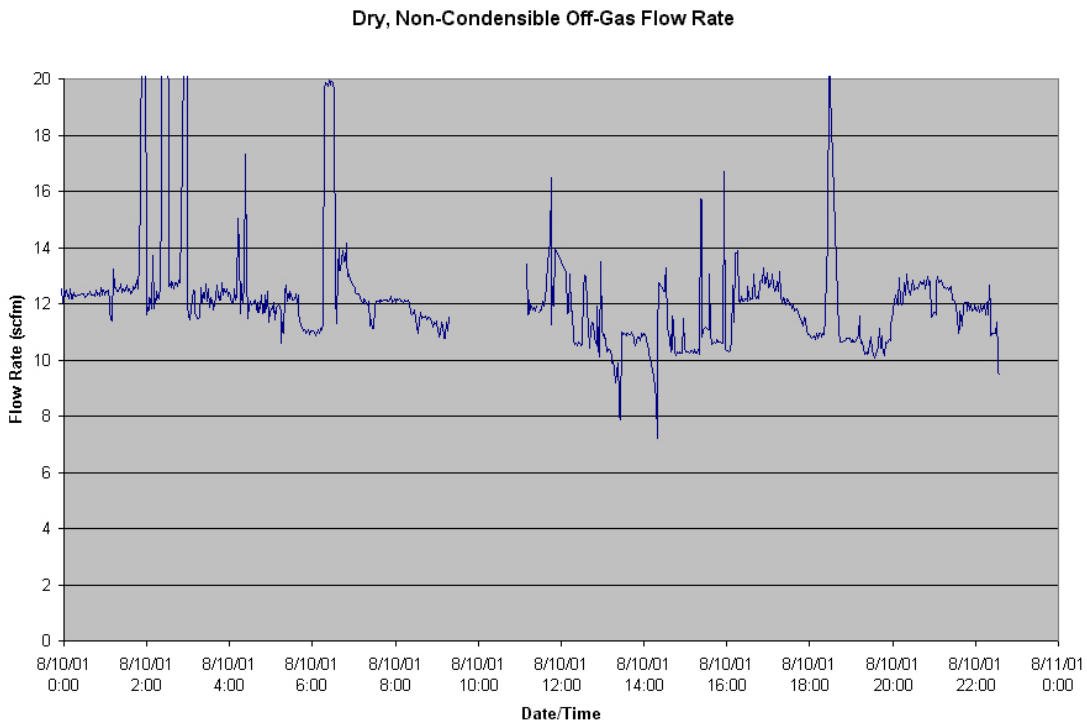





Dry, Non-Condensible Off-Gas Flow Rate
Flow Rate (scfm)
20
18
16
14
12
10
8
6
4
2
0
8/7/01 0:00
8/7/01 2:00
8/7/01 4:00
8/7/01 6:00
8/7/01 8:00
8/7/01 10:00
8/7/01 12:00
8/7/01 14:00
8/7/01 16:00
8/7/01 18:00
8/7/01 20:00
8/7/01 22:00
8/8/01 0:00
Date/Time


Dry, Non-Condensible Off-Gas Flow Rate
Flow Rate (scfm)
20
18
16
14
12
10
8
6
4
2
0
8/8/01 0:00
8/8/01 2:00
8/8/01 4:00
8/8/01 6:00
8/8/01 8:00
8/8/01 10:00
8/8/01 12:00
8/8/01 14:00
8/8/01 16:00
8/8/01 18:00
8/8/01 20:00
8/8/01 22:00
8/9/01 0:00
Date/Time

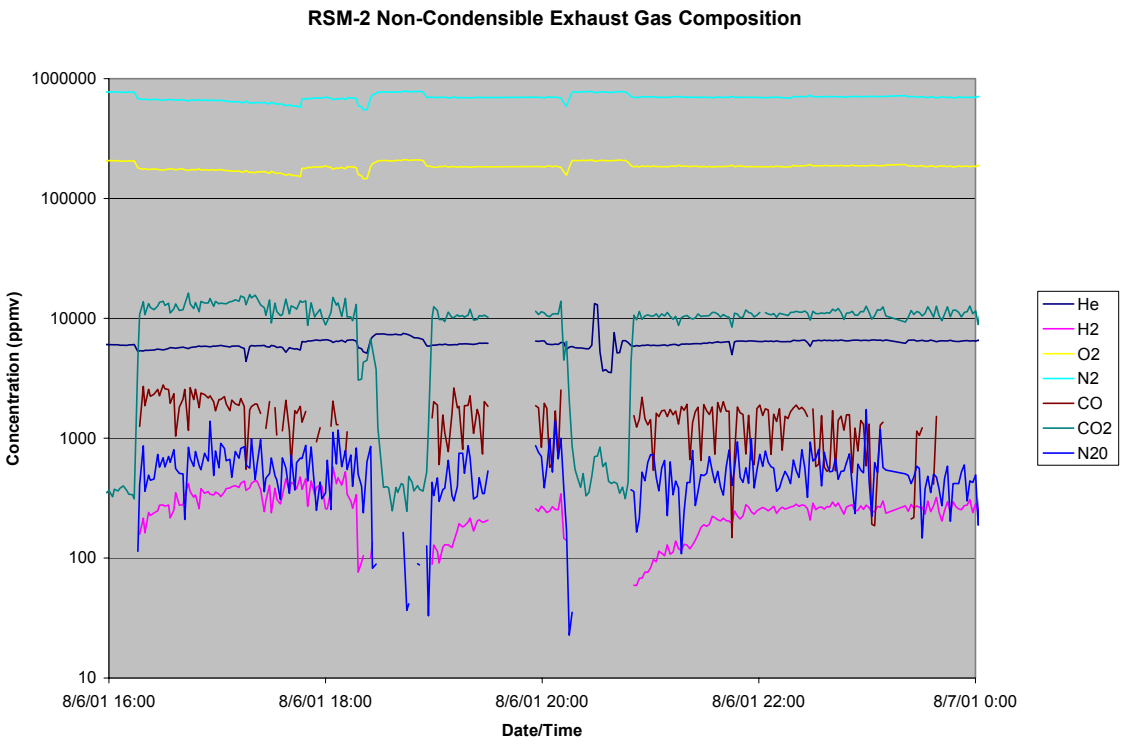

Dry, Non-Condensible Off-Gas Flow Rate
Flow Rate (scfm)
20
18
16
14
12
10
8
6
4
2
0
8/9/01 0:00
8/9/01 2:00
8/9/01 4:00
8/9/01 6:00
8/9/01 8:00
8/9/01 10:00
8/9/01 12:00
8/9/01 14:00
8/9/01 16:00
8/9/01 18:00
8/9/01 20:00
8/9/01 22:00
8/10/01 0:00
Date/Time

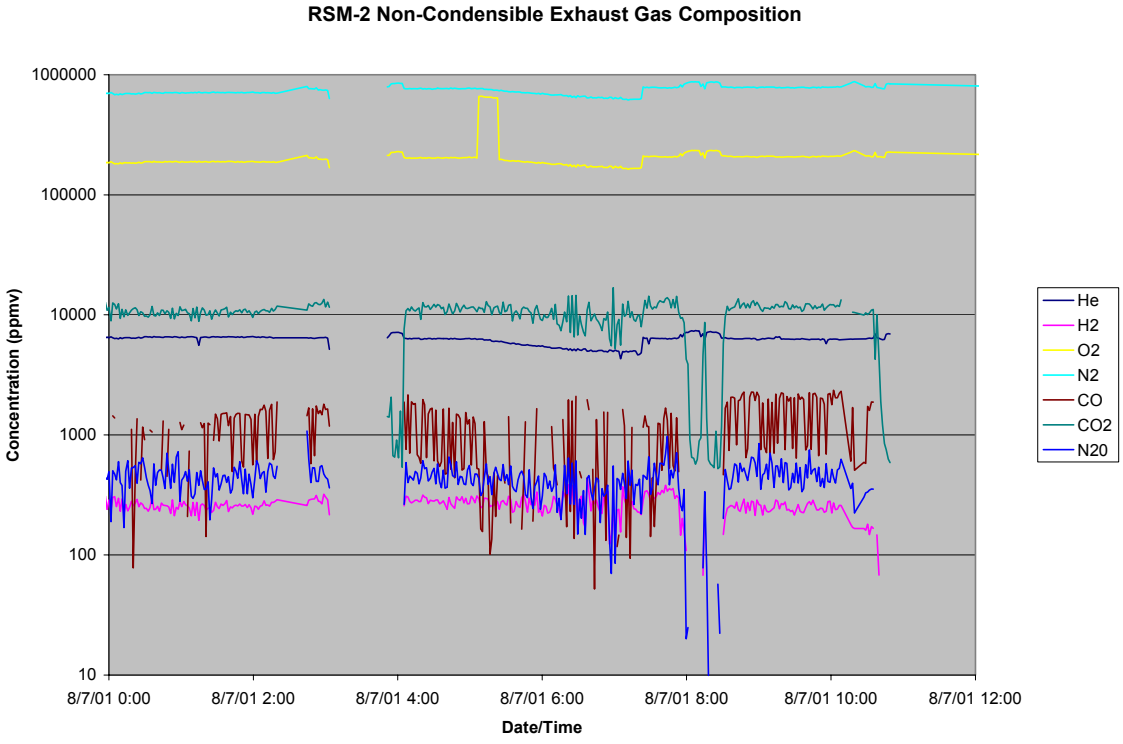

Dry, Non-Condensible Off-Gas Flow Rate
Flow Rate (scfm)
20
18
16
14
12
10
8
6
4
2
0
8/10/01 0:00
8/10/01 2:00
8/10/01 4:00
8/10/01 6:00
8/10/01 8:00
8/10/01 10:00
8/10/01 12:00
8/10/01 14:00
8/10/01 16:00
8/10/01 18:00
8/10/01 20:00
8/10/01 22:00
8/11/01 0:00
Date/Time

