



# **Test Summary Report INEEL Sodium-Bearing Waste Vitrification Demonstration RSM-01-1**

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May 2001



Prepared for the U.S. Department of Energy  
under Contract DE-AC06-76RL01830

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Pacific Northwest National Laboratory  
Richland, Washington 99352

## 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 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 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 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 includes 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 first of the melter flowsheet tests conducted in support of INEEL's vitrification facility design is discussed below.

The Pacific Northwest National Laboratory's (PNNL's) Research-Scale Melter (RSM) was used to conduct these initial melter-flowsheet evaluations. The RSM is a small (1/100-scale) 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 surrogate melter feed stream
- acceptability of various SBW to glass-forming additive ratios
- possible formation of a secondary sodium sulfate phase



- effectiveness of sugar as a glass oxidation-state modifier and nitrate reductant
- off-gas effluent emission characteristics of the melter
- quality and durability of the process' vitreous waste-form product.

During the 120 h of experimental testing, melter feeds with three different SBW waste loadings (30, 32, and 35 Wt%) were successfully processed. In addition, the impacts of varying reductant concentrations (135, 155, 166, 177, 184, and 197 g/L) upon processing rates, molten-salt accumulations, and glass oxidation state [Fe(II):Fe(III)] were evaluated. The effects of a 40% increase in SBW sulfate concentration upon melter performance and molten-salt-phase accumulations were also assessed during the final hours of melter testing.

The melting kinetics of all feeds processed, irrespective of their SBW waste loading, was found to be nominally limited to liquid boil-off rates. There is very little evidence, during the processing of the SBW melter-feed material, for the presence of a conventional solid cold cap composed of dried feed and calcine. Moreover, after boil off, residual melter-feed constituents were found to be almost immediately incorporated into the molten vitreous pool.

Feeding rates were generally lower at the beginning of RSM testing, which was due to operator conservatism and the lack of a significant salt phase that facilitates feed-to-glass conversion and the heat transfer between the glass pool and the cold cap. Increased sugar content that provided additional thermal-energy release also likely contributed to the enhanced processing rates observed during the latter stages of melter testing.

Melter glass production rates varied from 7.1 to 11.1 lbs/h/ft<sup>2</sup> for the various feed batches processed. These values comfortably exceed the reference (cold-lid) LFCM design production rate of 4 lbs/h/ft<sup>2</sup> that is often quoted and used for flowsheet and equipment sizing estimates. Indeed, this reference-normalized production rate is exceeded even when projections are based upon the overall average rate data (5.8 lbs/h/ft<sup>2</sup>) that are inclusive of all idle-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, 6.1 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. Recognizing that the SBW process flowsheet does not provide for pretreatment or concentration and that much of the power required in processing slurry feeds is consumed by boiling away water, the higher specific-energy requirements for vitrifying SBW is largely due to the higher-than-normal weight fraction of water in the melter feed stream.

Monitoring for molten salt accumulations was conducted throughout all phases of RSM testing. A salt phase was slow to develop during initial testing using 30% SBW feed batches. Moreover, the salt phase that did develop at baseline reductant loadings (135 g of sucrose/L of SBW) appeared stable and was confined to small non-contiguous pools. At 35%, SBW significant increases were observed in the

melter's molten-salt inventory. By increasing feed reductant loadings, the apparent increase in the melter salt accumulation rate was successfully counteracted. A contiguous molten-salt phase never developed on the RSM's glass pool, even though the sulfate concentrations in the final batch were 140% of baseline.

The increased reductant loading used to control molten-salt accumulations in the latter stages of testing also affected the oxidation state of the melter's glass product. Fe(II):Fe(III) ratio measurements were conducted colorimetrically on 32% and 35% SBW glasses in order to quickly assess the impact of changing reductant levels upon the chemical state of the glass product. These data suggest that SBW sugar concentrations as high as 154 g/L are adequate for maintaining acceptable glass oxidation conditions: Fe(II):Fe(total)<0.3. Slightly higher sugar concentrations might also be acceptable, but longer term testing at these higher concentrations and product testing of the more highly reduced glass would be necessary to optimize this parameter. However, all glass samples, even those that were highly reduced, were found, upon completion of toxicity characteristic leach procedure (TCLP) testing, to be compliant with all existing Resource Conservation and Recovery Act (RCRA) land-disposal limits.

The partitioning behavior of sulfur was assessed by conducting post-test analysis of all collected process streams. The results of this assessment indicate that 55% of the sulfur processed was incorporated within the melter's vitreous product, and 26% was condensed/collected as soluble sulfates by the off-gas system. It is believed that much of the remaining unaccounted for sulfur (~20%) is residing on the high-efficiency mist eliminator (HEME) fiber bed and/or in ejector venturi scrubber (EVS) insoluble material, as, historically, SO<sub>2</sub> generation rates in nonboosted, cold-lid melters are usually quite low. However, if all the accounted for sulfur is assumed to have been released to the environment as SO<sub>2</sub>, the average noncondensable off-gas concentration of this gas would have been <20 ppm. This is much less than the detection limits of the available on-line instrument that failed to detect this effluent during RSM testing.

For the surrogate SBW melter feed used during RSM testing, CO<sub>2</sub> and NO<sub>x</sub> (specifically NO) were the major non-condensable (~25°C) gases produced by the vitrification process. An analyzer's failed catalytic converter precluded NO<sub>2</sub> concentrations from being measured, although lower detection limits suggested that NO<sub>2</sub>:NO ratios as high as 1 might have been present. The combustible gases CO and H<sub>2</sub> were also detected, but at much lower concentrations: 0.15% and 0.025%, respectively. These concentrations are well below the lower flammability limits of these combustible gases, 4.65% for H<sub>2</sub> and 15.5% for CO.

The responses of the total hydrocarbon analyzer indicated that volatile organic compounds (VOCs) were present in melter exhaust throughout most periods of testing. Although the off-gas concentrations of these thermal byproducts of incomplete oxidation 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 VOC concentration spikes observed during RSM testing.

Melter condensed-phase effluents were also monitored during SBW melter testing. The melter's aerosol mass decontamination factors (DFs), as measured by non-isokinetic filter catches, were

determined for each of the distinct waste-loaded feeds processed. These melter aerosol mass DFs are fairly consistent and do not appear to have been significantly affected by the SBW waste-loading fraction. Their magnitudes (~50), moreover, are consistent with previous small-scale melter flowsheet tests that proved successful when tested on a larger scale.

Melter partitioning for individual feed components was also derived from off-gas sampling data. Due to low off-gas stream temperatures during SBW testing (~100°C), essentially feed constituents (excluding C, N, H<sub>2</sub>O, etc) were found to be primarily in a condensed state downstream of the film cooler. However, the overall reproducibility of all melter-feed component DFs is quite good, and the magnitudes of most DFs reported are reasonably close to expectations. On the other hand, the DFs recorded for the radiologically important semivolatiles Cs and Ru appear to be atypically low: 5 and 8, respectively.

A value of 10 to 20 is a much more typical DF value for Cs; however, unique chemical effects associated with SBW processing could be responsible for enhancing cesium loss rates. Similarly, Ru DF values also seem artificially depressed. Given the level of reductant used during all phases of testing, a DF value of 40 might normally be expected. However, analytical uncertainty is likely the cause of the latter DF anomaly, as detection limits are being approached in most samples. This also helps to explain the lack of agreement between analytical feed/glass data and corresponding Ru target values. Additional testing will, in time, resolve this apparent cesium loss rate anomaly, but higher levels of Ru will be needed in subsequent testing if reliable melter-performance values for this elements are to be obtained.

During SBW melter-flowsheet evaluation studies, 168-L of SBW simulated waste having a total mass of 210 kg were successfully processed by the RSM producing 22 L of glass having a total mass of 57 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<sub>2</sub>O and NO<sub>3</sub><sup>-</sup>) are nonvitrifiable, volatile species. On the other hand, since most of the hazardous and rad-waste SBW components can be incorporated and immobilized in the melter's vitreous product and tank waste volumes of SBW are a physical reality, volume reduction has important waste-disposal implications. During the current RSM test, an overall SBW waste volume-reduction factor of 7.6 was achieved.

## Acronyms

APEL	Applied Process Engineering Laboratory
DF	decontamination factor
DOE	U.S. Department of Energy
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
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
NWCF	New Waste Calcining Facility
PCT	Product Consistency Test
PLC	Programmable Logic Controller
PM	particulate matter
PNNL	Pacific Northwest National Laboratory
POG	process off-gas
RCRA	Resource Conservation and Recovery Act
RSM	Research-Scale Melter
SBW	sodium-bearing waste
SCR	silicon-controlled rectifier
TCLP	toxicity characteristic leach procedure

TFF	Tank Farm Facility
TOE	total operating efficiency
VOC	volatile organic compound
WCF	Waste Calcining Facility

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## 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) has 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 has 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  $3.8 \times 10^6$  L (1-million gal) of SBW that was not calcined. The SBW is an aqueous, highly acidic (1–3 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 tank farm facility 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 fiscal year 2000, the INEEL High Level Waste (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 is the preferred alternative, for the SBW, based upon the liquid-fed melter technology. This program includes 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 first of the melter flowsheet tests conducted in support of INEEL's vitrification facility design. Specifically, the Pacific Northwest National Laboratory's (PNNL's) Research-Scale Melter (RSM) was used to evaluate 1) the processing characteristics of the newly formulated SBW surrogate melter-feed stream, 2) the acceptability of various SBW to glass-

forming additive ratios, 3) the possible formation of a secondary sodium sulfate phase, 4) the effectiveness of sugar as a glass-oxidation-state modifier and a nitrate reductant, 5) the off-gas effluent-emission characteristics of the melter, and 6) the quality and durability of the process' vitreous waste-form product.

## 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 was established from a series of laboratory crucible tests involving SBW surrogate material and suitable glass-forming additives that were chosen to maximize product glass waste loading. Although these crucible tests suggested that the solubility of waste constituent  $\text{Na}_2\text{SO}_4$  would limit maximum achievable waste loadings, continuous feeding and dynamic processing conditions were needed to establish what this bounding condition would be. Indeed, all aspects of process-flowsheet evaluations require the use of such conditions.

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 initial RSM test series involving INEEL's SBW were established to:

1. Determine the feasibility of vitrifying surrogate SBW without pretreatment to produce a regulatory acceptable borosilicate glass waste form by adding suitable glass-forming chemicals and an oxidation state modifier (sucrose).
2. Characterize the product glass, melter off-gas, and particulate matter (PM, material volatilized or otherwise entrained in the off-gas). This characterization includes determination of (a) amounts and elemental compositions of all products, (b) the fate of certain feed components (such as radionuclide surrogates, heavy metals, glass formers, carbon, nitrates, halogens, and sulfur), and (c) leachability (based on toxicity characteristic leach procedure [TCLP] testing) and oxidation state (based on the  $\text{Fe}^{+2}/\text{Fe}^{\text{total}}$  ratio) of the product glass, and (d) properties of the off-gas and particulate matter.
3. Establish a maximum glass production rate based on stable operation at a maximum surrogate SBW loading and maximum feed rate.
4. Resolve whether melt rate or melter-processing conditions can be optimized by changing melter-operating conditions, such as reductant loading.
5. Determine surrogate SBW volume and mass reduction.
6. Evaluate power requirements as a function of surrogate SBW mass processed and product glass produced.

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

PNNL'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 SBW testing, and Figure 3.2 schematically illustrates the system components and their relationships to one another.

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 process effluent.

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.

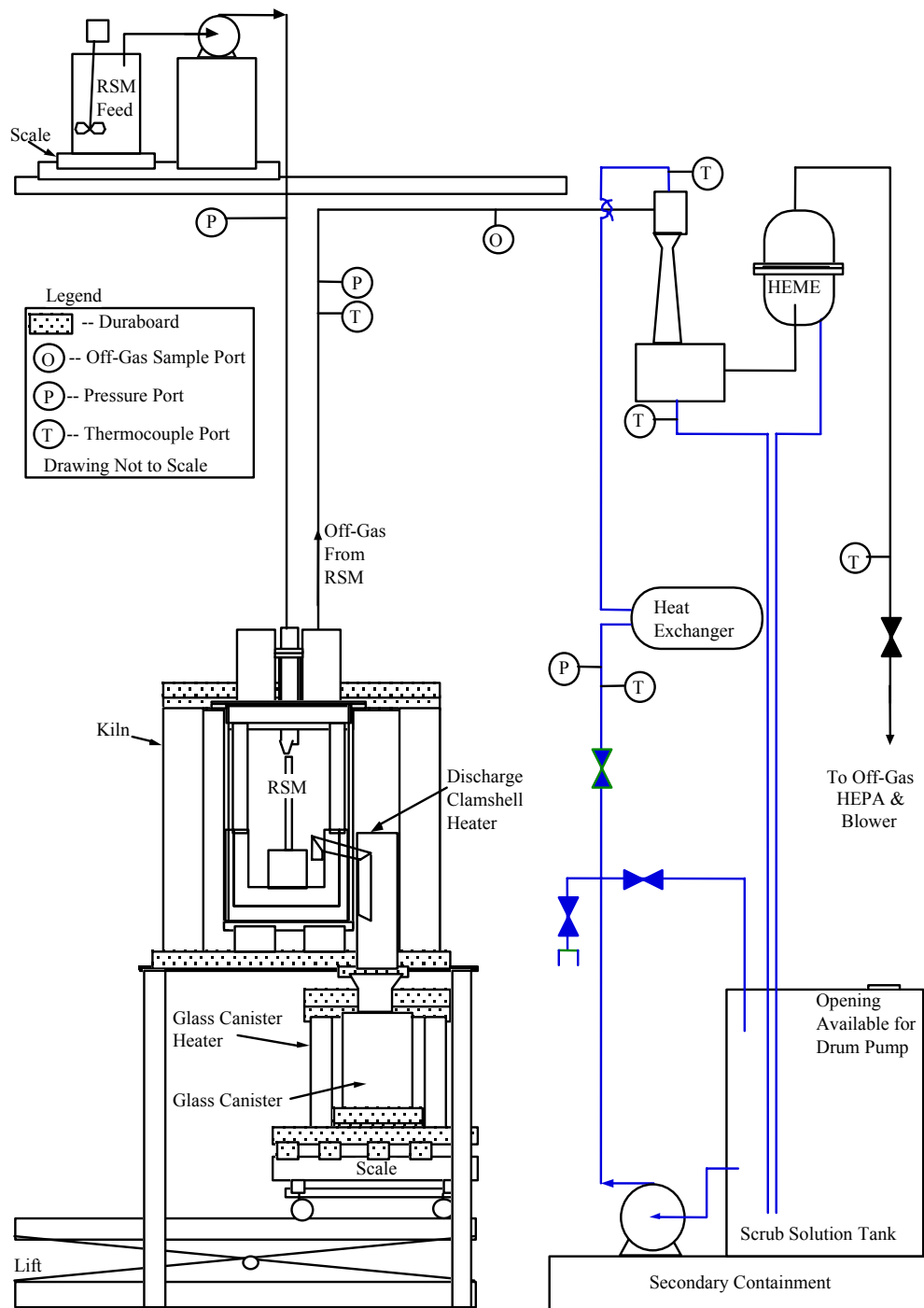
### 3.1 Melter

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 smaller kiln maintained between 700°C and 900°C to promote uniform canister filling. A platform scale that supports the smaller kiln allows glass-canister accumulations to be monitored as necessary. Two top-entering Inconel 690 electrodes (7.6-cm square x 0.64-cm thick [3-in. square x 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 3.3 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.

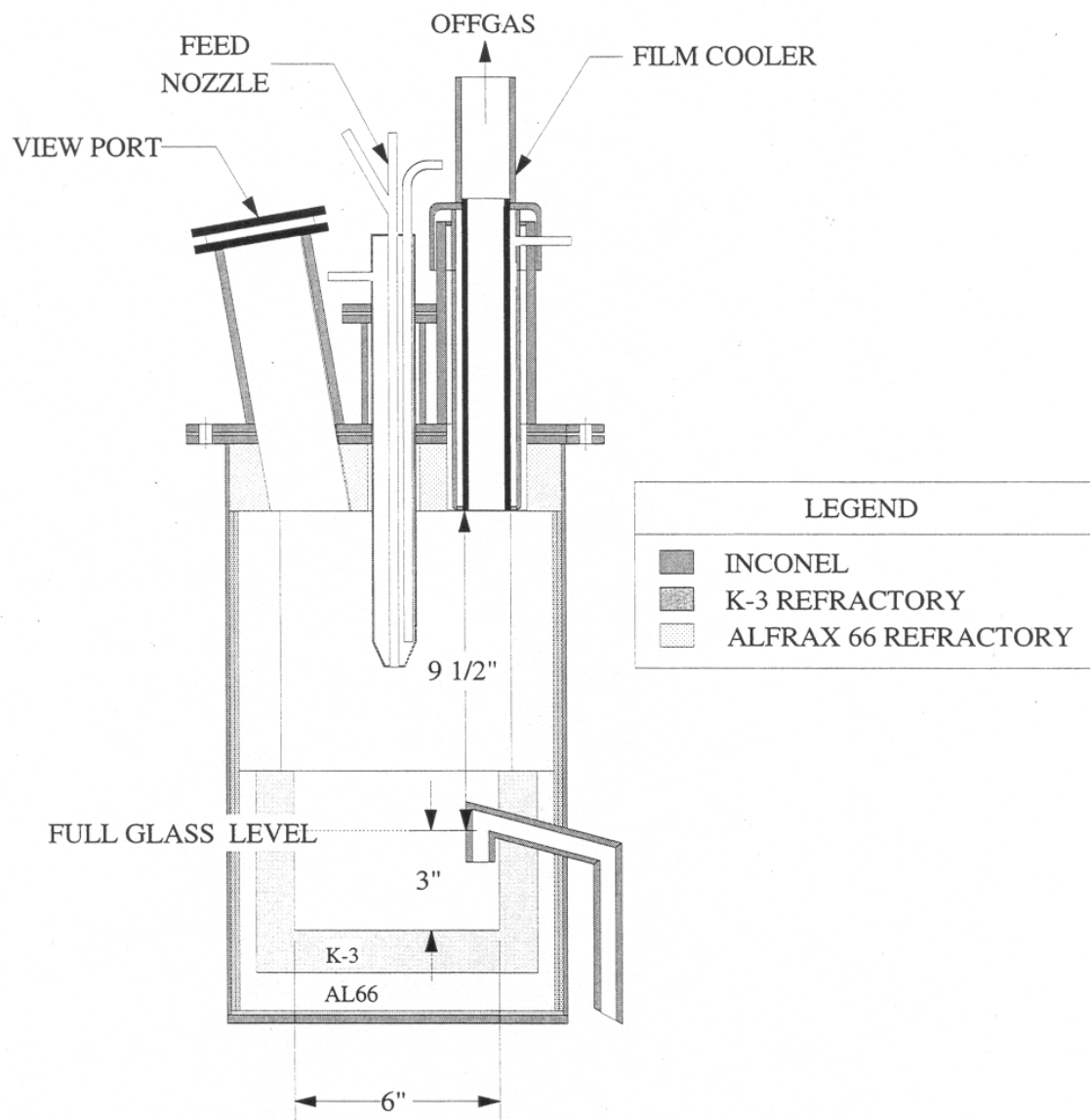


**Figure 3.1. Photograph of RSM Demonstration Unit**





**Figure 3.2. Research-Scale Melter Test Apparatus (Not shown is a high-efficiency particulate air [HEPA] filter that is installed downstream of the high efficiency mist eliminator [HEME])**



**Figure 3.3. Schematic View of the Research-Scale Melter**

**Table 3.1. RSM Dimensions and Operational Features**

Parameter	Value
Melter cavity diameter	15 cm
Melter cavity height	17 cm
Melter inside volume	4.5 L
Glass pool surface area	182 cm <sup>2</sup>
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 x 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 <sup>2</sup>

## 3.2 Feed System

The melter-feed system is located on the elevated steel platform adjacent to the melter (see Figure 3.2.). The tank used during current testing was a conical bottom tank with a maximum capacity of 5.7 L (15 gal). The melter feed tank, variable-speed agitator, peristaltic feed pump, and valve-control station are attached to a steel pallet that allows the tank to be lifted from the platform with the use of a forklift. The tank itself is located within a secondary containment that sits upon a load-cell platform scale that is monitored by the RSM's process control and data-acquisition system. The record of changes in feed-tank weight with time provides for a direct measure of the melter mass-feeding rate.

A peristaltic pump, located in a stainless steel enclosure designed to contain any elastomeric pump line slurry leaks, was used to extract feed from the bottom drain of the agitated feed 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 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.

### 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), heat exchanger, HEME, and high efficiency particulate arrestor (also known as 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 uses a caustic, high-pressure aqueous scrubbing liquor spray to contact the process exhaust stream in order to quench it and to remove steam, large-diameter aerosols, and some condensable and/or acid gases. A 94.6 L (25 gal) charge of fresh water adjusted to pH 10 with NaOH was put in the scrubbing liquor/condensate collection tank at the start of the test. Off-gas condensate and all collected solids were then allowed to accumulate within the condensate tank throughout the entire test, although the pH of this aqueous scrubbing media was always maintained between 9 and 12 by periodic NaOH additions. 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 treated with a certified HEPA filter to remove any significant remaining concentrations of aerosol matter from the process exhaust before it is released to the environment.

### 3.4 Off-Gas Sampling System

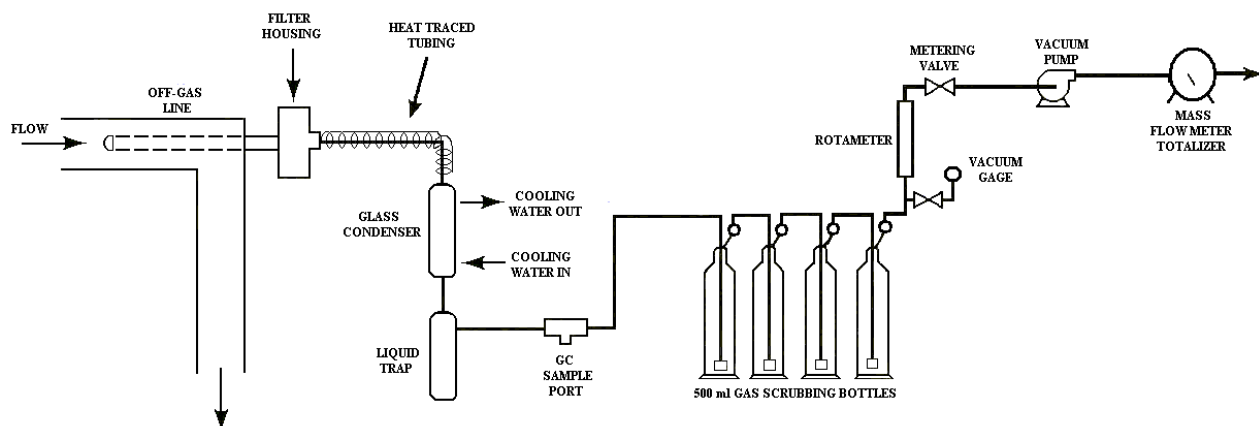
Process off-gas sampling during the current test was limited to characterizing the melter source. Melter effluents with significant room-temperature vapor pressures were nominally monitored continuously with gas analyzers, while discrete sampling campaigns were conducted to characterize condensed-phase effluents and condensable acid gases. The gas analyzers 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 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 diluted (~90%) with argon gas to reduce the dew point of the gas below the ambient operating temperature conditions of the gas analyzers.

**Table 3.2. RSM's Effluent Gas Analyzers**

Analyzer	Targeted Effluent Gases
Gas Chromatograph	H <sub>2</sub> , He, N <sub>2</sub> , CO, NO, O <sub>2</sub> , CO <sub>2</sub> , & N <sub>2</sub> O
Nitrogen Oxide	NO & NO <sub>2</sub>
Total Hydrocarbon	Volatile Hydrocarbons
Quadrupole Mass Spec	Survey Tool: HCl, H <sub>2</sub> S, NO <sub>2</sub> , SO <sub>2</sub> , etc.

Discrete sampling for process-generated aerosols (condensed phase matter) was also conducted using a multicomponent sampling system composed of a sampling probe with an appropriately sized sampling nozzle, an aerosol collection device, a condenser to remove condensable vapors, and a series arrangement of four aqueous chemical gas scrubbers used to collect reactive non-condensable gases. A schematic arrangement of this sampling system's components is illustrated in Figure 3.4. Since all four gas scrubbers were immersed in an ice-bath container, the first chemical scrubber also served as the system's condenser.



**Figure 3.4. Schematic Arrangement of Off-Gas Sampling System Components**

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. The sampling nozzle diameter at the end of the sampling probe 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 melter exhaust stream. The filtered gas stream was subsequently quenched (32°C) and chemically washed to remove reactive gases by a series arrangement of two H<sub>2</sub>SO<sub>4</sub> scrub solutions followed by a pair of NaOH solutions.

To establish isokinetic sampling conditions, the total off-gas flow rate has to be measured. During RSM testing, this was accomplished by injecting a helium tracer into the melter plenum at a fixed flow

rate (2 L/m) and measuring its resultant off-gas concentration with the online gas chromatograph discussed above. These POG flow rate data were also of fundamental importance in establishing effluent concentrations and emission rates.

### **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.

## 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 high quality, 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 sucrose reductant 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 solution prepared for the RSM testing was, 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. The procedure prepared and followed for the 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, Be, and Hg and all unstable radioactive components. Some of these radionuclides were, nevertheless, represented by their stable isotopes or chemical analog, 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, 200 L (53 gal) of SBW surrogate was prepared, and its resultant composition is compared to the defined SBW target in Table 4.3. The comparative data suggest that the prepared surrogate waste solution may have been about 11% more dilute than expected. Recognizing that the reported SBW analytical data were based on a single analysis, the make-up procedure and chemical certifications were reviewed to gain assurance that sufficient quantities of each material were indeed added to the SBW simulant mixture. 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 determined to be necessary.