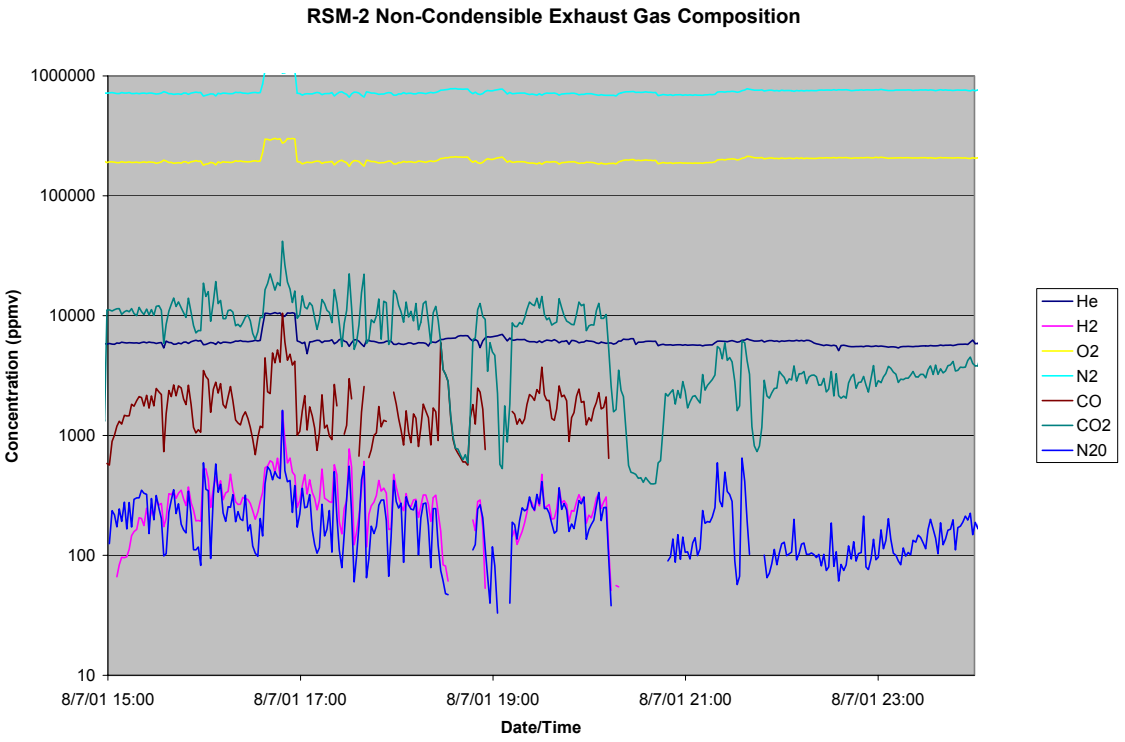
Appendix G

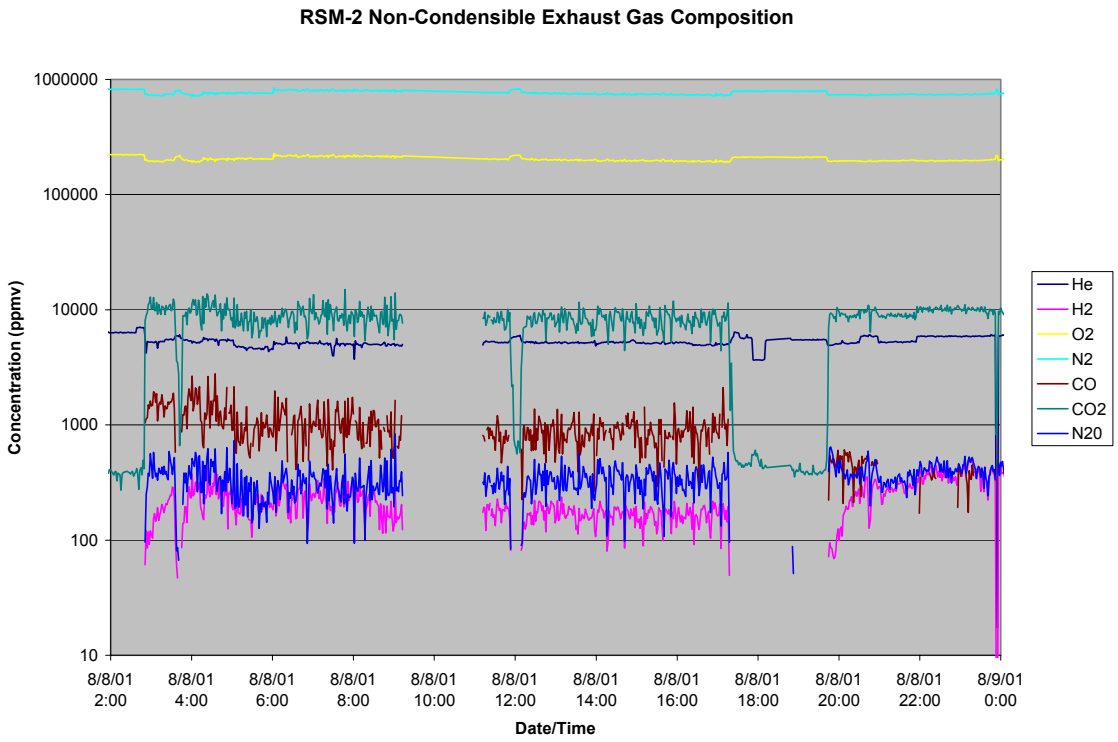
Temporal Behavior of Melter Off-Gas Effluents

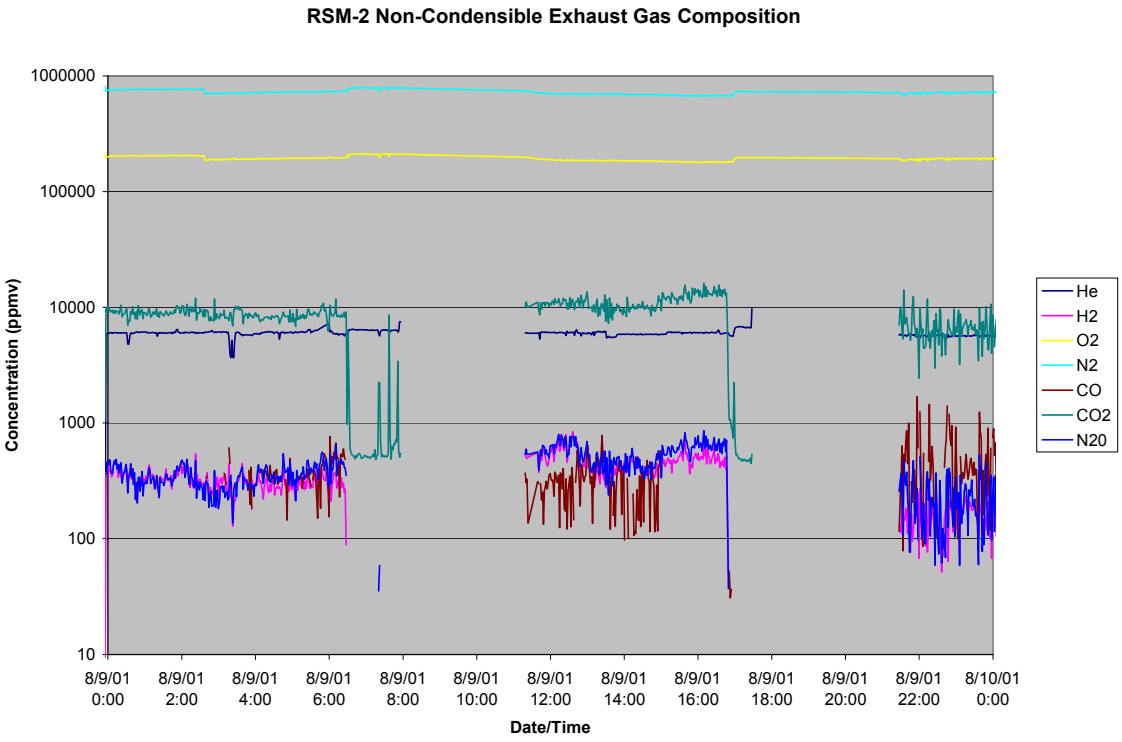
Appendix G: Temporal Behavior of Melter Off-Gas Effluents

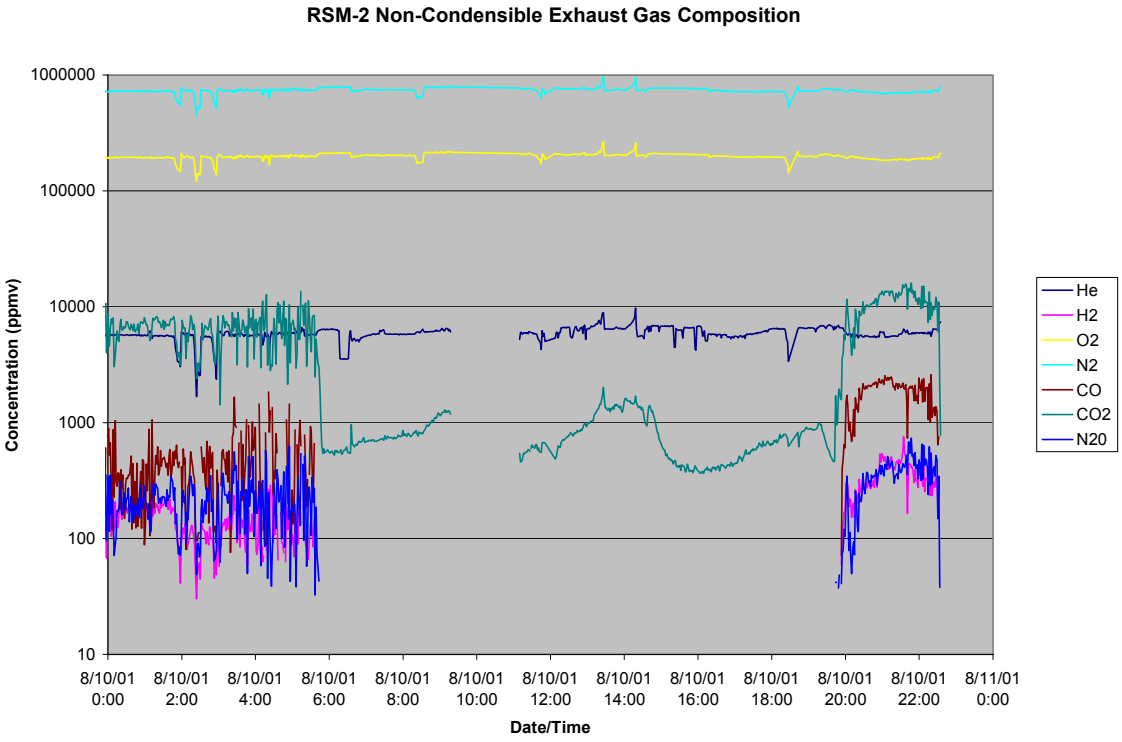


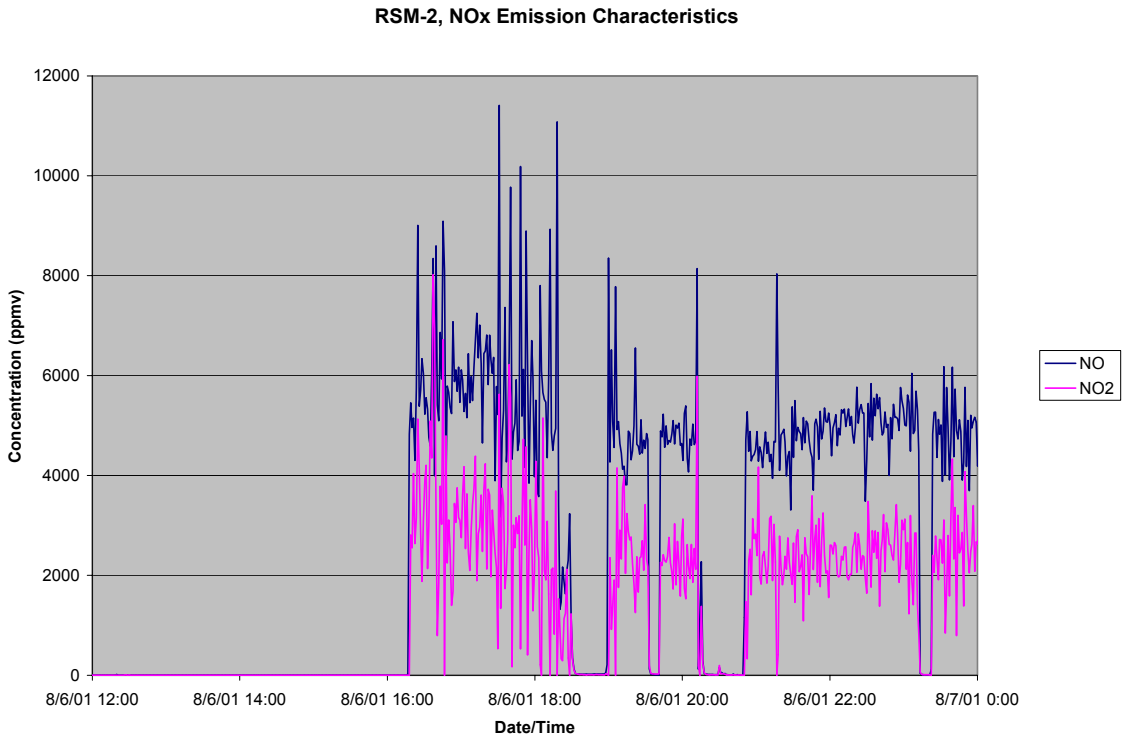


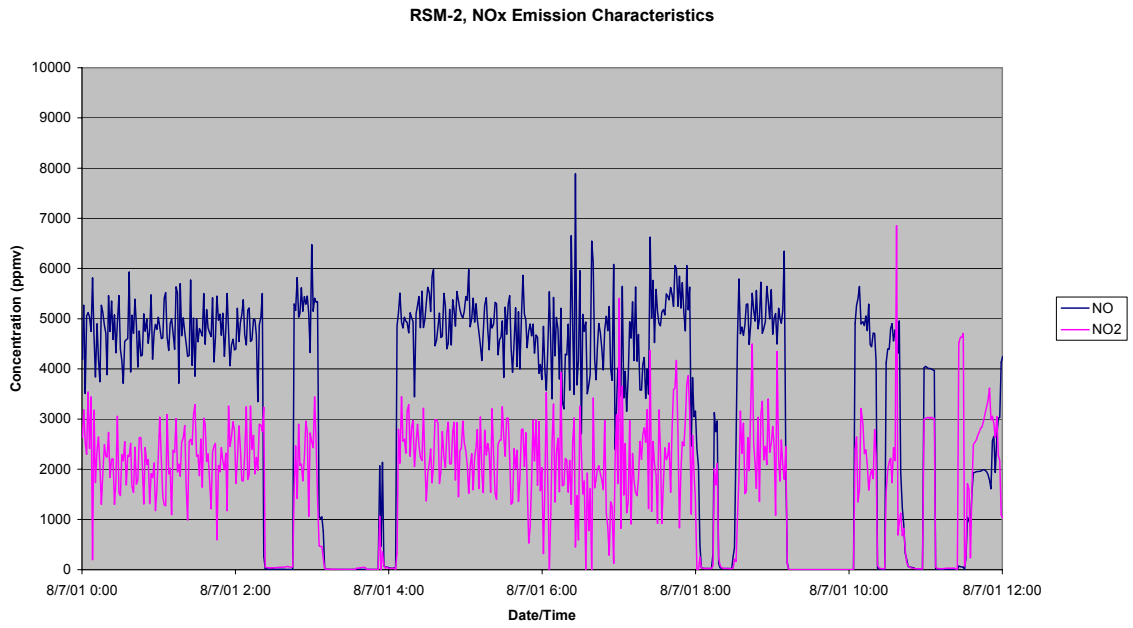

RSM-2 Non-Condensible Exhaust Gas Composition
1000000
100000
10000
1000
100
10
Concentration (ppmv)
8/7/01 15:00
8/7/01 17:00
8/7/01 19:00
8/7/01 21:00
8/7/01 23:00
Date/Time
He
H2
O2
N2
CO
CO2
N20