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

INEEL Waste Definition <sup>(a)</sup>		Simulant	RSM
Element	Reagent	Con (M)	SBW?
Aluminum	Al(NO <sub>3</sub> ) <sub>3</sub> *9H <sub>2</sub> O	6.3E-01	Yes
Arsenic	As <sub>2</sub> O <sub>3</sub>	2.4E-4	---
Barium	Ba(NO <sub>3</sub> ) <sub>2</sub>	5.3E-05	Yes
Beryllium	BeF <sub>2</sub>	7.3E-6	---
Boron	H <sub>3</sub> BO <sub>3</sub>	1.2E-2	Yes
Cadmium	Cd(NO <sub>3</sub> ) <sub>2</sub> *4H <sub>2</sub> O	7.1E-4	Yes
Calcium	Ca(NO <sub>3</sub> ) <sub>2</sub> *4H <sub>2</sub> O	4.5E-2	Yes
Cerium	Ce(NO <sub>3</sub> ) <sub>3</sub> *6H <sub>2</sub> O	4.5E-5	Yes
Cesium	CsNO <sub>3</sub> (Simu only)	1.65E-3	Yes
Chromium	Cr(NO <sub>3</sub> ) <sub>3</sub> *5H <sub>2</sub> O	3.2E-3	Yes
Cobalt	Co(NO <sub>3</sub> ) <sub>2</sub> *6H <sub>2</sub> O	1.8E-5	Yes
Copper	Cu(NO <sub>3</sub> ) <sub>2</sub> *3H <sub>2</sub> O	6.6E-4	Yes
Gadolinium	Gd(NO <sub>3</sub> ) <sub>3</sub> *5H <sub>2</sub> O	1.7E-4	Yes
Iron	Fe(NO <sub>3</sub> ) <sub>3</sub> *9H <sub>2</sub> O	2.1E-2	Yes
Lead	Pb(NO <sub>3</sub> ) <sub>2</sub>	1.2E-3	Yes
Lithium	LiNO <sub>3</sub>	3.2E-4	Yes
Magnesium	Mg(NO <sub>3</sub> ) <sub>2</sub> *6H <sub>2</sub> O	1.1E-2	Yes
Manganese	Mn(NO <sub>3</sub> ) <sub>2</sub>	1.3E-2	Yes
Mercury	Hg(NO <sub>3</sub> ) <sub>2</sub> *H <sub>2</sub> O	1.9E-3	---
Molybdenum	Mo in HNO <sub>3</sub>	1.8E-4	Yes
Nickel	Ni(NO <sub>3</sub> ) <sub>2</sub> *6H <sub>2</sub> O	1.4 <sup>E</sup> -3	Yes
Potassium	KNO <sub>3</sub>	1.9E-1	Yes
Ruthenium	RuCl <sub>3</sub>	1.2E-4	Yes
Sodium	NaNO <sub>3</sub>	1.9E+0	Yes
Strontium	Sr(NO <sub>3</sub> ) <sub>2</sub>	1.1E-4	Yes
Titanium	TiCl <sub>4</sub>	5.5E-5	Yes
Uranium	UO <sub>2</sub> (NO <sub>3</sub> ) <sub>2</sub> *6H <sub>2</sub> O	3.2E-4	---
Zinc	Zn(NO <sub>3</sub> ) <sub>2</sub> *6H <sub>2</sub> O	9.9E-4	Yes
Zirconium	ZrF <sub>4</sub>	6.0E-5	Yes
Chloride	HCl	2.8E-2	Yes
Fluoride	HF	3.4E-2	Yes
Iodide	KI	1.3E-4	Yes
Nitrate	HNO <sub>3</sub>	8.6E-1	Yes
Phosphate	H <sub>3</sub> PO <sub>4</sub>	1.3E-2	Yes
Sulfate	H <sub>2</sub> SO <sub>4</sub>	5.1E-2	Yes

(a) Unstable elements of insignificant mass not included



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 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 also suggest a slightly diluted SBW surrogate, but only by ~6%.

**Table 4.2. Stable Isotope Chemical Analog Surrogates**

<b>SBW Radio-Nuclide</b>	<b>Substitute/Chemical-Surrogate</b>
$^3\text{H}$	Stable Isotope
$^{60}\text{Co}$	Stable Isotope
$^{90}\text{Sr}$	Stable Isotope
$^{99}\text{Tc}$	---
$^{129}\text{I}$	Stable Isotope
$^{134}\text{Cs}$	Stable Isotope
$^{137}\text{Cs}$	Stable Isotope
$^{154}\text{Eu}$	Cerium
$^{234}\text{U}$	---
$^{235}\text{U}$	---
$^{236}\text{U}$	---
$^{238}\text{U}$	---
$^{237}\text{Np}$	---
$^{238}\text{Pu}$	---
$^{239}\text{Pu}$	---
$^{241}\text{Am}$	---

**Table 4.3. SBW and PNNL Surrogate Compositions**

Element	SBW Conc (ppm)*		Dev (%)
	Target	Surrogate	
Al	16900	13100	-22.7
B	126	120	-4.4
Ba	7	7	-0.3
Ca	1820	1800	-0.8
Cd	80	76	-5.6
Ce	6	---	---
Co	1	---	---
Cr	165	146	-11.6
Cs	216	---	---
Cu	42	35	-17.5
Fe	1140	1000	-12.7
Gd	26	---	---
K	7240	6550	-9.6
Li	2	2	-25.8
Mg	276	240	-13.1
Mn	731	610	-16.6
Mo	18	---	---
Na	44600	40800	-8.6
Ni	82	---	---
P	400	335	-16.5
Pb	255	213	-16.6
Ru	12	---	---
Sr	10	9	-5.6
Ti	3	---	---
Zn	65	61	-6.5
Zr	5	---	---
I <sup>-</sup>	16	---	---
Cl <sup>-</sup>	1010	---	---
SO <sub>4</sub> <sup>=</sup>	4900	4115	-16.0
NO <sub>3</sub> <sup>-</sup>	317000	---	---
F <sup>-</sup>	870	---	---

\* ---, element not detected/measured.

**Table 4.4. SBW Surrogate Physical Characteristics**

<b>Trial</b>	<b>Sp. Grav.</b>	<b>LOI (%)</b>	<b>Oxides (g/L)</b>
SBW -1	1.25	91.3	109
SBW-2	1.25	91.5	106
Avg	1.25	91.4	107
<b>SBW Target = 114</b>			

## 4.2 Target Glass Composition

The targeted vitreous product composition for the current RSM 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 product glass waste loading. Since these crucible tests suggested that the glass solubility of waste constituent  $\text{Na}_2\text{SO}_4$  would limit maximum achievable waste loadings, a highly durable glass with the highest possible sulfur solubility was formulated for melter testing. The target composition of this glass, designated SBW-9, is summarized in Table 4.5, where only major constituents are identified.

**Table 4.5. Target Glass Composition**

<b>Oxide</b>	<b>Wt%</b>
$\text{B}_2\text{O}_3$	10.53
$\text{CaO}$	4.13
$\text{Fe}_2\text{O}_3$	7.37
$\text{Li}_2\text{O}$	3.47
$\text{Na}_2\text{O}$	15.64
$\text{SiO}_2$	45.16
Balance	13.70

## 4.3 Glass Former and Chemical Additives

To produce the target glass composition for any specified waste-loading value, calculated quantities of glass-forming chemicals were blended with a prespecified batch volume of SBW 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 that were tested appear in Appendix C along with the other test-data logging sheets.

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

Frit Oxide	Oxide		Glass Former		Mass Ratio (g-chem /g-frit)
	Wt%	MW	Chemical	MW	
B <sub>2</sub> O <sub>3</sub>	15	69.6	H <sub>3</sub> BO <sub>3</sub>	61.8	0.27
Fe <sub>2</sub> O <sub>3</sub>	10	159.7	Fe <sub>2</sub> O <sub>3</sub>	160	0.10
Li <sub>2</sub> O	5	29.9	LiOH*H <sub>2</sub> O	41.9	0.14
SiO <sub>2</sub>	64	60.1	SiO <sub>2</sub>	60.1	0.65
CaO	6	56.1	Ca(OH) <sub>2</sub>	74.1	0.066

As mentioned above, an organic reductant, sucrose, was added to all the feed batches to control the oxidation states of multivalent elements in the product glass. 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 should be maintained under 0.3. The maximum concentration for sucrose, based on crucible tests, was recommended to be 135 g of sugar per liter of surrogate SBW. This maximum value is about 64% of the amount needed to stoichiometrically reduce all feed-stream nitrates to N<sub>2</sub>. 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 concentration upon processing rates, nitrogen oxide emissions, and glass-oxidation state.

#### 4.4 Melter Feed Characteristics

Melter-feed samples were collected from each feed batch prepared during the SBW melter 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 waste loadings. Also presented in this table, for comparison purposes, are the corresponding total-oxide target values derived from the SBW and the target-glass composition data previously discussed as well as the batch chemical masses.

**Table 4.7. Physical Properties of Surrogate SBW Melter Feeds**

SBW Wt%	Sp. Grav.	Weight Loss %		Total Oxide/Liter (g)		
		LOD	LOI	LOI	Batch Wts	Target
30	1.37	62.4	42.0	299	320	326
32	1.34	64.2	42.7	274	297	306
35	1.34	62.6	60.8	Reactive	272	280

Feed total-oxide values derived from laboratory LOI tests appear to be biased low relative to targeted values, but are all within 10% of expectations. The above data suggest that the glass-former additives create a ~15% SBW volume change. A larger feed-to-waste volume ratio could easily explain the observed bias. The total average oxide loading of the feed calculated from all the feed processed and the glass produced throughout the duration of RSM testing, 292 g/L, also agrees well with all measured and derived values listed in Table 4.7.

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-composition values. All of the major feed component concentrations were reasonably consistent throughout the nominal 4 days of testing. There were no trends or large variations of importance, and most 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 the values of those that could appear to be overestimated. However, 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.

Apart from the trace constituents 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.

## **4.5 Product Glass Characteristics**

Glass-grab samples were collected from each RSM pour, which nominally occurred every 2 h. Fe(II):Fe(III) ratio measurements were conducted throughout the later part of the 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 feeds of differing waste loadings were compositionally analyzed and subjected to toxicity characteristic leach procedure (TCLP) testing. The results of these measurements will now be discussed.

### **4.5.1 Oxidation State Results**

As previously discussed (see Section 4.3), the SBW sucrose concentration was an experimental parameter during RSM testing. The purpose of varying the SBW reductant was to establish an optimum concentration that would maximize nitrate destruction while maintaining acceptable glass-oxidation-state conditions. To accomplish this, oxidation states were promptly measured for glass samples taken before and after melter-feed reductant levels were altered.

**Table 4.8. Melter Feed Composition for Batch Samples RSM-01-1(-NM)**

Oxide	Wt%													
	30% SBW				32% SBW				35% SBW					
	-08	-13	Avg	Trgt	-29	-35	Avg	Trgt	-49	-66	-75	-93A	Ave*	Trgt
Al <sub>2</sub> O <sub>3</sub>	8.120	6.680	7.400	8.370	6.820	7.270	7.040	8.930	7.850	7.990	8.750	7.970	8.140	9.770
B <sub>2</sub> O <sub>3</sub>	13.500	14.800	14.200	10.600	10.300	11.100	10.700	10.300	9.730	9.370	9.810	9.570	9.620	9.870
BaO	0.004	0.004	0.004	0.002	0.005	0.005	0.005	0.002	0.004	0.005	0.065	0.005	0.020	0.002
CaO	3.640	2.910	3.270	4.160	3.330	3.000	3.170	4.110	3.220	3.200	3.060	3.240	3.180	4.020
CdO	0.027	0.022	0.025	0.024	0.024	0.024	0.024	0.026	0.025	0.018	0.026	0.016	0.021	0.028
Ce <sub>2</sub> O <sub>3</sub>	---	---	---	0.002	---	---	---	0.002	---	---	---	---	---	0.002
Co <sub>2</sub> O <sub>3</sub>	0.016	0.015	0.016	0.0004	0.015	0.016	0.016	0.0004	0.016	0.015	0.017	0.016	0.016	0.0005
Cr <sub>2</sub> O <sub>3</sub>	0.072	0.058	0.065	0.063	0.067	0.065	0.066	0.067	0.069	0.069	0.078	0.070	0.071	0.073
Cs <sub>2</sub> O	0.046	0.049	0.048	0.060	0.052	0.047	0.049	0.064	0.044	0.050	0.040	0.041	0.044	0.070
CuO	---	---	---	0.014	---	---	---	0.015	0.005	0.004	0.005	0.003	0.004	0.016
Fe <sub>2</sub> O <sub>3</sub>	8.730	7.140	7.930	7.430	7.440	7.990	7.720	7.260	7.180	6.630	7.360	6.610	6.950	7.000
Gd <sub>2</sub> O <sub>3</sub>	0.022	0.016	0.019	0.008	0.020	0.018	0.019	0.008	0.018	0.025	0.025	0.017	0.021	0.009
K <sub>2</sub> O	2.640	2.220	2.430	2.280	2.250	2.380	2.310	2.430	2.510	2.560	3.200	2.700	2.750	2.660
Li <sub>2</sub> O	4.510	3.870	4.190	3.500	3.470	3.720	3.590	3.400	3.690	3.690	3.770	3.670	3.700	3.250
MgO	0.175	0.144	0.160	0.120	0.154	0.159	0.156	0.128	0.166	0.173	0.187	0.166	0.173	0.140
MnO	0.309	0.250	0.280	0.247	0.276	0.284	0.280	0.263	0.308	0.308	0.354	0.312	0.320	0.288
MoO <sub>3</sub>	0.012	0.010	0.011	0.007	0.012	---	0.006	0.007	0.012	0.014	0.014	0.012	0.013	0.008
Na <sub>2</sub> O	18.100	16.200	17.100	15.700	18.600	16.500	17.500	16.800	19.300	19.900	14.900	18.900	18.200	18.400
NiO	---	---	---	0.027	---	---	---	0.029	---	---	---	---	---	0.032
P <sub>2</sub> O <sub>5</sub>	0.063	0.062	0.063	0.240	0.062	0.041	0.051	0.256	0.081	0.105	0.095	0.094	0.094	0.280
PbO	0.091	0.074	0.083	0.072	0.080	0.080	0.080	0.077	0.085	0.084	0.127	0.085	0.095	0.084
RuO <sub>2</sub>	0.010	0.008	0.009	0.004	0.012	0.010	0.011	0.004	0.013	0.015	0.013	0.012	0.013	0.005
SO <sub>3</sub>	1.290	1.050	1.170	1.070	1.250	1.170	1.210	1.140	1.310	1.320	1.460	1.830	1.360	1.250
SiO <sub>2</sub>	38.600	44.400	41.500	45.500	45.800	46.100	45.900	44.200	44.300	44.400	46.600	44.400	44.900	42.300
SrO	0.007	0.007	0.007	0.003	0.008	0.007	0.007	0.003	0.007	0.007	0.008	0.007	0.007	0.004
TiO <sub>2</sub>	0.016	0.017	0.017	0.001	0.018	0.018	0.018	0.001	0.019	0.017	0.021	0.018	0.019	0.001
ZnO	0.022	0.018	0.020	0.021	0.020	0.020	0.020	0.023	0.022	0.022	0.025	0.022	0.023	0.025
ZrO <sub>2</sub>	---	---	---	0.002	---	---	---	0.002	---	---	---	0.243	0.061	0.002

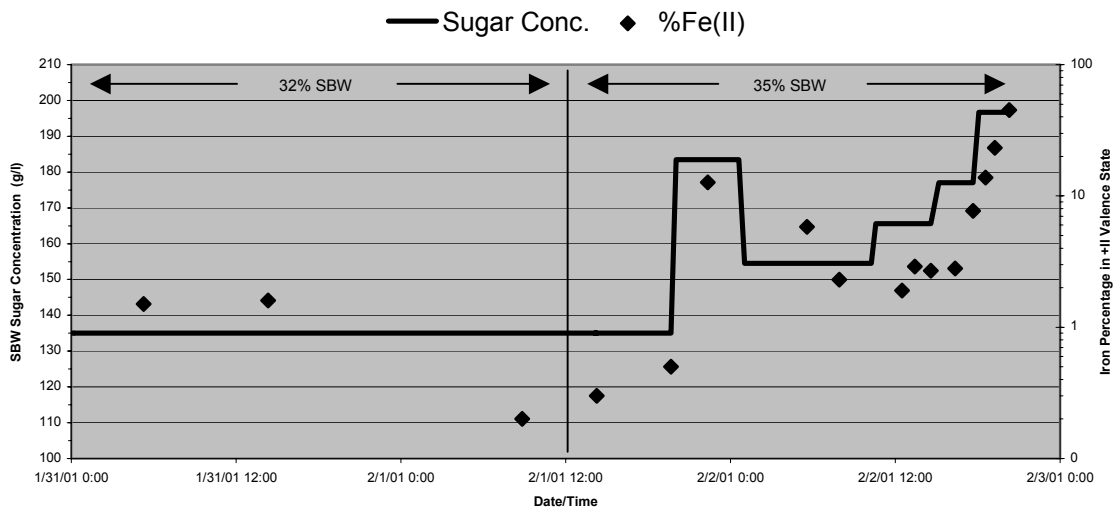
\* RSM-01-1-93A not included in average, as batch contained 140% SBW sulfur content!

A chemical method for measuring a Fe(II) complex colorimetrically at a wavelength of 515 nm was used to establish the vitreous iron fraction in the +II valence state. This is accomplished by dissolving a powdered glass sample in H<sub>2</sub>SO<sub>4</sub> and HF, buffering the resultant solution with sodium acetate/boric acid solution containing o-phenanthroline complexing agent, and conducting an Fe(II)-specific absorbance measurement at 515 nm. Ascorbic acid is subsequently used to reduce all remaining iron in the dissolved sample to the Fe(II) state, which allows the total Fe to be measured by a subsequent absorption measurement. These results allow the fraction of iron in the +II valence state in the glass sample to be directly determined. The detailed procedure used for these measurements is reproduced in Appendix D.

The glass oxidation state results, which were only obtained for 32% and 35% SBW glasses using this colorimetric procedure, are summarized in Table 4.9 along with corresponding SBW sugar loading values. The historical SBW sugar-loading and glass-oxidation-state results for the RSM test are also displayed graphically in Figure 4.1. These data suggest that SBW sugar concentrations as high as 154 g/L are adequate for maintaining acceptable glass-oxidation conditions. Slightly higher sugar concentrations might also be acceptable, but longer term testing at these higher concentrations and product testing of the more highly reduced glass would be necessary to optimize this parameter. It should be noted that the variation in Fe(II) percentage exhibited by 32 and 35 wt% glasses produced with a SBW sugar loading of 135 g/L is not considered significant, as variabilities in this parameter of up to  $\pm 2\%$  are normally observed.

**Table 4.9. SBW Feed, and Sugar Loadings Vs. Glass Oxidation State**

Sample #	Date/Time	%SBW	Sugar (g/L)	%Fe(II)
RSM-1-01-30	01/31/01 05:16	32	135.0	1.5
RSM-1-01-39	01/31/01 14:20	32	135.0	1.6
RSM-1-01-53	02/01/01 08:50	35	135.0	0.2
RSM-1-01-58	02/01/01 14:15	35	135.0	0.3
RSM-1-01-64	02/01/01 19:40	35	135.0	0.5
RSM-1-01-69	02/01/01 22:21	35	183.5	12.7
RSM-1-01-77	02/02/01 05:34	35	154.5	5.8
RSM-1-01-78	02/02/01 07:55	35	154.5	2.3
RSM-1-01-79	02/02/01 12:30	35	165.6	1.9
RSM-1-01-80	02/02/01 13:25	35	165.6	2.9
Not Assigned	02/02/01 14:35	35	165.6	2.7
RSM-1-01-83	02/02/01 16:22	35	177.0	2.8
RSM-1-01-85	02/02/01 17:40	35	177.0	7.7
RSM-1-01-87	02/02/01 18:34	35	196.7	13.8
RSM-1-01-89	02/02/01 19:15	35	196.7	23.3
RSM-1-01-92	02/02/01 20:18	35	196.7	45.0



**Figure 4.1. Historical SBW Sugar Concentration and Glass Oxidation-State Values**

## 4.5.2 Compositional Data

Glass-product compositional data associated with all melter processed feeds of differing waste loadings are summarized in Table 4.10 along with their corresponding target values. 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 and others that could be greater than expectations presumably due to unintended contributions from the bulk chemicals used. The classic volatiles/semi-volatiles feed components (e.g., B, alkalis, etc), with the possible exception of S, do not appear to have partitioned significantly to the process exhaust. Partitioning values or melter DFs will be discussed in a later section dealing with off-gas emission characterization. The average specific gravities of the 30%, 32%, and 35 % SBW glasses were determined to be 2.60, 2.61, and 2.61, respectively.

## 4.5.3 TCLP Results

Representative glass samples generated by all SWB feeds processed during the January 2001 RSM campaign were subjected to EPA's TCLP test. The results obtained from the TCLP testing are summarized in Table 4.11.

All analytical results obtained from these tests were below detection limits except for Pb in one of the 35% SBW glasses tested. The sample exhibiting the positive Pb result was obtained at the end of RSM testing when the most highly reduced process glass was produced. Specifically, this glass sample,



RSM-01-1-92, was obtained at the very end of the test after the RSM had processed 12 L (3 gal) of feed-containing SBW sugar loadings of 197g/L. Referring to Table 4.9 and Figure 4.1 in the previous section, the %Fe(II) in this particular sample was 45%. Recognizing, as previously discussed, that glasses containing toxic constituents become measurably less durable when their %Fe(II) values are >30%, the above result is less surprising than predictable. It should be noted, however, that even at this highly reduced state, this glass still conforms with all existing RCRA land-disposal limits.

**Table 4.10. Oxide Composition of Vitrified SBW Melter Feeds**

Oxide	Wt%							
	Startup	30% SBW		32% SBW		35% SBW		
	RSM-4	RSM-21	Target	RSM-44	Target	RSM-72	RSM-92	Target
Al <sub>2</sub> O <sub>3</sub>	7.000	8.070	8.370	8.580	8.930	9.670	9.660	9.770
B <sub>2</sub> O <sub>3</sub>	11.900	10.800	10.600	10.400	10.300	9.840	9.990	9.870
BaO	0.009	0.006	0.002	0.007	0.002	0.007	0.007	0.002
CaO	1.620	3.400	4.160	3.550	4.110	3.480	3.490	4.020
CdO	0.007	0.021	0.024	0.024	0.026	0.021	0.015	0.028
Ce <sub>2</sub> O <sub>3</sub>	---	---	0.002	---	0.002	---	---	0.002
Co <sub>2</sub> O <sub>3</sub>	---	---	---	---	---	---	0.017	---
Cr <sub>2</sub> O <sub>3</sub>	0.101	0.114	0.063	0.159	0.067	0.178	0.143	0.073
Cs <sub>2</sub> O	0.013	0.033	0.060	0.036	0.064	0.041	0.039	0.070
CuO	0.006	0.007	0.014	0.006	0.015	0.012	0.007	0.016
Fe <sub>2</sub> O <sub>3</sub>	10.200	8.070	7.430	7.680	7.260	7.370	7.300	7.000
Gd <sub>2</sub> O <sub>3</sub>	---	---	0.008	---	0.008	---	---	0.009
K <sub>2</sub> O	4.190	2.460	2.280	2.210	2.430	2.550	2.310	2.660
Li <sub>2</sub> O	3.460	3.560	3.500	3.380	3.400	3.360	3.150	3.250
MgO	0.645	0.218	0.120	0.192	0.128	0.197	0.225	0.140
MnO	0.621	0.311	0.247	0.298	0.263	0.321	0.318	0.288
MoO <sub>3</sub>	0.013	---	0.007	---	0.007	---	---	0.008
Na <sub>2</sub> O	10.600	15.400	15.700	16.300	16.800	17.800	18.200	18.400
NiO	0.168	0.080	0.027	0.688	0.029	0.090	0.100	0.032
P <sub>2</sub> O <sub>5</sub>	0.608	---	0.240	---	0.256	0.117	---	0.280
PbO	0.046	0.090	0.072	0.090	0.077	0.103	0.095	0.084
RuO <sub>2</sub>	0.031	0.033	0.004	0.031	0.004	0.024	0.035	0.005
SO <sub>3</sub>	0.216	0.679	1.070	0.664	1.140	0.697	0.885	1.250
SiO <sub>2</sub>	44.300	46.300	45.500	45.600	44.200	44.000	43.900	42.300
SrO	0.255	0.033	0.003	0.016	0.003	0.013	0.012	0.004
TiO <sub>2</sub>	0.560	0.073	0.001	0.041	0.001	0.034	0.029	0.001
ZnO	0.010	0.018	0.021	0.019	0.023	0.022	0.022	0.025
ZrO <sub>2</sub>	3.360	0.276	0.002	0.060	0.002	0.119	0.044	0.002

**Table 4.11. TCLP Leachate Concentrations from SBW Product Glasses**

Element	Limit µg/cc	Concentration (µg/cc)			
		30% SBW	32% SBW	35% SBW	35% SBW
Barium	100	<0.2	<0.2	<0.2	<0.2
Chromium	5	<0.05	<0.05	<0.05	<0.05
Lead	5	<0.2	<0.2	<0.2	0.37
Cadmium	1	<0.05	<0.05	<0.05	<0.05

## 5.0 Discussion of Results

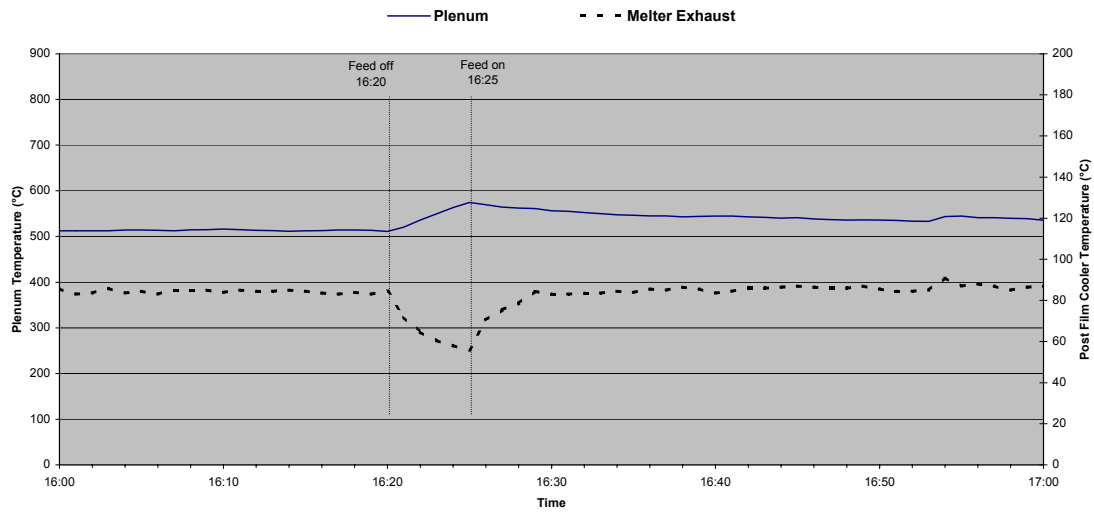
RSM testing of INEEL's SBW flowsheet was initiated on January 29, 2001, and concluded on February 2, 2001. During this 120-h period, melter feeds with three different SBW waste loadings (30, 32, and 35 Wt%) were successfully processed. In addition, the impacts of varying reductant concentrations (135, 155, 166, 177, 184, and 197 g/L) upon processing rates, molten salt accumulations, and glass oxidation state [Fe(II):Fe(III), see Sect 4.5.1] were evaluated. The effects of a 40% increase in SBW sulfate concentration upon melter performance and molten salt phase accumulations were also assessed during the final hours of melter testing. The observations and experimental test results derived from all phases of SBW melter testing will now be discussed.