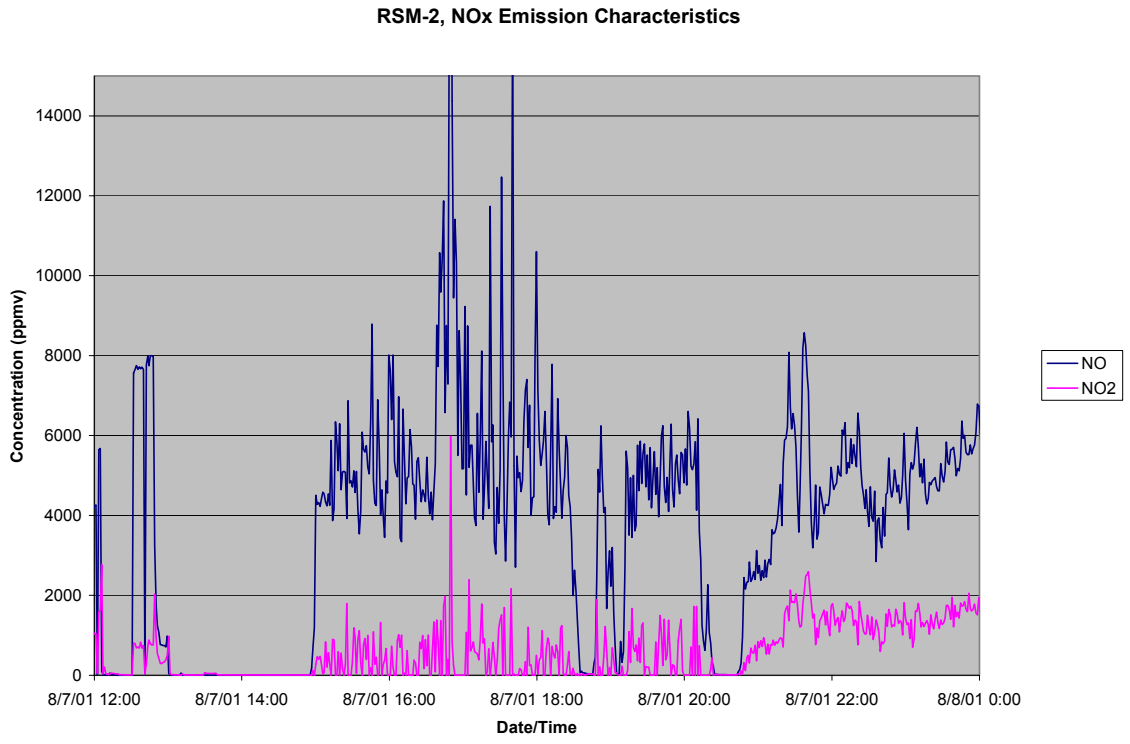

RSM-2 Non-Condensible Exhaust Gas Composition
1000000
100000
10000
1000
100
10
Concentration (ppmv)
8/8/01 2:00
8/8/01 4:00
8/8/01 6:00
8/8/01 8:00
8/8/01 10:00
8/8/01 12:00
8/8/01 14:00
8/8/01 16:00
8/8/01 18:00
8/8/01 20:00
8/8/01 22:00
8/9/01 0:00
Date/Time
He
H2
O2
N2
CO
CO2
N20

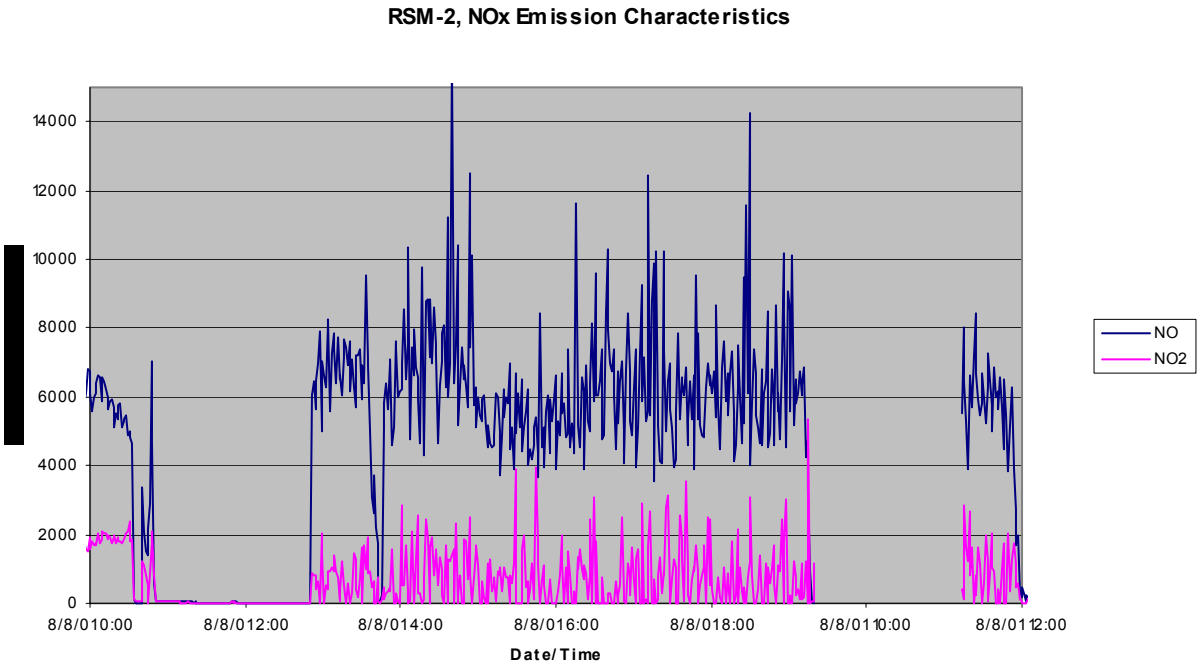

RSM-2 Non-Condensible Exhaust Gas Composition
Concentration (ppmv)
1000000
100000
10000
1000
100
10
8/9/01 0:00
8/9/01 2:00
8/9/01 4:00
8/9/01 6:00
8/9/01 8:00
8/9/01 10:00
8/9/01 12:00
8/9/01 14:00
8/9/01 16:00
8/9/01 18:00
8/9/01 20:00
8/9/01 22:00
8/10/01 0:00
Date/Time
He
H2
O2
N2
CO
CO2
N20

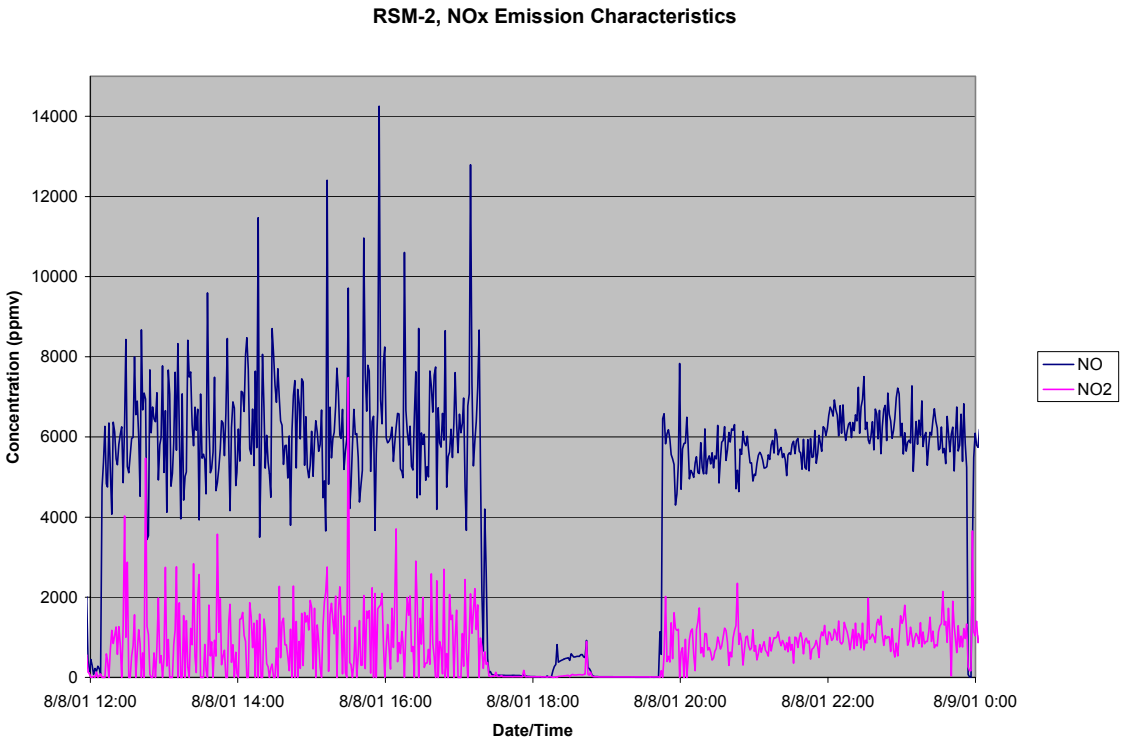

RSM-2 Non-Condensible Exhaust Gas Composition
Concentration (ppmv)
1000000
100000
10000
1000
100
10
8/10/01 0:00
8/10/01 2:00
8/10/01 4:00
8/10/01 6:00
8/10/01 8:00
8/10/01 10:00
8/10/01 12:00
8/10/01 14:00
8/10/01 16:00
8/10/01 18:00
8/10/01 20:00
8/10/01 22:00
8/11/01 0:00
Date/Time
He
H2
O2
N2
CO
CO2
N20

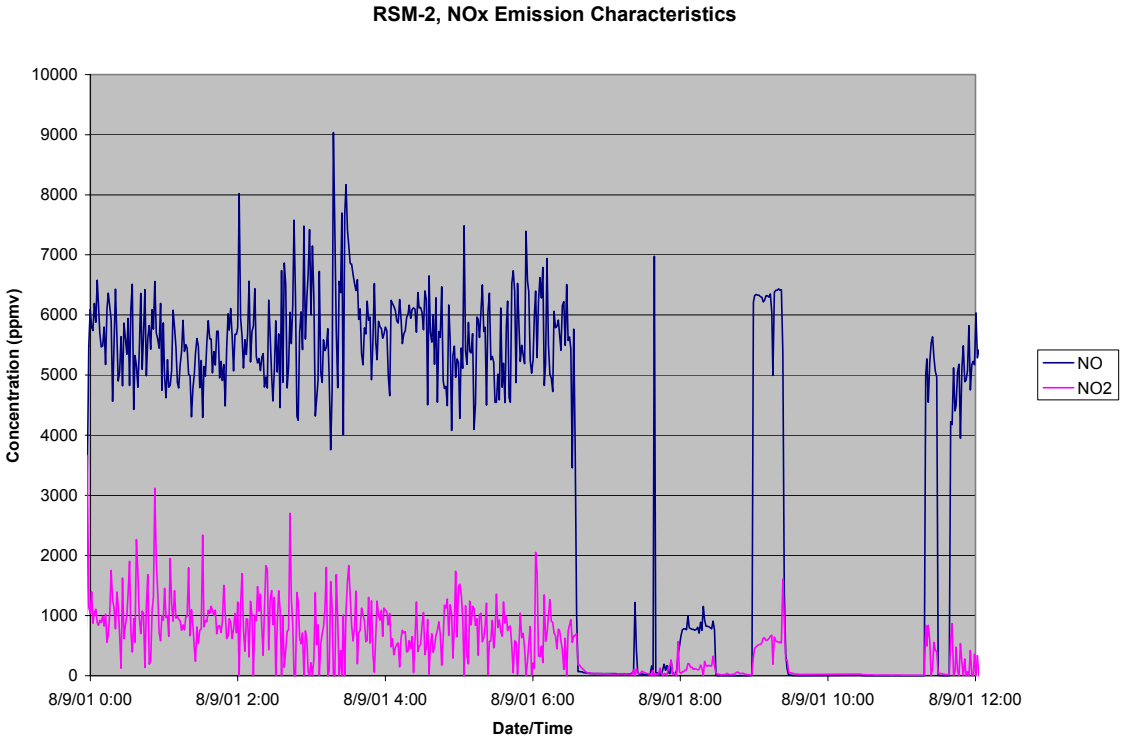

RSM-2, NOx Emission Characteristics
Concentration (ppmv)
12000
10000
8000
6000
4000
2000
0
8/6/01 12:00
8/6/01 14:00
8/6/01 16:00
8/6/01 18:00
8/6/01 20:00
8/6/01 22:00
8/7/01 0:00
Date/Time
NO
NO2