### 5.1 Processing Observations and Parameters

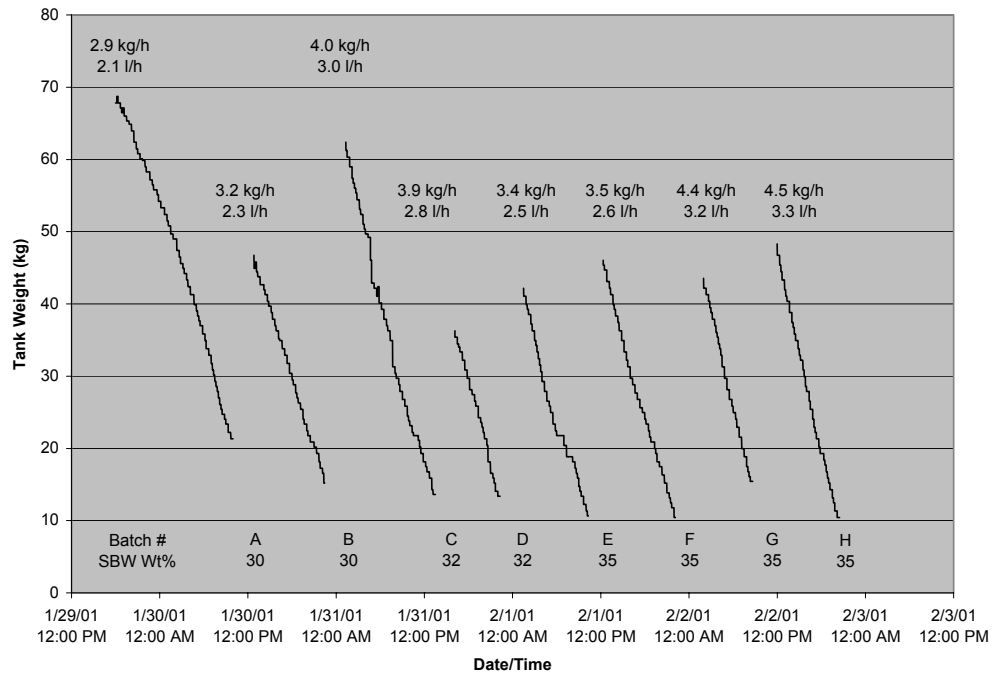
The melting kinetics of all feeds processed, irrespective of their SBW waste loading, was found to be nominally limited to liquid boil-off rates. That is, after dry out, residual melter-feed constituents were found to be almost immediately incorporated into the molten vitreous pool. There is very little evidence, during the processing of the SBW melter feed material, for the presence of a conventional solid cold cap composed of dried feed and calcine. Figure 5.1 illustrates how quickly the melter plenum temperature rises when feeding is interrupted. The time period between feed interruption and the rise in plenum temperature is nominally equivalent to the boil-off period of the melter's feed pool.

Since boiling occurs across the entire feed pool, the prospect of creating a process upset by cold-cap bridging of the melt pool was not ever a serious threat during SBW melter testing. However, feeding rates were lower at the beginning of RSM testing due to the lack of a significant salt phase that can facilitate feed-to-glass conversion and a healthy respect for the usual consequences associated with melter over feeding conditions. Figure 5.2 presents melter-feeding rates recorded for each of the feed batches prepared during RSM testing. These data reflect the average rates observed during active feed-processing periods. Prolonged non-feeding time intervals have been eliminated from these data so melter-processing rates could be cleanly extracted.

As explained earlier, the ~50% increase in melter feeding rate between the beginning and end of SBW melter testing had much more to do with operator conservatism than feed-stream characteristics. In other words, the fact that 35 wt% feeds were processed faster than those of lower waste-loading feeds has a lot to do with the sequencing of the feed formulations processed. However, the gradual accumulation of a molten salt phase that facilitated heat-transfer rates and the increased sugar content that provided additional thermal energy release also contributed to the enhanced processing rates observed during the latter stages of melter testing.

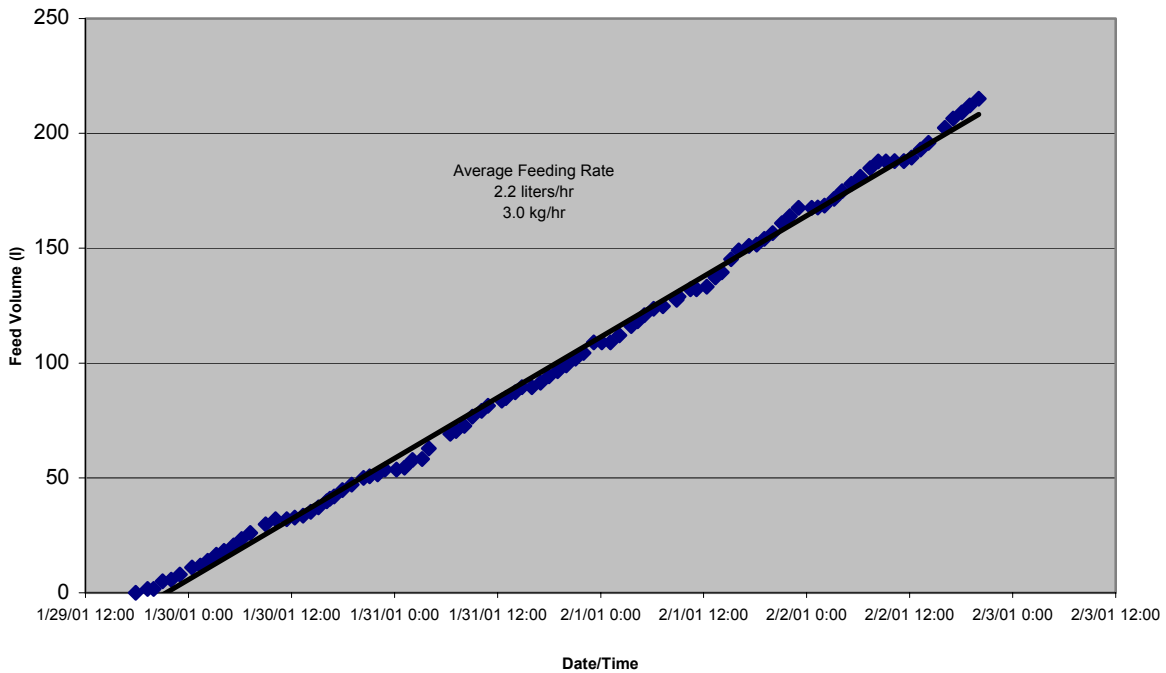


**Figure 5.1. Operating Temperature Characteristics During Cold Cap Burn Off**



**Figure 5.2. Processing History of Sequential RSM Feed Batches**

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 5.3. 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. A much higher process efficiency (total operating efficiency [TOE]) would result if batch preparation down times were excluded for these data. Table 5.1 summarizes all the graphical feeding rate data previously discussed.

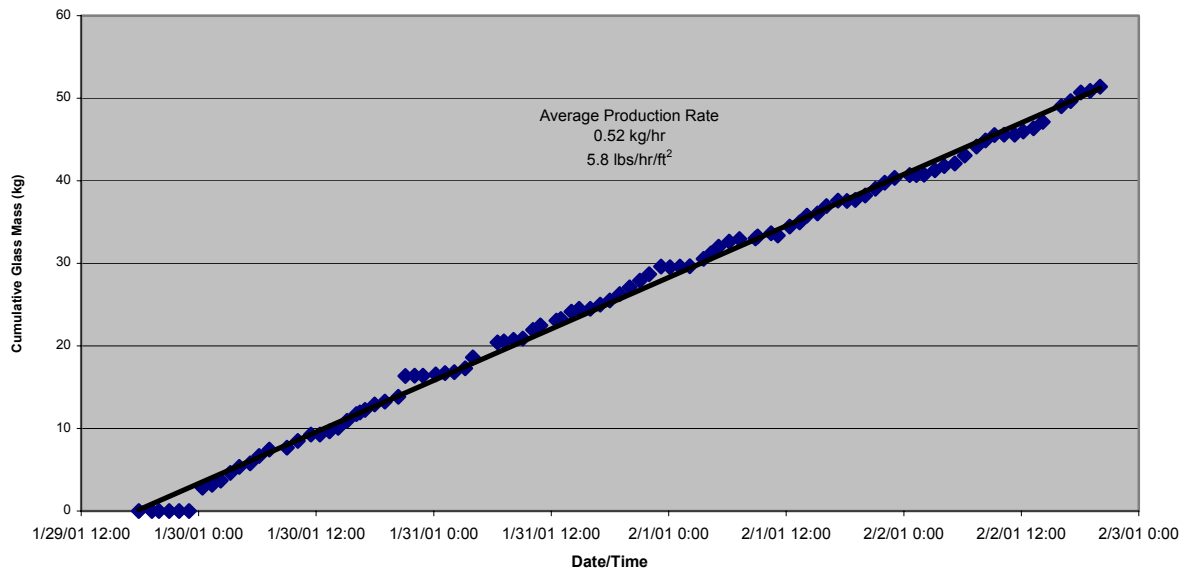


**Figure 5.3. Overall Average RSM Processing Rate**

Also included in Table 5.1 are area-normalized glass-production rates derived from the nominal total oxide loading of the feed (300 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 4 lbs/h/ft<sup>2</sup> that is often quoted and used for flowsheet and equipment-sizing estimates. Indeed, this reference normalized-production rate is even exceeded when projections are based upon the overall average feeding rate that is inclusive of all idle batching periods. This derived overall average-production-rate value also agrees fairly well with the actual glass-accumulation information that was manually recorded throughout the duration of the test if we take into account the inherent bias introduced by glass-sampling activities. These glass accumulation data are summarized in Figure 5.4.

**Table 5.1. SBW Feeding and Glass Production Rates**

Feed Batch	SBW Wt%	Feeding Rate		Glass Lbs/h/ft <sup>2</sup>
		L/h	kg/h	
A	30	2.1	2.9	7.1
B	30	2.3	3.2	7.7
C	32	3.0	4.0	10.1
D	32	2.8	3.9	9.4
E	35	2.5	3.4	8.4
F	35	2.6	3.5	8.8
G	35	3.2	4.4	10.8
H	35	3.3	4.5	11.1
---	All	2.2	3.0	7.4



**Figure 5.4. RSM Glass Accumulation Data**

## 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
- 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 during the active processing periods for each melter feed batch (see also Figure 5.2). 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 batch-processing stability. Table 5.3 provides similar daily tabular data for the melter's kiln, overflow spout, and canister oven.

All variable process-temperature data collected during the SBW flowsheet evaluations are graphically presented in Appendix E using various logical groupings. The pairing of melter plenum and post-film cooler off-gas temperature traces reveals a novel mirror image symmetry (inverse relationship), which is not necessarily intuitive, but nevertheless, logical. Unlike the plenum and melter off-gas (MOG) temperatures, the temperature variations of post-film cooler off-gas and EVS exhaust temperatures are, as expected, highly correlated. Although the temperature history of the EVS scrubbing liquor is correlated with the MOG 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.

### **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. Recognizing that automatic off-gas control based on plenum pressure was only used during the first 36 h of processing, it is apparent that vacuum control of the melter and its off-gas

system was easily maintained throughout all phases of testing, except possibly for the overfeeding event that occurred at 06:00 on 1/31/01.

**Table 5.2. RSM's Operating Temperature Characteristics**

Batch	Item	Temperature (°C)			
		Ave	Std Dev	Min	Max
<b>A</b>	Glass	1158	9	1098	1192
	Plenum	530	38	457	679
	MOG	82	11	44	121
	EVS Exh	18	1	15	20
	Srub Liq	27	3	18	35
<b>B</b>	Glass	1159	14	1017	1199
	Plenum	557	52	491	695
	MOG	91	15	54	147
	EVS Exh	19	---	17	20
	Srub Liq	29	2	22	33
<b>C</b>	Glass	1162	13	1039	1200
	Plenum	544	68	289	715
	MOG	93	15	44	117
	EVS Exh	19	1	18	22
	Srub Liq	31	4	20	48
<b>D</b>	Glass	1160	3	1149	1168
	Plenum	525	31	493	646
	MOG	101	8	72	147
	EVS Exh	20	---	18	21
	Srub Liq	32	1	25	45

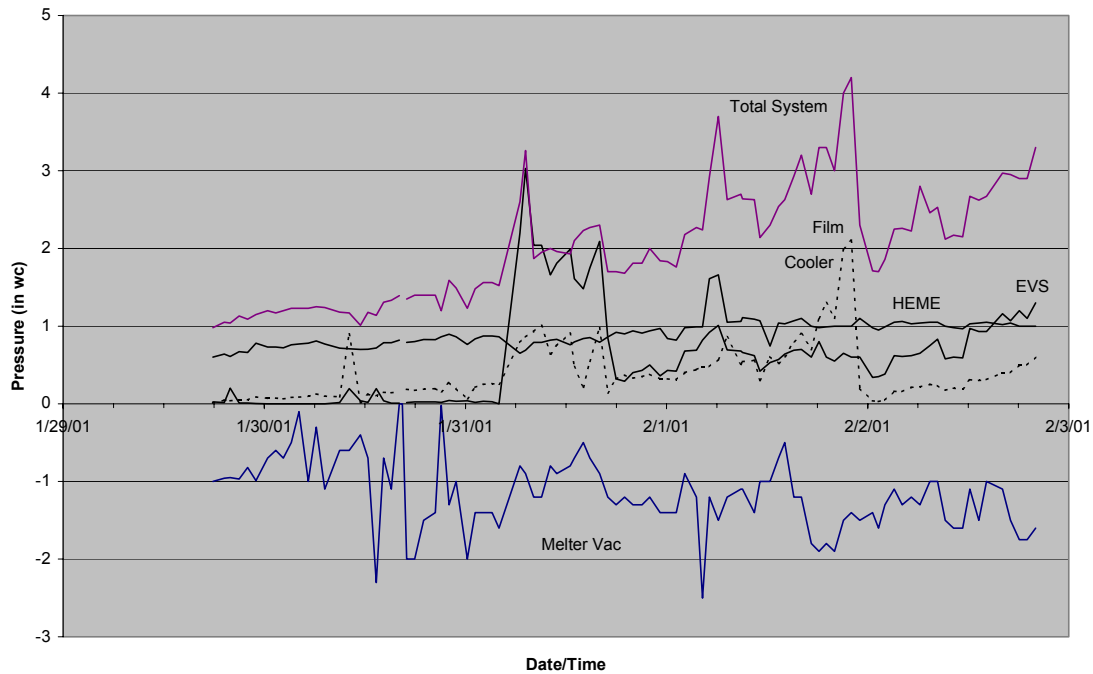


**Table 5.2 (contd)**

Batch	Item	Temperature (°C)			
		Ave	Std Dev	Min	Max
<b>E</b>	Glass	1152	24	1003	1240
	Plenum	557	67	454	719
	MOG	99	15	63	144
	EVS Exh	20	1	18	22
	Srub Liq	31	3	22	34
<b>F</b>	Glass	1148	12	1062	1164
	Plenum	547	47	477	713
	MOG	95	7	72	124
	EVS Exh	21	1	18	22
	Srub Liq	33	1	25	36
<b>G</b>	Glass	1147	5	1101	1156
	Plenum	510	49	465	707
	MOG	111	8	76	126
	EVS Exh	21	1	18	23
	Srub Liq	34	2	22	37
<b>H</b>	Glass	1152	5	1140	1168
	Plenum	502	34	467	643
	MOG	111	12	69	145
	EVS Exh	23	1	20	24
	Srub Liq	36	2	25	44
<b>All</b>	Glass	1155	14	1003	1240
	Plenum	536	54	289	719
	MOG	96	15	44	147
	EVS Exh	20	2	15	24
	Srub Liq	31	4	18	48