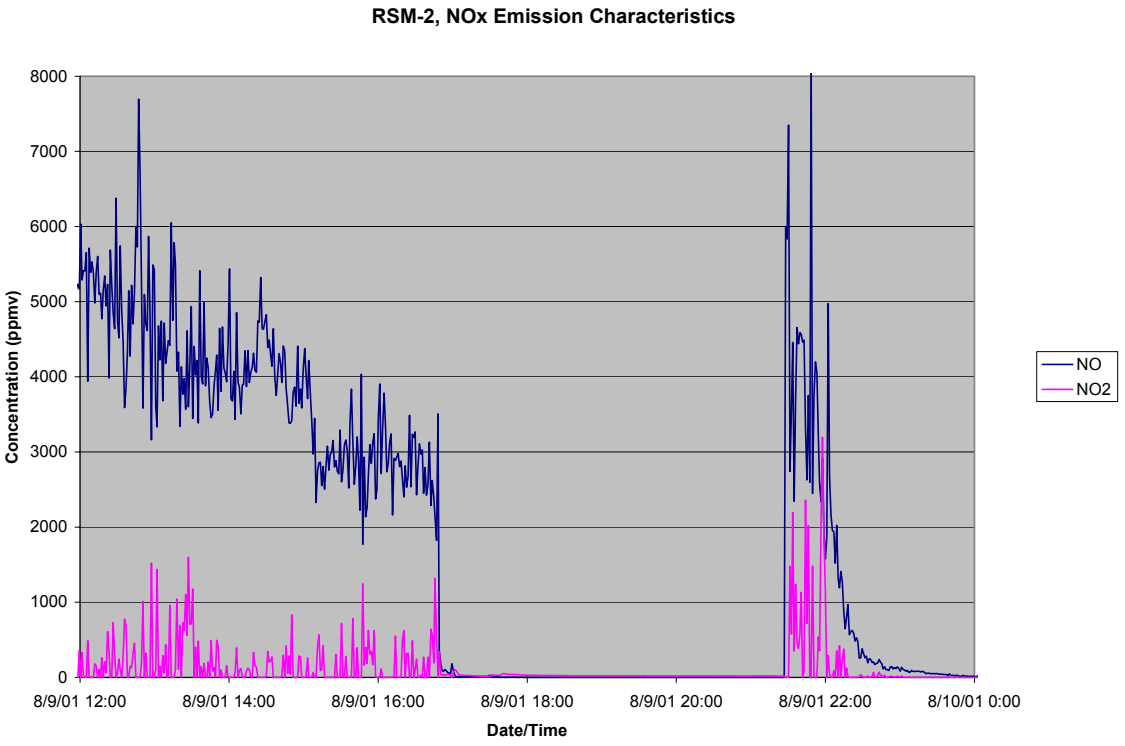

RSM-2, NOx Emission Characteristics
Concentration (ppmv)
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
8/7/01 0:00
8/7/01 2:00
8/7/01 4:00
8/7/01 6:00
8/7/01 8:00
8/7/01 10:00
8/7/01 12:00
Date/Time
NO
NO2

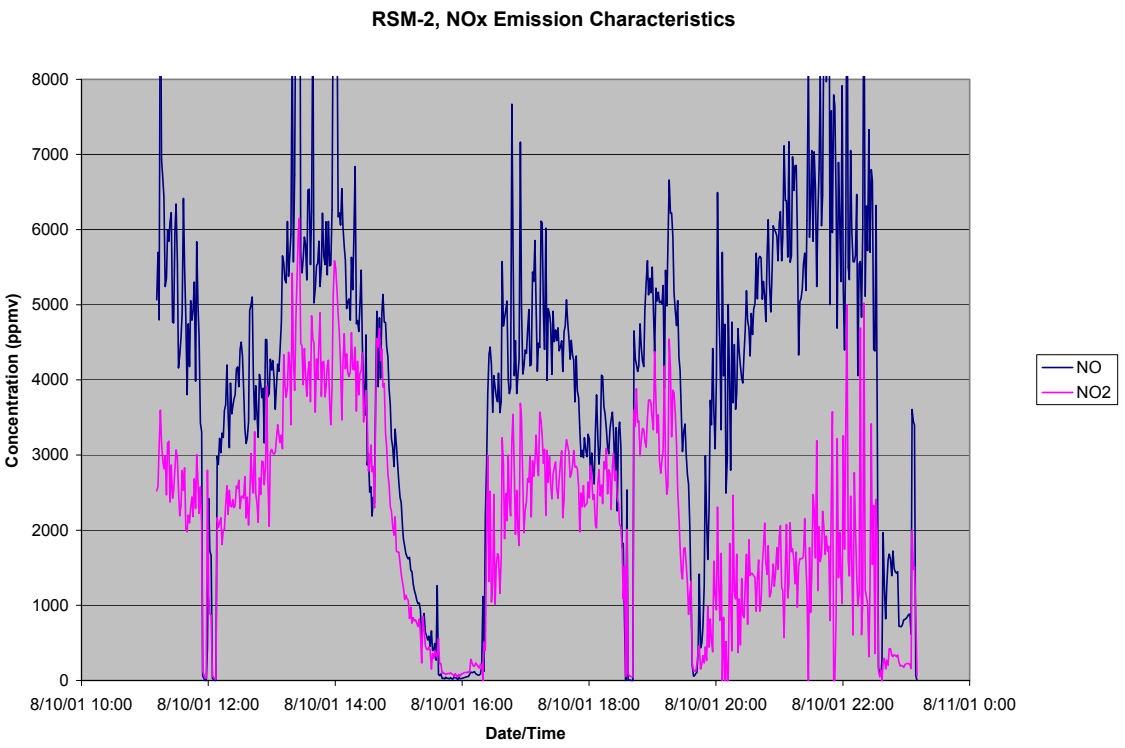

RSM-2, NOx Emission Characteristics
Concentration (ppmv)
14000
12000
10000
8000
6000
4000
2000
0
8/7/01 12:00
8/7/01 14:00
8/7/01 16:00
8/7/01 18:00
8/7/01 20:00
8/7/01 22:00
8/8/01 0:00
Date/Time
NO
NO2


RSM-2, NOx Emission Characteristics
14000
12000
10000
8000
6000
4000
2000
0
8/8/010:00
8/8/012:00
8/8/014:00
8/8/016:00
8/8/018:00
8/8/010:00
8/8/012:00
Date/Time
NO
NO2


RSM-2, NOx Emission Characteristics
Concentration (ppmv)
14000
12000
10000
8000
6000
4000
2000
0
8/8/01 12:00
8/8/01 14:00
8/8/01 16:00
8/8/01 18:00
8/8/01 20:00
8/8/01 22:00
8/9/01 0:00
Date/Time
NO
NO2


RSM-2, NOx Emission Characteristics
Concentration (ppmv)
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
8/9/01 0:00
8/9/01 2:00
8/9/01 4:00
8/9/01 6:00
8/9/01 8:00
8/9/01 10:00
8/9/01 12:00
Date/Time
NO
NO2


RSM-2, NOx Emission Characteristics
Concentration (ppmv)
8000
7000
6000
5000
4000
3000
2000
1000
0
8/9/01 12:00
8/9/01 14:00
8/9/01 16:00
8/9/01 18:00
8/9/01 20:00
8/9/01 22:00
8/10/01 0:00
Date/Time
NO
NO2


RSM-2, NOx Emission Characteristics
Concentration (ppmv)
8000
7000
6000
5000
4000
3000
2000
1000
0
8/10/01 10:00
8/10/01 12:00
8/10/01 14:00
8/10/01 16:00
8/10/01 18:00
8/10/01 20:00
8/10/01 22:00
8/11/01 0:00
Date/Time
NO
NO2

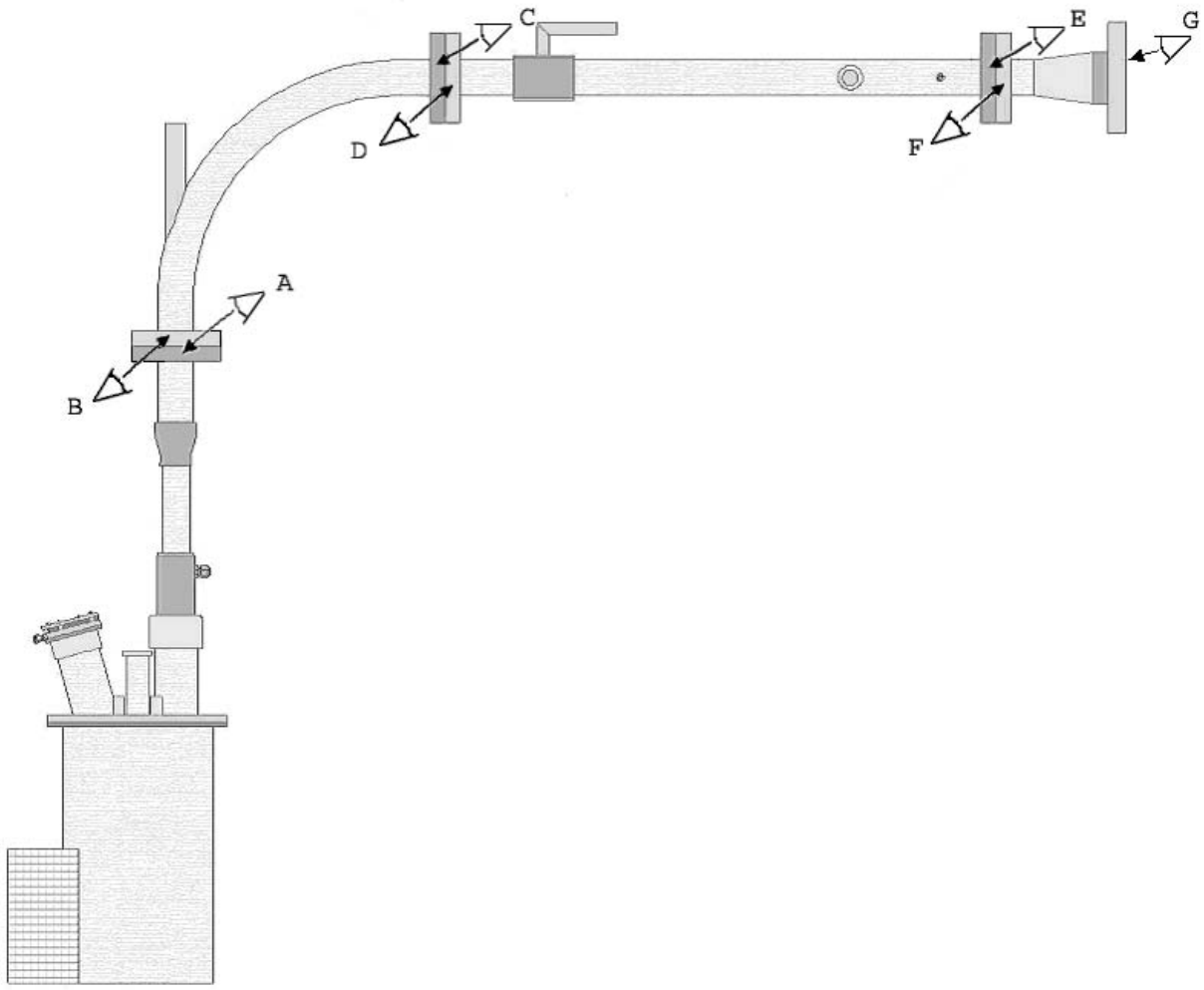
Appendix H

Photographs of Melter Feed Nozzle Off-Gas Line Deposits and Glass Canisters

Appendix H: Photographs of Melter Feed Nozzle Off-Gas Line Deposits and Glass Canisters

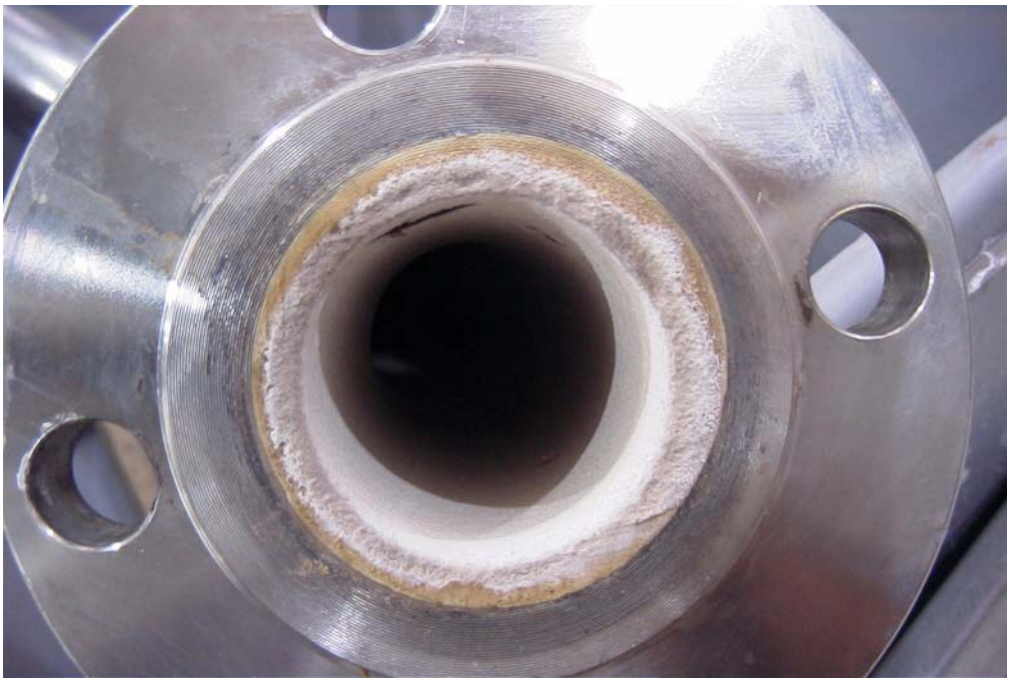
1-Feed Nozzle

2-Feed Nozzle Tip



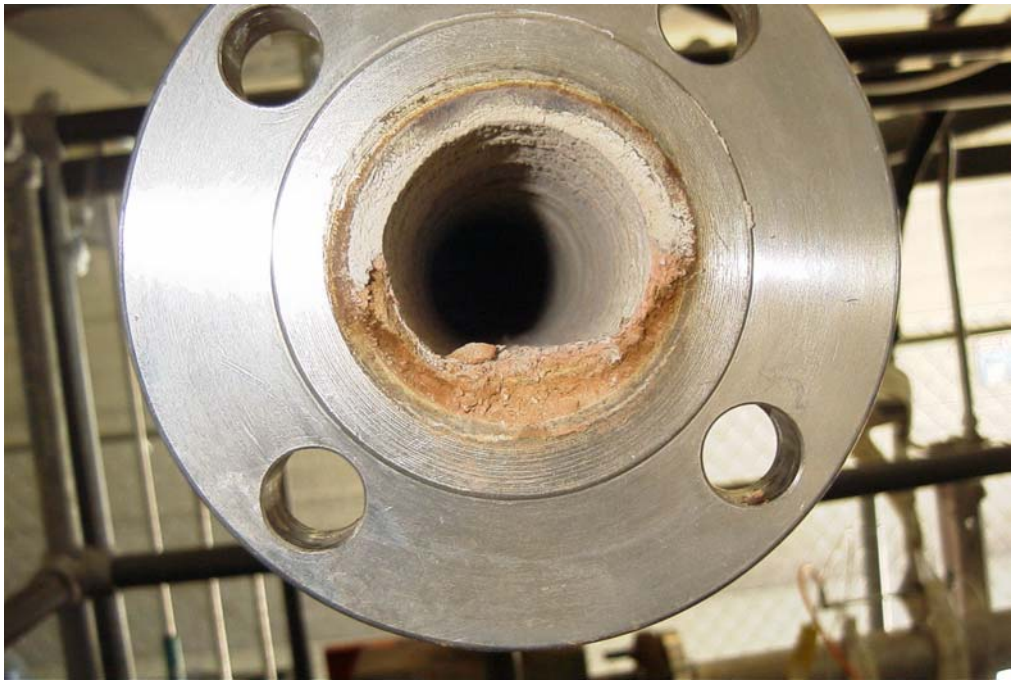
3-RSM Off-gas Line Component Views

A-Film-Cooler Outlet



B-Inlet to Curved Elbow

C-Curved Elbow Outlet



D-Horizontal Pipe Inlet

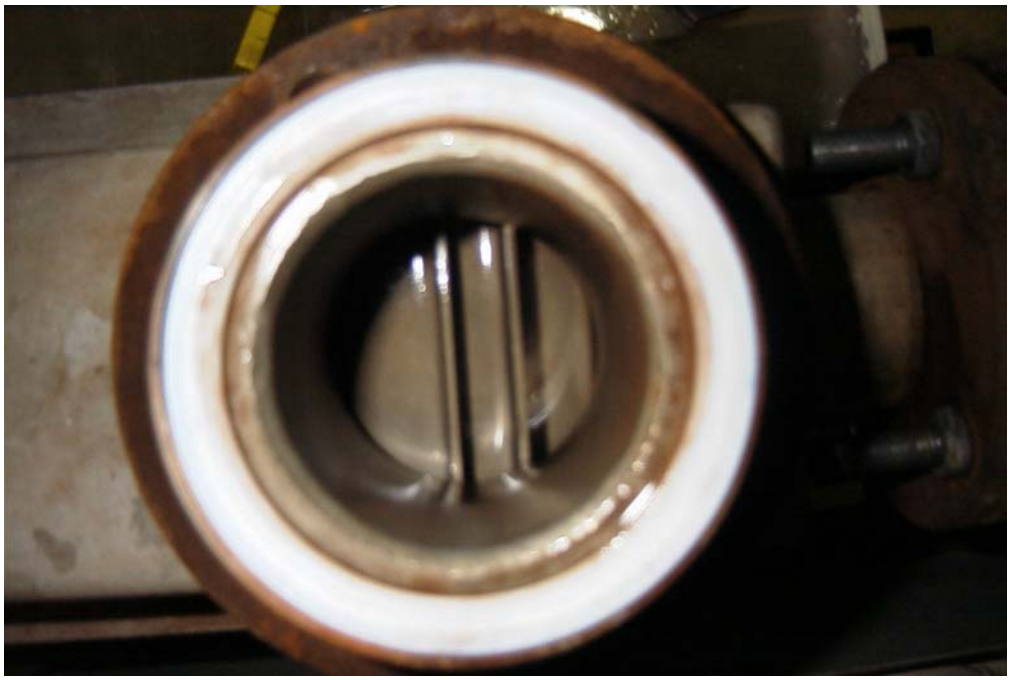
E-Horizontal Pipe Outlet

F-Reducer Inlet

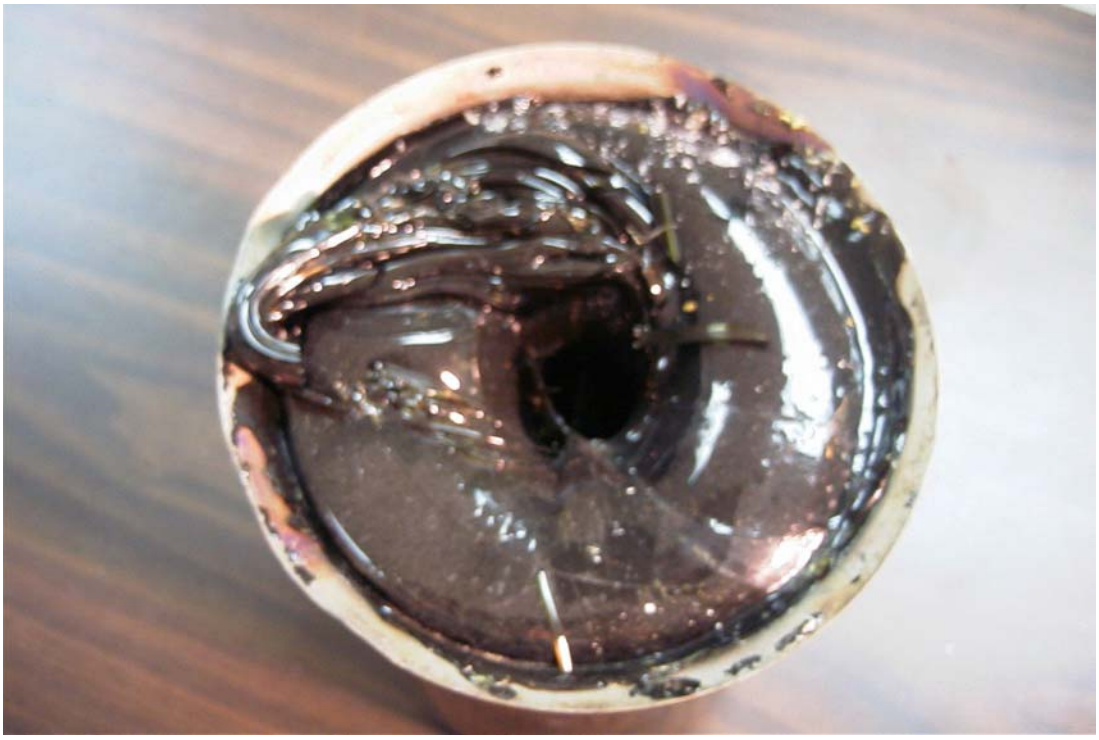
G-Reducer Outlet

H-EVS Inlet

H-EVS Inlet and Spray Nozzle



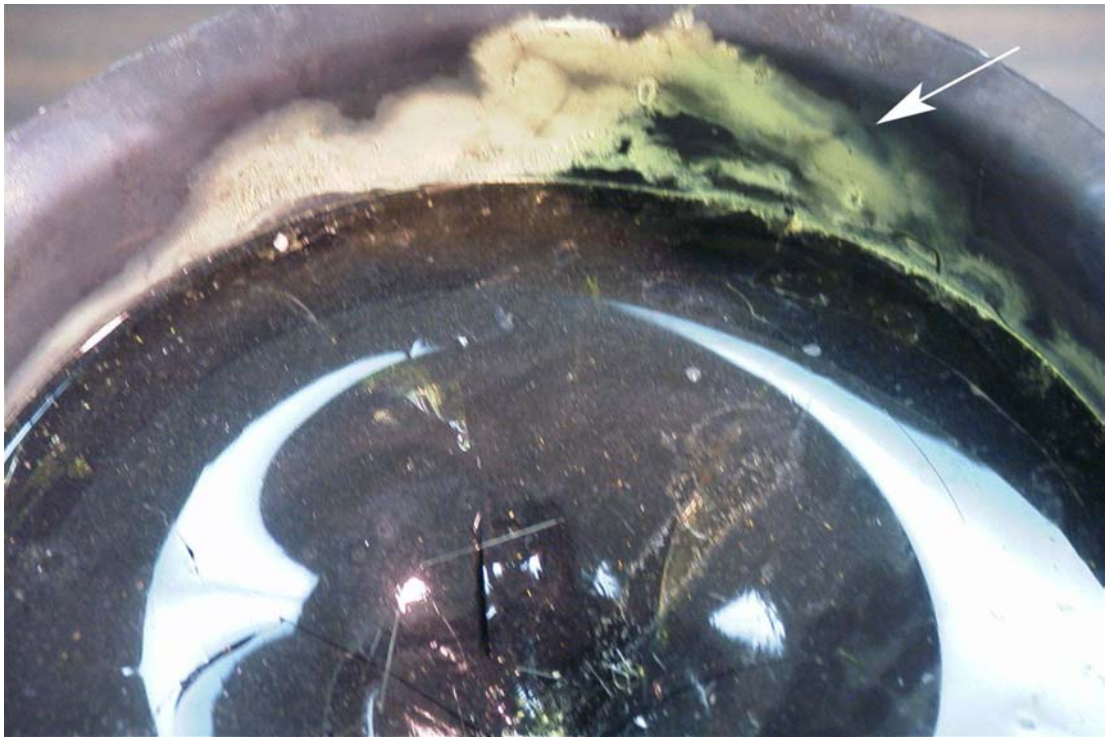
I-EVS Disengagement Box



Can 1

Can 1 Closeup

Can 2—Insoluble Salt Exclusions and/or Surface Deposits

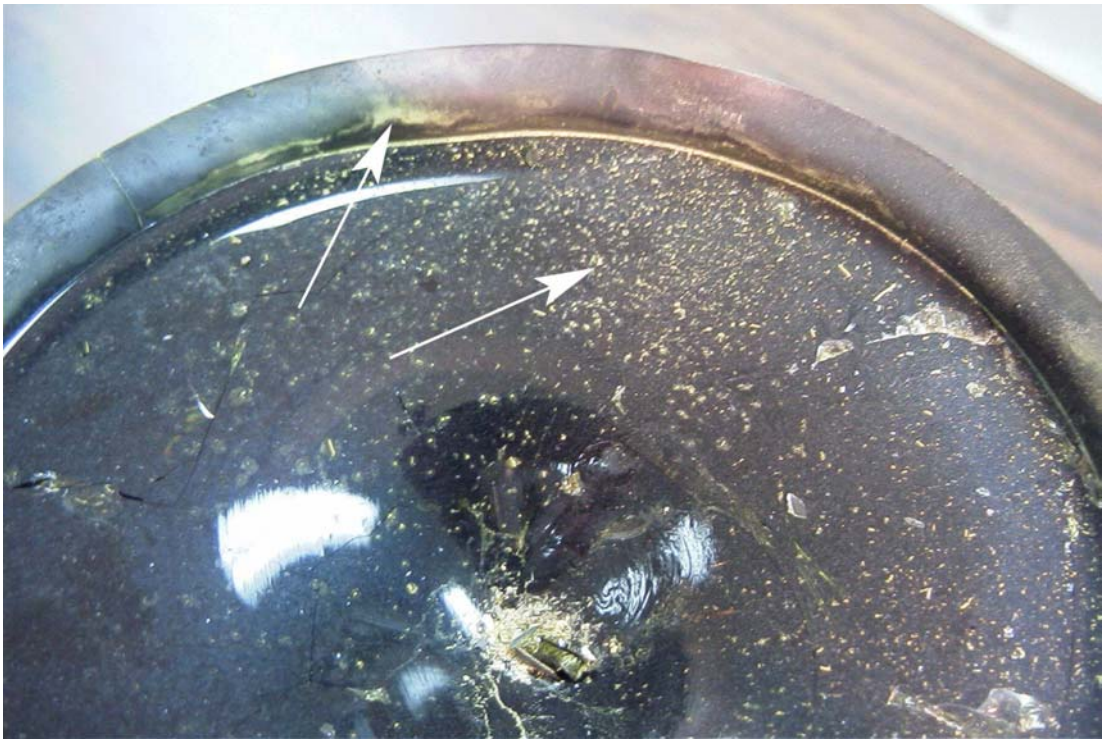


Can 2 Closeup—Insoluble Salt Exclusions and/or Surface Deposits

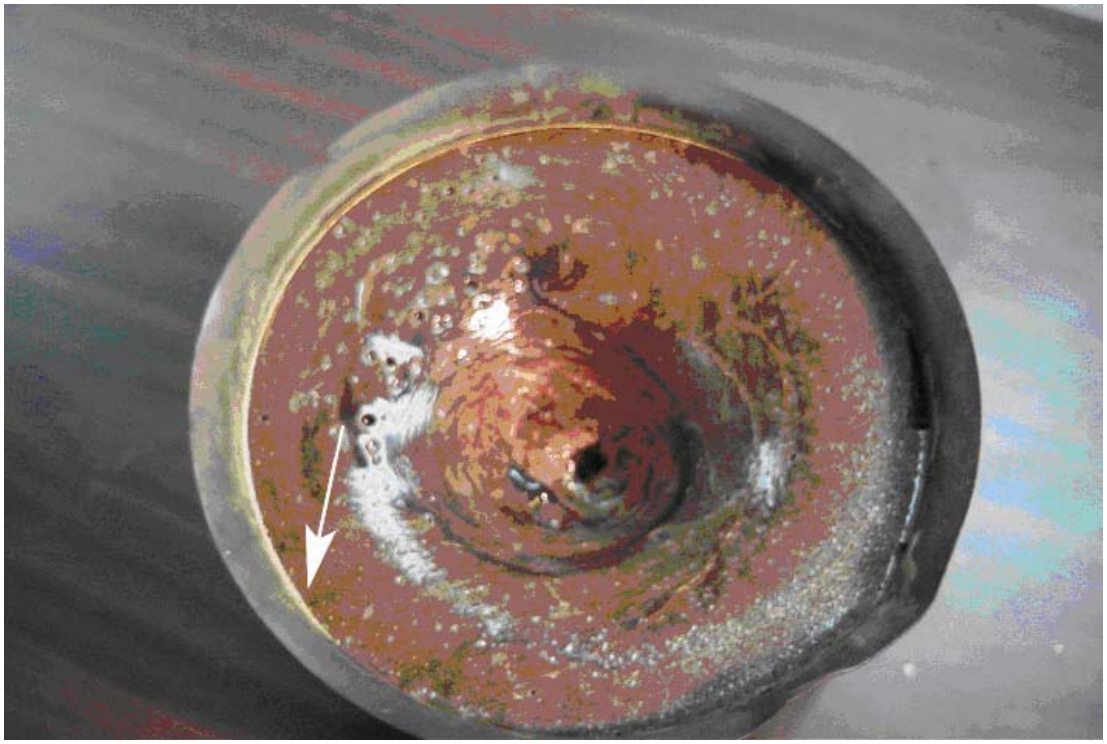
Can 3—Insoluble Salt Exclusions and/or Surface Deposits