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

Date /Statistic	Temperature (°C)				
	Kiln			Pour	Can
	Bot	Mid	Top	Spout	Oven
<b>1/29/01</b>					
Average	855	845	827	1060	734
StdDev.	10	11	11	26	41
Minimum	834	821	803	998	597
Maximum	867	862	842	1080	762
<b>1/30/01</b>					
Average	856	845	826	1040	748
StdDev.	14	15	16	23	16
Minimum	799	789	760	832	500
Maximum	876	865	847	1070	773
<b>1/31/01</b>					
Average	862	851	830	1050	749
StdDev.	4	5	5	2	15
Minimum	849	832	817	1040	475
Maximum	881	869	845	1060	788
<b>2/1/01</b>					
Average	862	852	832	1050	748
StdDev.	4	6	7	2	19
Minimum	851	840	822	1040	408
Maximum	874	872	855	1060	775
<b>2/2/01</b>					
Average	864	854	832	1050	747
StdDev.	6	9	10	1	22
Minimum	851	839	820	1040	486
Maximum	882	881	860	1060	761
<b>1/29/01 - 2/2/01</b>					
Average	860	850	830	1050	747
StdDev.	9	10	11	14	20
Minimum	799	789	760	832	408
Maximum	882	881	860	1080	788



**Figure 5.5. Melter and Off-Gas Operating Pressures**

As an explanatory note, the steady rise in off-gas system pressure drops indicated by the graphical-pressure data is due to a combination of the following factors:

- Increasing plenum vacuum
- Increasing melter feeding rates
- Decreasing conductance (i.e., blockage) of the melter/EVS jumper.

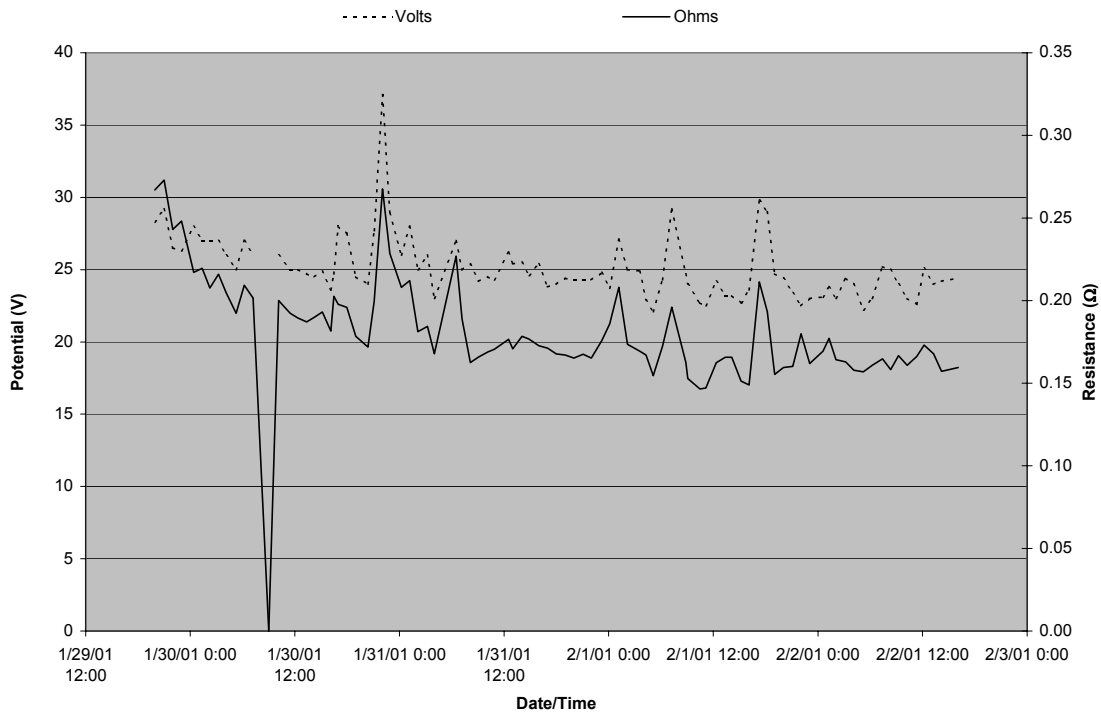
It should be noted that the apparent buildup of off-gas debris in the melter's off-gas jumper is not a natural outcome of normal process operations. Rather, it is a consequence of the overfeeding upset event that created partial off-gas line obstructions that continually grew in size by capturing and accumulating entrained debris. This subject will be further discussed in Section 7.

### 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).

Under normal operating conditions, an interface module provides the RSM's PLC with analog signals directly related to the voltage current and power outputs of each SCR. During the current test, the electrode's SCR interface module failed, requiring these electrical operating parameters to be measured and recorded manually (see Appendix C). Figures 5.6 to 5.8 graphically summarized these electrical quantities in various groupings to allow correlations to be easily recognized.

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 apparent from the various parameter pairings shown are strong correlations between voltage, power, and resistance, as is expected from their functional interrelationships. Consequently, the short-term average values of both voltage and glass resistance also remained fairly constant throughout the testing period, although conductance, as expected, appears to have increased with waste loading.



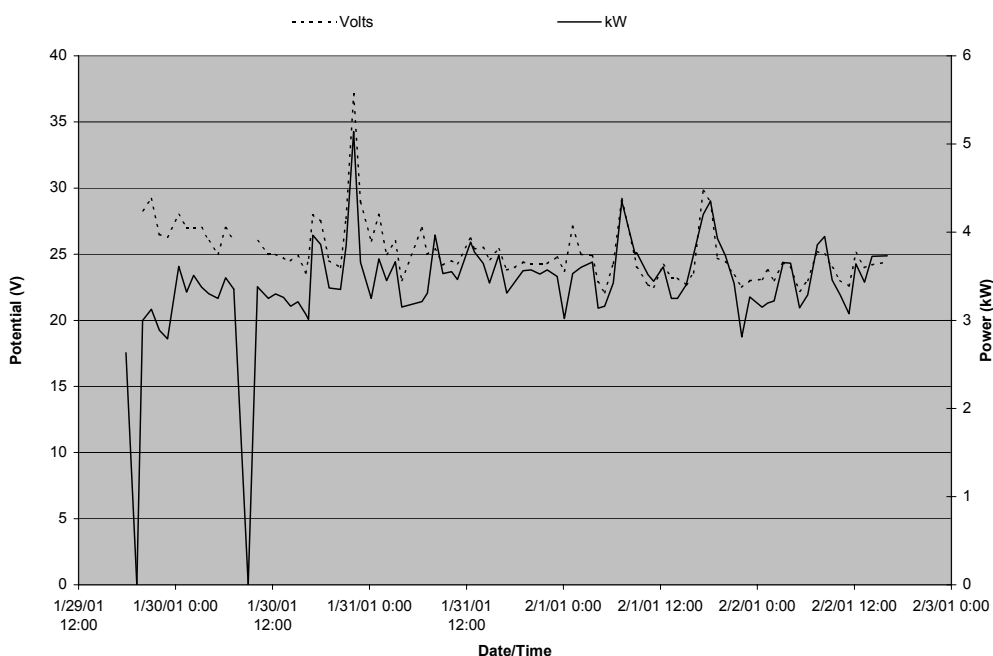
**Figure 5.6. RSM Electrode Voltage/Power Operating Characteristics**

In the afternoon of February 1st at about 16:49, almost 72 h into the test the electrode voltage and current readings were observed to be erratic. Coincidentally, the glass temperature thermocouple readings were declining with the indicated temperature being 1080°C. This indicated that electrical shorting between the electrodes was occurring. The feed was turned off to melt the cold cap, and power to the electrodes was turned off to allow observation and probing of the melter. Probing of the melter indicated a detectable salt layer that was estimated to be 1 to 3 mm in depth and did not cover a majority

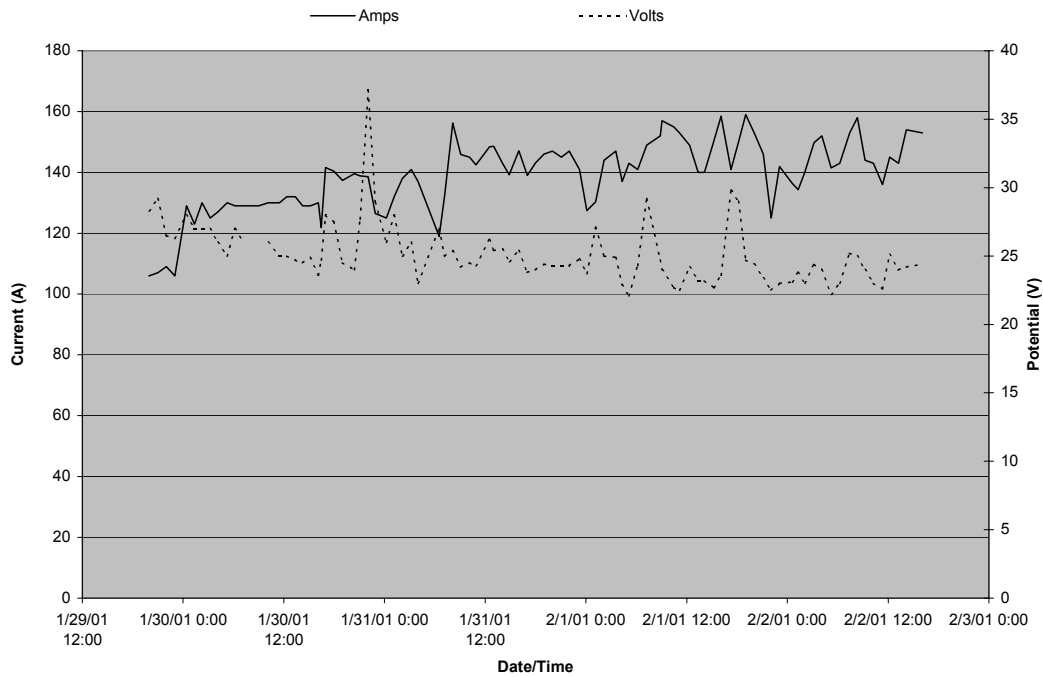
of the glass surface. Upon turning power back on to the electrodes, the current and voltage readings were stable. Two possible explanations or hypotheses can be proposed. When the cold cap is present, it creates “bands” of molten salt that can contact and short the electrodes. The second, and considered more likely explanation, is that during this period of processing, nearly fully flooded conditions were being maintained. Additionally, the SBW melter feed was not creating a solids cold cap; rather, a thin boiling layer was observed to occur. In this condition, molten salt would be dissolved into the aqueous phase creating a highly conductive salt solution. Further, the dissociated sodium sulfate in the aqueous phase would be very electrically conductive compared to the molten sodium sulfate.

Because electrode shorting in past campaigns has led to electrode damage, this period of no feeding was also used to lift the electrodes from the glass and visually inspect them. Both electrodes looked to be in excellent condition, possessing sharp shoulder corners. The east electrode appeared to be perhaps slightly thinner with “softer” corners. However, given the restricted viewing conditions, it was hard to be certain.

Table 5.4 summarizes the maximum, minimum, and average electrode operating parameters and their standard deviations during the active processing periods for each melter feed batch (see Figure 5.2). The average Joule heating requirements of each batch in this table were used with corresponding average batch-feeding rates (see Table 5.1) to derive specific Joule-energy processing requirements for SBW feeds. These data are summarized in Table 5.5.