Can 3 Closeup—Insoluble Salt Exclusions and/or Surface Deposits

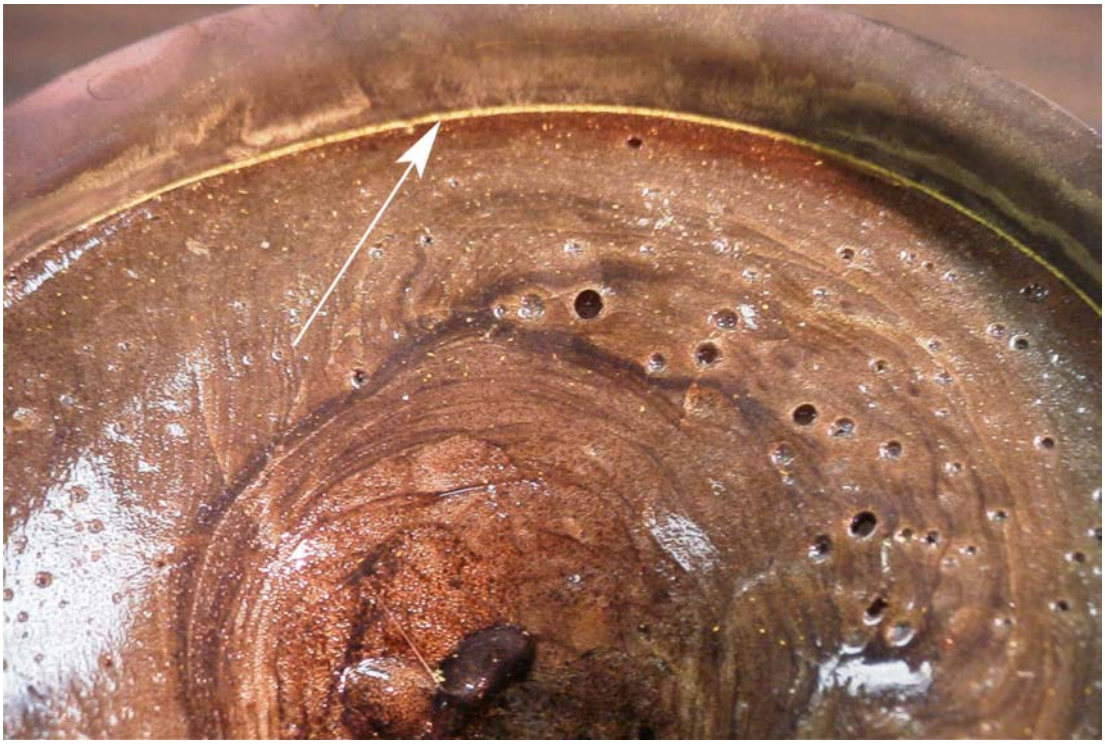
Can 4—Insoluble Salt Exclusions and/or Surface Deposits



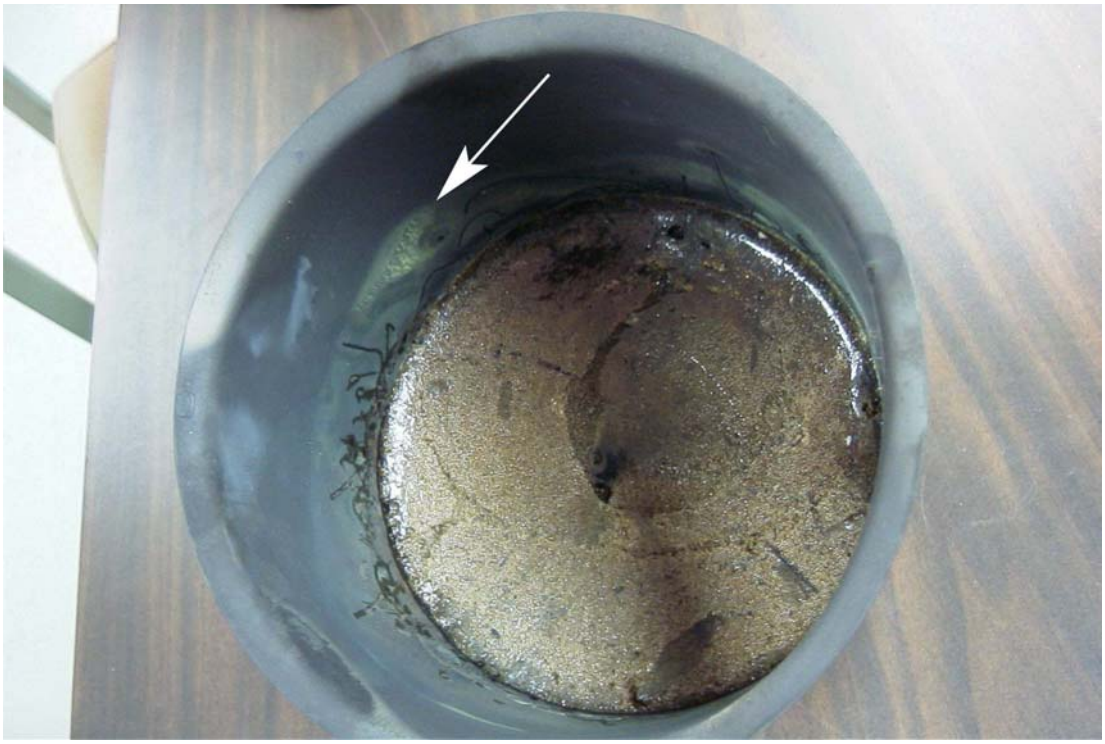
Can 4 Closeup—Insoluble Salt Exclusions and/or Surface Deposits



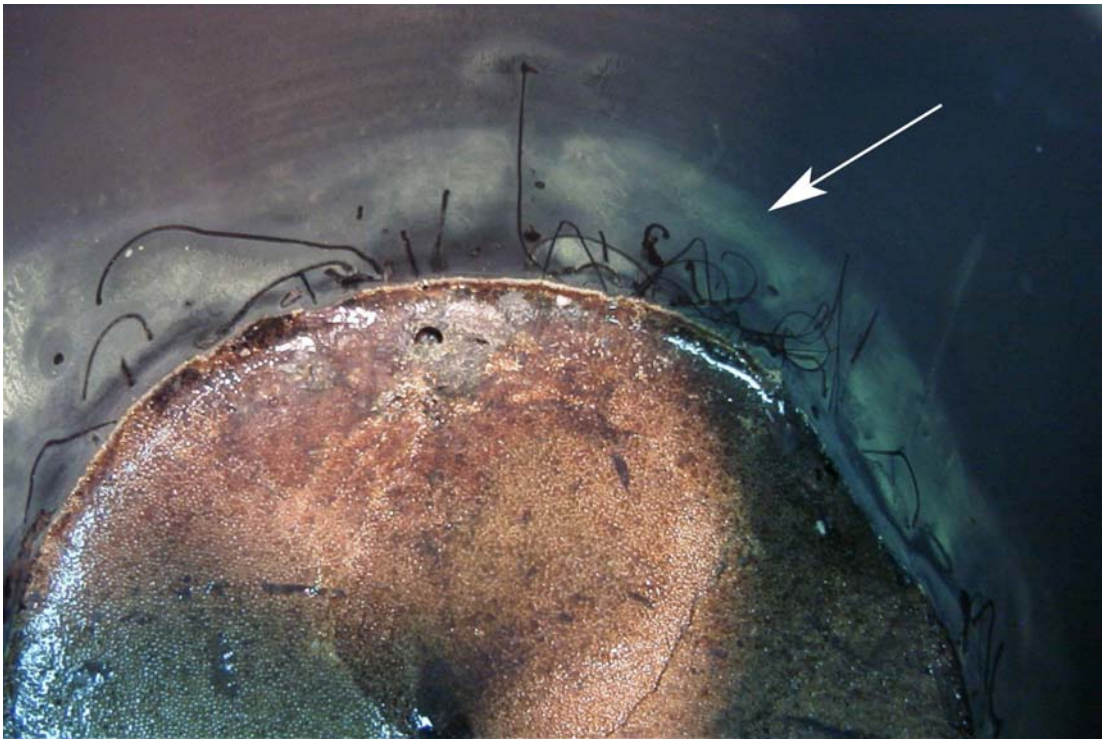
Can 5—Insoluble Salt Exclusions and/or Surface Deposits



Can 5 Closeup—Insoluble Salt Exclusions and/or Surface Deposits



Can 6—Insoluble Salt Exclusions and/or Surface Deposits



Can 6 Closeup—Insoluble Salt Exclusions and/or Surface Deposits

Appendix I

Tabular Off-Gas Sampling Data

Appendix I: Tabular Off-Gas Sampling Data

Table 1. Melter and off-gas Conditions at the Film Cooler Outlet During Manual Off-gas Sample Train Periods