**Figure 5.7. RSM Electrode-Voltage/Glass-Resistance Properties**



**Figure 5.8. RSM Electrode Voltage/Current Operating Characteristics**

**Table 5.4. RSM Electrode Circuit Operating Characteristics**

Batch	Item	RSM Electrode Parameters			
		Ave	Std Dev	Min	Max
<b>A</b>	Volts	27	1	25	29
	Amps	119	13	94	130
	kVA	3.2	0.3	2.6	3.6
	Ohms	0.23	0.03	0.19	0.30
<b>B</b>	Volts	26	3	24	37
	Amps	134	6	122	142
	kVA	3.6	0.5	3.0	5.1
	Ohms	0.20	0.02	0.17	0.27
<b>C</b>	Volts	25	1	23	28
	Amps	141	7	119	156
	kVA	3.5	0.2	3.2	4.0
	Ohms	0.18	0.01	0.16	0.23

**Table 5.4 (contd)**

<b>Batch</b>	<b>Item</b>	<b>RSM Electrode Parameters</b>			
		<b>Ave</b>	<b>Std Dev</b>	<b>Min</b>	<b>Max</b>
<b>D</b>	Volts	24	---	24	25
	Amps	145	3	139	147
	kVA	3.5	0.1	3.3	3.7
	Ohms	0.17	0.00	0.17	0.17
<b>E</b>	Volts	25	2	22	29
	Amps	145	7	130	157
	kVA	3.7	0.4	3.1	4.4
	Ohms	0.17	0.02	0.15	0.21
<b>F</b>	Volts	24	2	23	30
	Amps	147	10	125	159
	kVA	3.5	0.4	2.8	4.4
	Ohms	0.16	0.01	0.15	0.21
<b>G</b>	Volts	24	1	22	25
	Amps	147	6	134	158
	kVA	3.5	0.3	3.1	4.0
	Ohms	0.16	0.00	0.16	0.18
<b>H</b>	Volts	24	1	20	25
	Amps	150	6	134	156
	kVA	3.6	0.2	2.7	3.7
	Ohms	0.16	0.01	0.15	0.17
<b>All</b>	Volts	25	2	20	37
	Amps	139	14	94	159
	kVA	3.5	0.4	2.6	5.1
	Ohms	0.18	0.03	0.15	0.30

Typical energy requirements for slurry feeding Joule-heated ceramic melters range from 2 to 4 kW\*h/kg of glass produced. Recognizing that the SBW process flowsheet does not provide for pretreatment or concentration and that much of the power required in processing of slurry feeds is consumed by boiling away water, the slightly higher specific energy requirements for vitrifying SBW is largely due to the higher-than-normal weight fraction of water in the melter feed stream. It should also be noted that in addition to the electrical power delivered to the melt pool, a significant combustion energy is also being provided by the reductant feed-stream component. By combining this combustion power 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 5.5. Specific Process Energy Requirements For SBW Feeds**

Feed Batch	SBW Wt%	Feeding Rate (L/h)	Glass Lbs/h/ft <sup>2</sup>	Sucrose (g/L-SBW)	Power (kW)		Spec Engy (kW-h/kg)	
					Joule	Combust	Joule	Total
A	30	2.1	7.1	135.0	3.2	1.0	5.1	6.7
B	30	2.3	7.7	135.0	3.6	1.1	5.2	6.8
C	32	3.0	10.1	135.0	3.5	1.4	3.9	5.5
D	32	2.8	9.4	135.0	3.5	1.4	4.2	5.8
E	35	2.5	8.4	135.0	3.7	1.2	4.9	6.5
F	35	2.6	8.8	149.5	3.5	1.4	4.5	6.3
G	35	3.2	10.8	154.5	3.5	1.8	3.6	5.5
H	35	3.3	11.1	178.1	3.6	2.1	3.6	5.8
							Average = 6.1 Std Dev = 9%	

#### 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' scrubbing-liquor temperature remained fairly constant (~30°C) throughout RSM testing. Consequently, the EVS also removed condensable melter-generated gases of which steam is a primary component. Figure 5.9 provides a historical summary of the EVS' scrubbing liquor/condensate tank volume throughout the duration of the RSM test.



The 1.1 L/h condensate accumulation rate is 57% of the overall average rate to which water was fed to the melter. If one directly compares the accumulated condensate volume (94.6 L [25 gal], neglecting solids contributions) and the total water volume (167 L [44 gal]) fed to the melter, a 56% steam recovery is obtained, which is in good agreement with the value derived from average process-rate values.

Also presented in Figure 5.9 is the manually recorded Film Cooler, injection-air rotameter reading. It should be pointed out that the recorded values are apparent and not actual flow rates. This is because of the backpressure at the exhaust of this rotameter, which was not routinely measured. However, before the test, the nominal back-pressure operating condition was determined to be 40 psig at 6 scfm. Under these conditions, the actual flow rate is ~2x the indicated flow. Compounding the rotameter reading uncertainty, the conductance of this device was probably not constant throughout the test period because of fouling that resulted from the melter overfeeding event (1/31/01 06:00). However, total melter off-gas 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, and 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 flow rates. These process flow rates are discussed and characterized in Section 6.1, and graphically summarized in Appendix F.

**Table 5.6. Operational Characteristics of Melter Kiln and Overflow Heaters**

Date /Statistic	Kiln			OverFlow Heater		
	Amps	Watts	Volts	Amps	Watts	Volts
<b>1/29/01</b>						
<b>Average</b>	4.95	0.30	21.20	3.65	0.45	118.00
<b>StdDev.</b>	3.88	0.11	16.30	5.77	0.71	5.82
<b>Minimum</b>	0.03	0.28	0.50	---	---	102.00
<b>Maximum</b>	14.40	0.78	61.30	20.20	1.58	131.00
<b>1/30/01</b>						
<b>Average</b>	6.83	0.31	29.20	12.80	1.47	127.00
<b>StdDev.</b>	3.32	0.07	14.30	1.78	0.29	11.00
<b>Minimum</b>	---	0.28	0.50	---	---	108.00
<b>Maximum</b>	16.20	0.80	71.80	14.20	1.80	209.00
<b>1/31/01</b>						
<b>Average</b>	5.92	0.31	25.30	12.80	1.57	123.00
<b>StdDev.</b>	3.21	0.07	13.70	0.29	---	2.55
<b>Minimum</b>	0.00	0.30	0.75	11.10	1.57	103.00
<b>Maximum</b>	15.90	0.81	68.50	13.50	1.57	131.00

Table 5.6 (contd)

Date /Statistic	Kiln			OverFlow Heater		
	Amps	Watts	Volts	Amps	Watts	Volts
<b>2/1/01</b>						
Average	5.54	0.30	23.60	12.90	1.57	124.00
StdDev.	3.16	---	13.60	0.22	---	2.51
Minimum	0.09	0.30	0.75	11.70	1.57	109.00
Maximum	9.66	0.30	42.00	13.50	1.57	131.00
<b>2/2/01</b>						
Average	5.45	0.30	23.40	13.00	1.57	125.00
StdDev.	3.27	---	13.80	0.24	---	2.37
Minimum	0.08	0.30	0.75	12.00	1.57	115.00
Maximum	9.17	0.30	39.00	13.50	1.57	131.00
<b>1/29/01 - 2/2/01</b>						
Average	5.89	0.30	25.20	12.30	1.48	124.00
StdDev.	3.33	0.06	14.20	2.79	0.35	6.35
Minimum	---	0.28	0.50	---	---	102.00
Maximum	16.20	0.81	71.80	20.20	1.80	209.00

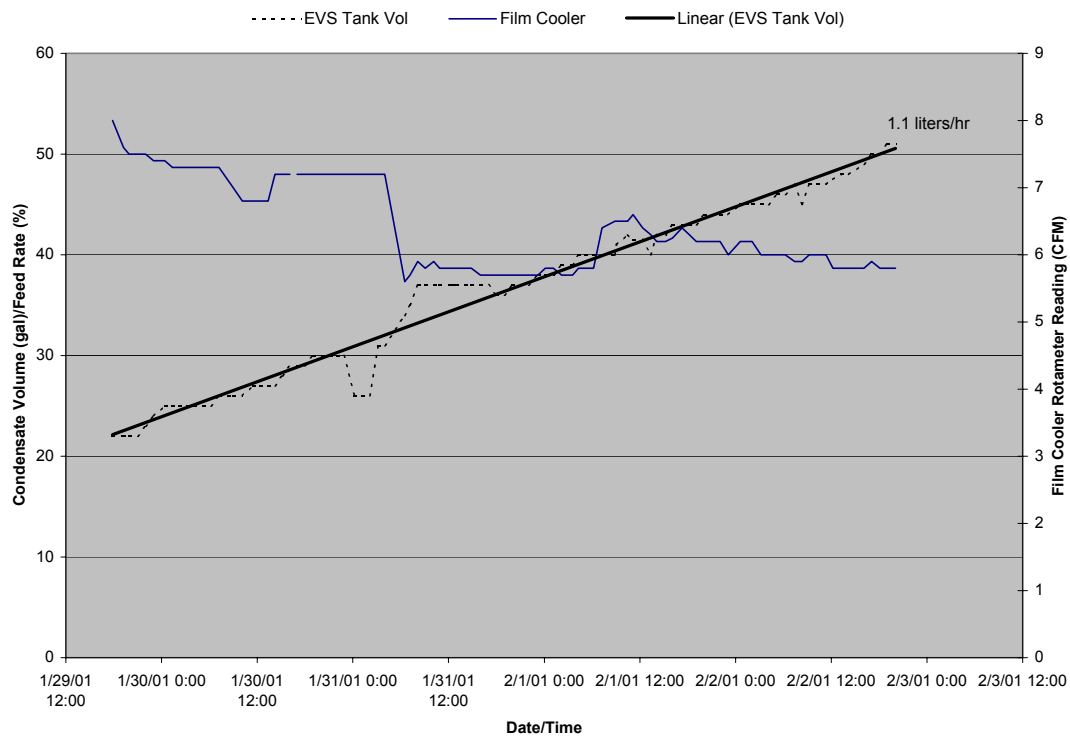


Figure 5.9. EVS Condensate Tank, Film-Cooler Operating History

## 6.0 Melter Off-Gas Emission Characterization

Off-gas effluent studies were conducted during SBW 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 effluent emissions was established using a variety of gas analyzers that are described Table 3.1. The instruments used were designed to continuously (or quasi-continuously) monitor and record process-exhaust concentrations of H<sub>2</sub>, He, N<sub>2</sub>, CO, NO, O<sub>2</sub>, CO<sub>2</sub>, N<sub>2</sub>O, NO<sub>2</sub>, and total hydrocarbons. In addition, a direct inlet mass spectrometer was used as a semi-quantitative tool to identify other gases that might be present, such as H<sub>2</sub>S, SO<sub>2</sub>, the halogens, and the hydrogen halides. Discrete sampling for the halogens and hydrogen halides was also conducted as described below.

To characterize condensable or reactive acid-gas effluent losses, a multi-component sampler composed of an absolute filter and a series of chemical gas scrubbers was 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 state or states assumed by this effluent 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 melter feed used during the January 2001 test, CO<sub>2</sub>, and NO<sub>x</sub> (specifically NO) 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 during the active processing periods for each melter feed batch (see Figure 5.2). Also presented in this table are the standard deviations associated with the average concentrations listed, and off-gas flow-rate data.

These tabular data, unfortunately, do not contain quantitative NO<sub>2</sub> concentrations due to poisoning of the NO<sub>x</sub> analyzer's catalytic reactor. Since the gas chromatograph was not responsive to NO<sub>2</sub>, the detection limit of the direct inlet mass spectrometer was used to place an upper bound for this effluent's concentration, which is discussed more fully further below. For a non-condensable off-gas flow rate of ~15 scfm and a steady-state feeding rate of 3 L/h, the MOG concentrations of the major effluent gases (CO<sub>2</sub> and NO) were found to be, nominally, 0.71% and 0.31%, respectively. The combustible gases CO and H<sub>2</sub> were also detected, but at much lower concentrations: 0.15% and 0.025%, respectively. These concentrations are well below the lower flammability limits of these combustible gases, 4.65% for H<sub>2</sub> and 15.5% for CO. Even without the benefit of film cooler dilution, off-gas concentrations of these

**Table 6.1. Unquenched Melter Off-Gas Composition**

Date/Batch/ SBW	Post Film Cooler Concentration (ppm)										FlwRate (scfm)
	He	H <sub>2</sub>	O <sub>2</sub>	N <sub>2</sub>	NO	CO	CO <sub>2</sub>	N <sub>2</sub> O	THA	NO*	
<b>1/29-1/30 / A / 30%</b>											
Average	4,000	90	161,000	615,000	1,840	756	5,810	677	62	#N/A	18
Std. Dev.	229	29	5,940	22,200	318	177	978	185	30	#N/A	1
Minimum	3,490	20	144,000	553,000	1,270	216	1,190	246	---	#N/A	14
Maximum	5,060	180	177,000	673,000	3,810	1,500	10,200	978	245	#N/A	20
<b>1/30-1/30 / B / 30%</b>											
Average	3,800	109	156,000	598,000	2,290	907	5,810	674	61	3,490	19
Std. Dev.	119	32	1,760	6,550	272	267	683	234	34	577	1
Minimum	3,200	25	152,000	582,000	1,620	94	4,040	130	1	263	17
Maximum	4,060	183	165,000	631,000	2,950	1,430	7,610	1,700	431	4,330	22
<b>1/31-1/31 / C / 32%</b>											
Average	4,290	146	160,000	613,000	2,960	1,210	7,020	750	86	3,840	17
Std. Dev.	738	39	12,000	46,000	574	299	1,160	199	94	519	2
Minimum	3,400	67	138,000	532,000	228	530	1,150	269	---	117	7
Maximum	9,480	417	198,000	761,000	5,540	2,120	15,100	1,380	767	5,260	21
<b>1/31-1/31 / D / 32%</b>											
Average	4,750	162	172,000	658,000	3,400	1,390	7,450	782	96	3,970	15
Std. Dev.	635	41	22,700	86,600	869	388	1,380	201	32	473	5
Minimum	989	26	35,400	135,000	324	267	1,120	127	1	1,760	10
Maximum	7,060	285	246,000	942,000	5,610	2,670	12,000	1,190	345	5,680	71
<b>2/01-2/01 / E / 35%</b>											
Average	4,160	162	183,000	704,000	4,060	1,600	7,040	774	78	2,470	17
Std. Dev.	574	50	22,600	85,700	558	319	1,210	132	42	361	2
Minimum	3,070	72	154,000	596,000	3,120	1,010	5,040	374	---	385	13
Maximum	5,620	285	246,000	941,000	5,510	2,510	10,200	1,050	434	2,890	23
<b>2/01-2/02 / F / 35%</b>											
Average	4,030	267	166,000	643,000	3,280	1,840	6,790	658	119	2,770	18
Std. Dev.	252	171	7,420	28,100	629	608	1,240	164	82	608	1
Minimum	2,190	40	129,000	499,000	30	51	761	150	---	297	12
Maximum	5,750	1,000	190,000	730,000	5,070	3,990	13,800	1,130	687	4,430	32

**Table 6.1 (contd)**

Date/Batch/ SBW	Post Film Cooler Concentration (ppm)										FlwRate (scfm)
	He	H <sub>2</sub>	O <sub>2</sub>	N <sub>2</sub>	NO	CO	CO <sub>2</sub>	N <sub>2</sub> O	THA	NO*	
<b>2/02-2/02 / G / 35%</b>											
<b>Average</b>	3,860	308	157,000	605,000	3,590	1,850	7,580	790	174	3,380	18
<b>Std. Dev.</b>	373	73	10,700	40,700	504	322	1,250	186	57	449	2
<b>Minimum</b>	2,570	54	106,000	412,000	149	291	754	241	4	48	12
<b>Maximum</b>	5,760	511	222,000	851,000	4,650	2,730	10,600	1,300	468	4,600	27
<b>2/02-2/02 / H / 35%</b>											
<b>Average</b>	3,950	487	150,000	581,000	3,380	2,550	9,320	780	209	3,410	18
<b>Std. Dev.</b>	194	126	6,560	25,400	398	474	932	203	146	597	1
<b>Minimum</b>	2,980	37	120,000	469,000	1,920	993	5,780	266	1	173	16
<b>Maximum</b>	4,420	767	166,000	646,000	4,360	3,510	11,400	1,110	767	4,390	24
<b>1/29-2/2 / Overall</b>											
<b>Average</b>	4,090	245	161,000	618,000	3,100	1,470	7,100	735	113	3,420	17
<b>Std. Dev.</b>	1,540	163	13,400	50,900	789	717	1,590	200	97	688	3
<b>Minimum</b>	24	20	35,400	135,000	30	51	754	127	---	48	1
<b>Maximum</b>	64,900	1,000	246,000	942,000	5,610	3,990	15,100	1,700	767	5,680	71

\* Chemiluminescent, NO<sub>x</sub> Analyzer value.

gases were found to be well below lower flammability as is shown in Table 6.2, which uses the batch feeding rates in Table 5.1 and the above tabular information to project melter off-gas composition before film-cooler dilution. In the plenum and before the film cooler, steam is the primary factor that eliminates the flammability hazards of H<sub>2</sub> and CO. The dilution caused by air inleakage and the film cooler precludes any possibility for auto-ignition downstream of the film cooler and EVS.