			Film Cooler Data											
			Avg feedrate during feed period (a)							Outlet flowrate from He balance				
Date	Time	Manual sample train	kg/h	liter/h	Air flowrate, acfm @ 60 F	Air temp, C	Air pres-sure, psig	Air flow-rate, scfm (b, c)	Dilution factor (d)	wscfm (c)	acfm	dscfm (e)	Offgas temp., C (f)	Offgas velocity, f/s
7-Aug	1454	**Test Condition 1: SBW w/ 180 g/liter sucrose**												
		No manual sample trains during this feed condition.												
8-Aug	0247	**Test Condition 2: SBW w/ 170 g/liter sucrose**												
8-Aug	0955-1055	0050-1	2.73	1.90	7.0	35	11.2	8.7	2.4	15.1	23.7	13.8	143	18.1
8-Aug	1300-1350	0060-1	2.53	1.76	7.0	35	11.2	8.7	2.4	15.1	15.4	13.7	159	11.7
8-Aug	1522-1650	M8-1 (g)	2.32	1.61	7.0	35	11.2	8.7	2.4	15.2	15.4	13.8	161	11.8
8-Aug	1940	**Condition 3: SBW feed w/280 g/liter glycolic acid (400 gm GA solution), cold top drier, puffier, and higher**												
9-Aug	0040-0140	0050-2	2.52	1.76	7.0	35	11.2	8.7	3.1	13.0	13.2	11.8	129	10.1
9-Aug	0210-0310	0060-2	2.52	1.76	7.0	35	11.2	8.7	3.1	12.9	13.0	11.7	121	10.0
9-Aug	0335-0435	M8-2 (h)	2.55	1.77	7.0	35	11.2	8.7	2.7	14.0	14.2	11.9	126	10.9
9-Aug	0715	**Test Condition 4: Increased to 342 g/l gycolic acid (488 g/liter GA solution)**												
		No manual sample trains during this feed condition.												
9-Aug	1909	**Test Condition 5: Started SBW feed w/ 160 g/liter sucrose to obtain oxidizing glass**												
9-Aug	2310-0220	0060-3	2.66	1.85	7.0	35	11.2	8.7	3.2	12.8	12.9	12.2	129	9.9
10-Aug	633	**Test Condition 6: SBW feed w/o any reductant**												
10-Aug	1215-1315	0050-3	2.09	1.45	7.0	35	11.2	8.7	3.6	12.1	12.2	11.0	123	9.4
10-Aug	1350-1400	M8-3	2.09	1.45	7.0	35	11.2	8.7	3.5	12.2	12.4	11.0	127	9.5
10-Aug	1740-1905	0060-4	1.78	1.24	7.0	35	11.2	8.7	4.0	11.7	11.8	10.8	126	9.0
Averages (j)			2.38	1.65	7.0	35	11.2	8.7	3.0	13.4	14.4	12.2	134	11.0

a. Averages reported for distinct time periods when the gravimetric feedrate was determined.
b. This value is approximate because the true pressure in the rotameter is estimated, not directly measured.
c. Standard temperature is 0 degrees C, and standard pressure is 1 atmosphere.
d. Calculated from the wet standard flowrate and the measured moisture content.
e. Calculated from the dilution air flowrate and the total film cooler outlet flowrate based on the He balance.
f. Measured by a thermocouple in the duct downstream of the film cooler outlet sample location, and upstream of the EVS.
g. Run M8-1 required 2 PM filters; none of the other runs required any filter changes.
h. Lower PM emissions were observed during the M8-2 test, due perhaps to the reductant or to the cold top conditions.
i. SOx results from M8-3 are elevated because this test was shortened, allowing a much smaller sample volume.
j. Averages are straight linear averages, not time-weighted averages.

Table 2. Offgas composition at the film cooler outlet during manual offgas sample train periods.

			Film cooler offgas composition, dry basis unless otherwise indicated (a)																													
Date	Time	Manual sample train	N2, % (b)	O2, % (b)	CO2, % (b)	CO, ppm (b)	NO, % (b)	NO, % (c)	NO2, % (c)	N2O, % (b)	NOx, % (d)	NOx, % (e)	THC, ppm (f)	H2, ppm (b)	He, % (b)	H2O, % wet basis (g)	Gas composition total, %	SO2, ppm (h, i)	SO2, ppm (i, j)	SO3, ppm (i, j)	Total SOx as SO2 (i, k)	HCl, ppm (l)	Cl2, ppm (l)	Total Cl as HCl, ppm (l, m)	Total F, ppm (l)	HI, ppm (l, n)	I2, ppm (l, n)	Total I as HI, ppm (l, n)	Nitrate, ppm (l)	Nitrite, ppm (l)	Total NOx ions, ppm (l)	Total PM, mg/dscm (j, l)
7-Aug	1454	**Test Condition 1: SBW w/ 180 g/liter sucrose**																														
		No manual sample trains during this feed condition.																														
8-Aug	0247	**Test Condition 2: SBW w/ 170 g/liter sucrose**																														
8-Aug	0955-1055	0050-1	69.8	18.5	0.79	779	0.40	0.56	0.11	309	0.57	0.73	35	166	0.464	9.3	99.7	6	---	---	---	0.22	1.91	4.03	1.75	0.043	0.004	0.051	666	271	937	896
8-Aug	1300-1350	0060-1	68.1	18.1	0.76	768	0.40	0.61	0.13	311	0.59	0.79	30	154	0.470	10.5	98.8	7	---	---	8.52	---	---	---	---	---	---	---	---	---	---	---
8-Aug	1522-1650	M8-1 (o)	67.0	17.7	0.75	757	0.40	0.64	0.13	307	0.59	0.82	51	148	0.462	10.1	96.9	8	0.22	0.33	0.55	---	---	---	---	---	---	---	---	---	---	1,040
8-Aug	1940	**Test Condition 3: SBW feed w/280 g/liter glycolic acid (400 gm GA solution), cold top began to grow drier, puffier, and build in middle**																														
9-Aug	0040-0140	0050-2	69.4	18.5	0.81	165	0.37	0.54	0.10	286	0.53	0.70	49	294	0.548	10.1	100.2	4	---	---	---	< 0.06	1.51	3.02	5.44	0.031	0.006	0.043	597	147	744	204
9-Aug	0210-0310	0060-2	66.6	17.8	0.81	346	0.36	0.56	0.10	251	0.51	0.71	46	284	0.553	9.9	96.4	3	---	---	2.43	---	---	---	---	---	---	---	---	---	---	---
9-Aug	0335-0435	M8-2 (p)	65.1	17.4	0.76	318	0.35	0.59	0.07	306	0.48	0.72	41	277	0.540	17.8	102.4	4	< 0.28	0.28	< 0.55	---	---	---	---	---	---	---	---	---	---	183
9-Aug	0715	**Test Condition 4: Increased to 342 g/l gycolic acid (488 g/liter GA solution)**																														
		No manual sample trains during this feed condition.																														
9-Aug	1909	**Test Condition 5: Started SBW feed w/ 160 g/liter sucrose to obtain oxidizing glass**																														
9-Aug	2310-0220	0060-3	65.7	17.6	0.61	363	0.37			199	0.41		89	151	0.515	4.7	89.1	5	---	---	3.09	---	---	---	---	---	---	---	---	---	---	---
10-Aug	633	**Test Condition 6: SBW feed w/o any reductant**																														
10-Aug	1215-1315	0050-3	68.9	18.7	0.08	0	0.37	0.41	0.28	0	0.65	0.69	0	0	0.582	9.7	98.7	4	---	---	---	24.2	0.60	25.4	< 0.20	0.021	0.001	0.023	1,498	2	1,500	313
10-Aug	1350-1400	M8-3 (q)	67.6	18.5	0.12	0	0.45	0.69	0.43	0	0.88	1.12	0	0	0.587	11.1	99.1	5	< 1.62	0.81	< 2.43	---	---	---	---	---	---	---	---	---	---	291
10-Aug	1740-1905	0060-4	65.7	17.9	0.07	0	0.36	0.25	0.20	0	0.56	0.46	1	0	0.583	8.1	92.8	6	---	---	2.51	---	---	---	---	---	---	---	---	---	---	---
Averages (r)			67.4	18.1	0.56	350	0.38	0.54	0.17	197	0.58	0.75	34.2	147	0.530	10.1	97.5	5	< 0.70	0.47	< 2.87	8.15	1.34	10.8	2.46	0.03	0.004	0.04	920	140	1,060	488

a. When the continuous monitoring times were not simultaneous with the manual sample train times, then the average continuous monitoring data most representative of the conditions during the manual sample train times were used.
b. From GC analysis.
c. From NOx analyzer analysis.
d. Total of NO and N2O from the GC and NO2 from the NOx analyzer. The N2O value is doubled to put it on a NOx basis.
e. Total of N2O from the GC and NO and NO2 from the NOx analyzer. The N2O value is doubled to put it on a NOx basis.
f. From total hydrocarbon analyzer analysis.
g. From manual sample trains.
h. From the pulsed fluorescence SO2 monitor.
i. No SO2 or SO3 data is corrected; no blanks were analyzed, and results are near or under detection limits.
j. From the EPA Method 8 sample trains.
k. Total SOx from the Method 8 trains is the sum of the measured SO2 and SO3 values. Total SOx from the 60 trains is determined from the ICP analysis for total S in the sample train, and could include solid-phase sulfates.
l. From the EPA Method 0050 sample trains.
m. When nondetects are added to other nondetects, the total is a nondetect. When a nondetect is less than 10% of a detected level, it is considered to be 0 when they are added together.
n. The iodine analysis should be considered approximate, because of uncertainties in calibrating the ion chromatograph for iodine.
o. Run M8-1 required 2 PM filters; none of the other runs required any filter changes.
p. Lower PM emissions were observed during the M8-2 test, due perhaps to the reductant or to the cold top conditions.
q. SOx results from M8-3 are elevated because this test was shortened, allowing a much smaller sample volume.
r. Averages are straight linear averages, not time-weighted averages.

Table 3. Metals emission results from the metals sample trains (a).

	Metals concentrations, μg/dscm (at 68 F)				
	8-Aug-02	9-Aug-02	9-Aug-02	10-Aug-02	
	1300-1350	0210-0310	2310-0220	1740-1905	
	170 g/l sucose	280 g/l GA	160 g/l sucrose	no reductant	
	0060-1	0060-2	0060-3	0060-4	**Average**
Silver (Ag)	0.2	< 6	< 6	< 5	< **4**
Aluminum (Al)	3,213	2,343	2,081	13,004	**5,160**
Boron (B)	25,196	5,970	13,654	26,089	**17,727**
Barium (Ba)	6	29	24	25	**21**
Calcium (Ca)	7,987	2,147	4,398	15,914	**7,612**
Cadmium (Cd)	95	46	75	69	**71**
Chromium (Cr)	379	81	114	314	**222**
Cesium (Cs)	2,201	385	568	374	**882**
Copper (Cu)	46	14	15	29	**26**
Iron (Fe)	377	482	381	5,741	**1,745**
Gadolinium (Gd)	6	1	4	25	**9**
Mercury (Hg)	8,257	18,136	5,327	1,250	**8,243**
Potassium (K)	45,700	6,977	10,606	6,865	**17,537**
Lithium (Li)	23,424	3,321	8,099	11,482	**11,581**
Magnesium (Mg)	37	303	617	4,255	**1,303**
Manganese (Mn)	98	20	73	604	**199**
Molybdenum (Mo)	54	9	11	11	**21**
Sodium (Na)	251,078	39,740	71,332	56,075	**104,556**
Nickel (Ni)	40	17	21	71	**37**
Phosphorous (P)	320	54	134	469	**244**
Lead (Pb)	118	28	70	328	**136**
Ruthenium (Ru)	387	214	213	95	**227**
Sulfur (S)	11,355	3,241	4,117	3,347	**5,515**
Titanium (Ti)	0.3	0	0.6	14	**4**
Vanadium (V)	1,778	977	1,106	3,037	**1,725**
Zinc (Zn)	54	25	27	86	**48**
Zirconium (Zr)	94	31	16	2,804	**736**

a. Based on blank-corrrected train data.

Table 4. Partitioning of metals in solid phase, acid-soluble gas/vapor phase, and mercury as Hg or $HgCl_2$, based on mass captured in the metals train fractions (a).

	% of mass captured as PM (in front half, b)					% of mass captured in acidic impingers (c)					% of mass captured in $KMnO_4$ catch (d)				
	8-Aug 1300-1350	9-Aug 0210-0310	9-Aug 2310-0220	10-Aug 1740-1905		8-Aug 1300-1350	9-Aug 0210-0310	9-Aug 2310-0220	10-Aug 1740-1905		8-Aug 1300-1350	9-Aug 0210-0310	9-Aug 2310-0220	10-Aug 1740-1905	
	170 g/l sucrose	280 g/l GA	160 g/l sucrose	no reductant		170 g/l sucrose	280 g/l GA	160 g/l sucrose	no reductant		170 g/l sucrose	280 g/l GA	160 g/l sucrose	no reductant	
	0060-1	0060-2	0060-3	0060-4	**Average**	0060-1	0060-2	0060-3	0060-4	**Average**	0060-1	0060-2	0060-3	0060-4	Average
Silver (Ag)	> 0.0	0.0	0.0	0.0	**(e)**	< 100.0	0.0	0.0	0.0	**(e)**	---	---	---	---	---
Aluminum (Al)	100.0	100.0	100.0	96.6	**99.1**	0.0	0.0	0.0	3.4	**0.9**	---	---	---	---	---
Boron (B)	100.0	72.8	99.6	55.1	**81.9**	0.0	27.2	0.4	44.9	**18.1**	---	---	---	---	---
Barium (Ba)	90.5	99.4	98.3	99.7	**97.0**	9.5	0.6	1.7	0.3	**3.0**	---	---	---	---	---
Calcium (Ca)	99.1	100.0	99.7	94.6	**98.3**	0.9	0.0	0.3	5.4	**1.7**	---	---	---	---	---
Cadmium (Cd)	100.0	100.0	100.0	91.9	**98.0**	0.0	0.0	0.0	8.1	**2.0**	---	---	---	---	---
Chromium (Cr)	100.0	100.0	100.0	85.3	**96.3**	0.0	0.0	0.0	14.7	**3.7**	---	---	---	---	---
Cesium (Cs)	99.97	99.88	99.92	85.8	**96.4**	0.0	0.1	0.1	14.2	**3.6**	---	---	---	---	---
Copper (Cu)	99.9	100.0	100.0	91.1	**97.7**	0.1	0.0	0.0	8.9	**2.3**	---	---	---	---	---
Iron (Fe)	100.0	100.0	100.0	96.9	**99.2**	0.0	0.0	0.0	3.1	**0.8**	---	---	---	---	---
Gadolinium (Gd)	97.5	91.9	97.2	94.6	**95.3**	2.5	8.1	2.8	5.4	**4.7**	---	---	---	---	---
Mercury (Hg)	0.9	0.3	0.1	5.1	**1.6**	5.4	2.0	9.9	31.1	**12.1**	93.7	97.7	90.0	63.8	**86.3**
Potassium (K)	100.0	100.0	100.0	95.6	**98.9**	0.0	0.0	0.0	4.4	**1.1**	---	---	---	---	---
Lithium (Li)	100.0	100.0	100.0	91.4	**97.9**	0.0	0.0	0.0	8.6	**2.1**	---	---	---	---	---
Magnesium (Mg)	0.0	100.0	97.5	91.9	**72.4**	100.0	0.0	2.5	8.1	**27.6**	---	---	---	---	---
Manganese (Mn)	100.0	100.0	100.0	97.6	**99.4**	0.0	0.0	0.0	2.4	**0.6**	---	---	---	---	---
Molybdenum (Mo)	99.8	100.0	100.0	91.3	**97.8**	0.2	0.0	0.0	8.7	**2.2**	---	---	---	---	---
Sodium (Na)	100.0	100.0	100.0	97.4	**99.3**	0.0	0.0	0.0	2.6	**0.7**	---	---	---	---	---
Nickel (Ni)	100.0	100.0	100.0	96.5	**99.1**	0.0	0.0	0.0	3.5	**0.9**	---	---	---	---	---
Phosphorous (P)	100.0	100.0	100.0	100.0	**100.0**	0.0	0.0	0.0	0.0	**0.0**	---	---	---	---	---
Lead (Pb)	100.0	100.0	100.0	95.2	**98.8**	0.0	0.0	0.0	4.8	**1.2**	---	---	---	---	---
Ruthenium (Ru)	100.0	100.0	100.0	100.0	**100.0**	0.0	0.0	0.0	0.0	**0.0**	---	---	---	---	---
Sulfur (S)	100.0	100.0	100.0	100.0	**100.0**	0.0	0.0	0.0	0.0	**0.0**	---	---	---	---	---
Titanium (Ti)	0.0	0.0	0.0	96.2	**24.0**	100.0	0.0	100.0	3.8	**51.0**	---	---	---	---	---
Vanadium (V)	100.0	100.0	100.0	96.7	**99.2**	0.0	0.0	0.0	3.3	**0.8**	---	---	---	---	---
Zinc (Zn)	92.9	89.6	84.3	78.3	**86.3**	7.1	10.4	15.7	21.7	**13.7**	---	---	---	---	---
Zirconium (Zr)	100.0	100.0	100.0	99.97	**100.0**	0.0	0.0	0.0	0.03	**0.0**	---	---	---	---	---

a. Based on blank-corrrected train data.

b. This material is in the form either species that are relatively nonvolatile at 250F, or species that are more volatile but that are absorbed onto PM.

c. This material is in the form of either volatile species or solid species of very small particle size that passed through the train filter that has HEPA efficiency (99.97% removal for 0.3 micron PM).

d. This material consists of Hg species, most likely elemental Hg and $HgCl_2$, that passed through the train filter as a gas and also were not appreciably soluble in the acid impingers.

e. Silver was sporadically detected at levels near the detection limit, and partitioning for silver cannot be reliably determined.

Table 5. Partitioning of feed components to the offgas (a).

Feed component	Feedrate, gm/hr					Emission rates, gm/hr					% of feed component in offgas					Average decontamination factor (b)
	8-Aug 1300-1350	9-Aug 0210-0310	9-Aug 2310-0220	10-Aug 1740-1905		8-Aug 1300-1350	9-Aug 0210-0310	9-Aug 2310-0220	10-Aug 1740-1905		8-Aug 1300-1350	9-Aug 0210-0310	9-Aug 2310-0220	10-Aug 1740-1905		
	170 g/l sucrose (119 g/l feed)	400 g/l GA (280 g/l feed)	160 g/l sucrose (112 g/l feed)	no reductant	**Average**	170 g/l sucrose (119 g/l feed)	400 g/l GA (280 g/l feed)	160 g/l sucrose (112 g/l feed)	no reductant	**Average**	170 g/l sucrose (119 g/l feed)	400 g/l GA (280 g/l feed)	160 g/l sucrose (112 g/l feed)	no reductant	**Average**	
H2O (c)	1,730	1,847	1,800	1,335	**1,678**	2,064	2,293	812	1,580	**1,687**	119	124	45	118	**102**	---
C	87.9	155.9	87.2	0.0	**82.8**	117	100	77	10	**76**	133	64	88		**95**	---
Nitrates (as NO2) (d)	423	425	445	332	**406**	414	328		316	**353**	98	77		95	**90**	---
Cl (as HCl)	1.55	1.56	1.64	1.22	**1.49**	0.169	0.111		0.84	**0.374**	11	7		69	**29**	3.4
F (as HF)	0.105	0.105		0.082	**0.10**	0.040	0.110		0.0036	**0.051**	39	104		4	**49**	2.0
I (as HI)	0.0	0.0		0.0	**0.01**	0.008	0.006		0.0027	**0.005**	98	72		45	**71**	1.4
Aluminum (Al)	17.4	17.5	18.3	13.7	**16.7**	0.0528	0.0406	0.0397	0.2371	**0.0925**	0.3	0.2	0.2	1.7	**0.6**	161
Boron (B)	11.0	11.0	11.6	8.6	**10.6**	0.4142	0.1033	0.2604	0.4757	**0.3134**	3.8	0.9	2.2	5.5	**3.1**	32
Barium (Ba)	0.0077	0.0077	0.0081	0.0060	**0.0074**	0.0001	0.0005	0.0005	0.0005	**0.0004**	1.4	6.6	5.7	7.6	**5.3**	19
Calcium (Ca)	30.2	30.4	31.9	23.8	**29.0**	0.1313	0.0372	0.0839	0.2902	**0.1356**	0.4	0.1	0.3	1.2	**0.5**	196
Cadmium (Cd)	0.0830	0.0834	0.0875	0.0653	**0.0798**	0.0016	0.0008	0.0014	0.0013	**0.0013**	1.9	1.0	1.6	1.9	**1.6**	62
Chromium (Cr)	0.247	0.248	0.261	0.194	**0.238**	0.0062	0.0014	0.0022	0.0057	**0.0039**	2.5	0.6	0.8	2.9	**1.7**	58
Cesium (Cs)	0.326	0.328	0.344	0.256	**0.314**	0.0362	0.0067	0.0108	0.0068	**0.0151**	11.1	2.0	3.2	2.7	**4.7**	21
Copper (Cu)	0.0496	0.0498	0.0522	0.0390	**0.0476**	0.0008	0.0002	0.0003	0.0005	**0.0005**	1.5	0.5	0.5	1.4	**1.0**	101
Iron (Fe)	9.68	9.72	10.20	7.61	**9.30**	0.0062	0.0084	0.0073	0.1047	**0.0316**	0.1	0.1	0.1	1.4	**0.4**	250
Gadolinium (Gd)	No Gd in feed					0.0001	0.0000	0.0001	0.0005	**0.0002**	No Gd in feed					
Mercury (Hg)	0.188	0.189	0.198	0.148	**0.181**	0.1357	0.3139	0.1016	0.0228	**0.1435**	72	166	51	15	**76**	1.3
Potassium (K)	10.3	10.3	10.8	8.1	**9.86**	0.7512	0.1208	0.2023	0.1252	**0.2999**	7.3	1.2	1.9	1.6	**3.0**	34
Lithium (Li)	17.5	17.6	18.5	13.8	**16.8**	0.3850	0.0575	0.1545	0.2094	**0.2016**	2.2	0.3	0.8	1.5	**1.2**	82
Magnesium (Mg)	7.27	7.31	7.66	5.72	**6.99**	0.0006	0.0052	0.0118	0.0776	**0.0238**	0.008	0.072	0.153	1.357	**0.398**	251
Manganese (Mn)	0.496	0.499	0.523	0.390	**0.477**	0.0016	0.0003	0.0014	0.0110	**0.0036**	0.3	0.1	0.3	2.8	**0.87**	115
Molybdenum (Mo)	0.026	0.026	0.027	0.020	**0.025**	0.0009	0.0002	0.0002	0.0002	**0.0004**	3.5	0.6	0.8	1.0	**1.5**	68
Sodium (Na)	68.6	69.0	72.4	54.0	**66.0**	4.1271	0.6879	1.3603	1.0225	**1.7995**	6.0	1.0	1.9	1.9	**2.7**	37
Nickel (Ni)	0.570	0.573	0.601	0.448	**0.548**	0.0007	0.0003	0.0004	0.0013	**0.0007**	0.1	0.1	0.1	0.3	**0.13**	772
Phosphorous (P)	1.133	1.139	1.194	0.891	**1.09**	0.0053	0.0009	0.0026	0.0086	**0.0043**	0.5	0.1	0.2	1.0	**0.43**	233
Lead (Pb)	0.277	0.279	0.293	0.218	**0.267**	0.0019	0.0005	0.0013	0.0060	**0.0024**	0.7	0.2	0.5	2.7	**1.02**	98
Ruthenium (Ru)	0.129	0.130	0.136	0.101	**0.12**	0.0064	0.0037	0.0041	0.0017	**0.0040**	4.9	2.9	3.0	1.7	**3.1**	32
Sulfur (S) from 0060	2.80	2.81	2.95	2.20	**2.69**	0.1867	0.0561	0.0785	0.0610	**0.0956**	6.7	2.0	2.7	2.8	**3.5**	28
Titanium (Ti)	1.29	1.30	1.36	1.01	**1.24**	0.0000	0.0000	0.0000	0.0003	**0.0001**	0.0003	0.0000	0.0008	0.0254	**0.0066**	15,048
Vanadium (V)	18.2	18.3	19.2	14.3	**17.5**	0.0292	0.0169	0.0211	0.0554	**0.0307**	0.2	0.1	0.1	0.4	**0.19**	533
Zinc (Zn)	0.0601	0.0604	0.0633	0.0472	**0.0578**	0.0009	0.0004	0.0005	0.0016	**0.0008**	1.5	0.7	0.8	3.3	**1.6**	63
Zirconium (Zr)	12.18	12.24	12.84	9.58	**11.71**	0.0016	0.0005	0.0003	0.0511	**0.0134**	0.013	0.004	0.002	0.534	**0.138**	723
Total glass/PM	777	781	820	611	**747**	22.6	3.87		5.60	**10.7**	2.91	0.496		0.917	**1.44**	69

a. Based on blank-corrrected train data. Blanks indicate data that was not obtained.

b. Decontamination factor is defined as the mass of the element feed divided by the mass of the element in the offgas; which is the inverse of the % of the feed component found in the offgas.

c. The total input H2O includes (a) H2O from the feed, (b) water of combustion from the reductant, and (c) H2O in the dilution air. The feed H2O is based on LOD analysis of slurry feed. Water of combustion is calculated assuming all of the H in the reductant is reacted to form H2O, less the small amount of H that was converted to H2. H2O from dilution air, assuming 75% humidity and 80 degree F. ambient temperature, is 314 g/hr.

d. Emission rates are based on the higher NO values from the NOx analyzer, not the lower NO values from the GC.