A dilution-control-system instability specific to the NO<sub>x</sub> analyzer is thought to be responsible for the lack of agreement between gas chromatograph (GC) and NO<sub>x</sub> analyzer responses for NO; as a result, the GC's NO data are preferred over the corresponding NO<sub>x</sub> analyzer responses.

As with NO<sub>2</sub>, the on-line gas chromatograph was found to be unresponsive to the effluent gas species SO<sub>2</sub>, and the sensitivity of the direct-inlet mass spectrometer was insufficient to detect it. Consequently, an upper-bound concentration was assigned to this effluent species based on the noise floor and detection efficiency of the mass spectrometer. The lack of SO<sub>2</sub> results in this data does not necessarily mean that volatility losses for sulfur are small; rather, it is a reflection of the fact that SO<sub>2</sub> production is not thermodynamically favored when plenum temperatures are <600°C. SO<sub>3</sub> is a reactive condensable gas that could not, therefore, be detected by the ambient-temperature gas analyzers used; however, the magnitude of the SO<sub>3</sub> loss mechanism will be evaluated later in this section when the EVS scrubbing liquor composition is discussed.

**Table 6.2. Calculated Melter Effluent Concentrations Before Film Cooler Dilution**

Date/Batch/SBW	Projected Melter Exhaust Concentration (ppm)									FlwRate (scfm)	
	He	H <sub>2</sub>	O <sub>2</sub>	N <sub>2</sub>	NO	CO	CO <sub>2</sub>	N <sub>2</sub> O	THA		NO*
1/29-1/30 / A / 30%											
Average	51,700	1,170	---	---	23,800	9,760	75,100	8,750	801	#N/A	1.4
Std. Dev.	2,960	370	---	---	4,110	2,290	12,600	2,390	386	#N/A	---
Minimum	45,100	255	---	---	16,400	2,790	15,300	3,180	5	#N/A	---
Maximum	65,400	2,330	---	---	49,300	19,300	132,000	12,600	3,170	#N/A	---
1/30-1/30 / B / 30%											
Average	47,600	1,370	---	---	28,700	11,400	72,700	8,440	764	43,700	1.5
Std. Dev.	1,500	403	---	---	3,400	3,350	8,550	2,930	419	7,210	---
Minimum	40,000	310	---	---	20,200	1,170	50,600	1,630	10	3,300	---
Maximum	50,800	2,290	---	---	37,000	17,800	95,200	21,200	5,390	54,200	---
1/31-1/31 / C / 32%											
Average	38,300	1,310	---	---	26,400	10,800	62,700	6,690	770	34,300	1.9
Std. Dev.	6,590	352	---	---	5,120	2,670	10,400	1,780	840	4,630	---
Minimum	30,400	598	---	---	2,040	4,740	10,300	2,400	3	1,050	---
Maximum	84,600	3,730	---	---	49,500	18,900	135,000	12,400	6,850	47,000	---
1/31-1/31 / D / 32%											
Average	40,600	1,380	---	---	29,100	11,800	63,600	6,670	820	33,900	1.8
Std. Dev.	5,420	350	---	---	7,420	3,310	11,700	1,710	274	4,030	---
Minimum	8,440	220	---	---	2,770	2,280	9,540	1,080	7	15,000	---
Maximum	60,300	2,430	---	---	47,900	22,800	103,000	10,100	2,950	48,500	---
2/01-2/01 / E / 35%											
Average	45,800	1,780	---	---	44,700	17,700	77,600	8,540	856	27,200	1.6
Std. Dev.	6,330	552	---	---	6,150	3,520	13,300	1,460	467	3,980	---
Minimum	33,800	790	---	---	34,300	11,100	55,500	4,120	4	4,250	---
Maximum	61,900	3,140	---	---	60,800	27,700	112,000	11,600	4,790	31,900	---
2/01-2/02 / F / 35%											
Average	43,100	2,860	---	---	35,100	19,700	72,600	7,040	1,280	29,600	1.6
Std. Dev.	2,690	1,820	---	---	6,730	6,500	13,300	1,750	882	6,500	---
Minimum	23,400	428	---	---	319	545	8,150	1,600	4	3,180	---
Maximum	61,600	10,700	---	---	54,200	42,700	148,000	12,100	7,350	47,400	---

**Table 6.2 (contd)**

Date/Batch/SBW	Projected Melter Exhaust Concentration (ppm)										FlwRate (scfm)
	He	H <sub>2</sub>	O <sub>2</sub>	N <sub>2</sub>	NO	CO	CO <sub>2</sub>	N <sub>2</sub> O	THA	NO*	
<b>2/02-2/02 / G / 35%</b>											
<b>Average</b>	35,000	2,800	---	---	32,600	16,800	68,800	7,170	1,580	30,600	2.0
<b>Std. Dev.</b>	3,390	661	---	---	4,570	2,920	11,400	1,690	518	4,080	---
<b>Minimum</b>	23,300	493	---	---	1,350	2,640	6,840	2,180	38	436	---
<b>Maximum</b>	52,300	4,640	---	---	42,200	24,800	96,000	11,800	4,240	41,700	---
<b>2/02-2/02 / H / 35%</b>											
<b>Average</b>	33,800	4,170	---	---	29,000	21,800	79,800	6,670	1,790	29,200	2.1
<b>Std. Dev.</b>	1,660	1,070	---	---	3,400	4,060	7,980	1,730	1,250	5,110	---
<b>Minimum</b>	25,500	320	---	---	16,500	8,500	49,500	2,280	7	1,480	---
<b>Maximum</b>	37,800	6,570	---	---	37,300	30,100	97,400	9,500	6,560	37,500	---
<b>1/29-2/2 / Overall</b>											
<b>Average</b>	41,200	2,470	---	---	31,300	14,900	71,600	7,410	1,140	34,400	1.7
<b>Std. Dev.</b>	15,500	1,650	---	---	7,960	7,230	16,000	2,010	981	6,930	---
<b>Minimum</b>	240	199	---	---	301	514	7,600	1,280	4	484	---
<b>Maximum</b>	654,000	10,100	---	---	56,600	40,200	152,000	17,100	7,730	57,300	---

\* Chemiluminescent, NO<sub>x</sub> Analyzer value.

The responses of the total hydrocarbon analyzer indicated that volatile organic compounds (VOCs) were present in the melter exhaust throughout most periods of testing. Although the off-gas concentrations of these thermal byproducts of incomplete oxidation were relatively low, 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 VOC concentration spikes observed during RSM testing.

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 effluents remained relatively invariant. They were, however, 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.

Referring to the direct inlet quadrupole data in Appendix G, the basis for the upper-bound concentration assignments for NO<sub>2</sub> and SO<sub>2</sub> can now be discussed. At first glance, it appears that the quadrupole's response at mass 46 is directly related to the presence of NO<sub>2</sub> in the sampled off-gas stream and that its concentration is significantly below that of NO. Unfortunately, other effluent gases also produce responses at this mass number. Specifically, isotopic forms of CO<sub>2</sub> and N<sub>2</sub>O (nominally mass 44) both contribute to the observed mass-46 response. Although the contributions from both CO<sub>2</sub>

and  $\text{N}_2\text{O}$  are small fractions of their full mass responses, 0.4% and 0.1%, respectively, they are significant since  $\text{CO}_2$  and  $\text{N}_2\text{O}$  are major contributors to the melter's off-gas source term.

When these minor mass-46 contributions from these major effluent species are taken into account, the remaining residual partial pressure is indistinguishable from the noise floor level at this mass number. Assuming this mass-46 noise floor represents the maximum detectable  $\text{NO}_2$  partial pressure, and using the instrument's relatively low ionization efficiency and small fractional full-mass yield (50%) and applying a factor of  $\sim 10$  to account for off-gas to instrument dilution, the minimum  $\text{NO}_2$  off-gas concentration detectable by the quadrupole was determined to be  $\sim 3000$  ppm. Based on this value and Table 6.1, equivalent off-gas concentrations of  $\text{NO}$  and  $\text{NO}_2$  could have been present throughout most of the RSM test. Unfortunately, little meaningful process data regarding  $\text{NO}_2$  can be extracted from the available online analytical instruments.

The bounding concentration for  $\text{SO}_2$  was similarly determined, but for the case of  $\text{SO}_2$ , no significant process-related changes in the mass-64 responses were ever detected, as graphical data clearly show. Using the mass-64 noise floor, the instrument's  $\text{SO}_2$  detection efficiency, and the  $\sim 10\times$  sample-dilution factor, the minimum  $\text{SO}_2$  detectable off-gas concentration was determined to be 3000 ppm. Sulfate analysis of off-gas waste-stream solutions, resulting primarily from vapor carry over of condensable  $\text{H}_2\text{SO}_4$  and its anhydride  $\text{SO}_3$ , should help resolve whether this bounding  $\text{SO}_2$  concentration is at all reasonable. These mass-balance evaluations will be considered in Section 7.

## 6.2 Condensed Phase Effluents

The effluents that enter the MOG system that require long-term environmental isolation are primarily condensed-phase matter (i.e., aerosols). Many feed components 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 and chemical composition of these two types of airborne matter are markedly different.

Entrained aerosols typically have a mass median diameter of  $\gg 1\ \mu\text{m}$  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 compounds. 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.

### 6.2.1 Aerosol Mass DFs

The melter's aerosol mass DFs, as measured by the filter catches of the differential sampler previously described, are tabulated in Table 6.3 for each of the distinct waste-loaded feeds processed during RSM testing. These melter aerosol mass DFs are fairly consistent and do not appear to have been significantly affected by the SBW waste-loading fraction. Their magnitudes, moreover, are consistent with previous small-scale melter flowsheet tests that proved successful when tested on a larger scale. Also listed in this table are the related, source-emission parameters of melter partitioning and off-gas aerosol loading. The internal consistency of these off-gas loading and DF data suggest that steady-state melter processing conditions were easily achieved and maintained for all SBW feeds tested.

It should be noted that isokinetic sampling conditions were not achieved during RSM particulate sampling. Specifically, extraction velocities were always much lower than the off-gas stream velocities sampled. The effect of this condition is to overestimate the off-gas stream's aerosol mass loading. Therefore, the bias introduced by this miss-matched condition acts to decrease apparent melter DFs and increase the apparent magnitude of effluent partitioning coefficients. Since the sampler will tend to collect a greater and unrepresentative proportion of larger particles, the elemental composition of the collected aerosols would overemphasize the importance of entrainment relative to the volatilization/condensation loss mechanism. The relative proportions of fuming to entrained effluents will be examined in the following section.

**Table 6.3. Gross Melter Aerosol Emission Characteristics**

Date/Time	Feed			Sampling		MOG Flw (scfm)	Aerosol Catch		Melter	
	Batch	SBW %	Rate (L/h)	Time (m)	Flw (slpm)		Mass	Con (mg/sl)	DF	Loss%
1/30/01 17:55	B	30	2.3	52	5	18.7	0.27	0.93	58	1.7
1/31/01 18:33	D	32	2.8	49	5.4	14.6	0.33	1.14	51	2.0
2/1/01 18:51	F	35	2.6	60	5.8	17.3	0.49	1.31	41	2.4

### 6.2.2 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 these filter compositional data with 35% SBW feed shown in Table 6.4 illustrates the unmistakable influence of volatility upon the melter-emission source term. For all SBW feeds processed, the concentration of all the classic semivolatiles (B, Cd, Cr, Ru alkali earths, etc.) are seen to be enriched by up to a factor of 5 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.

**Table 6.4. Composition of Melter Generated Aerosols and Melter Feed**

Element	Melter Aerosol Wt% Comp			Feed Wt%
	30% SBW	32% SBW	35% SBW	
Al <sub>2</sub> O <sub>3</sub>	---	---	---	8.970
B <sub>2</sub> O <sub>3</sub>	22.100	21.900	20.100	10.400
BaO	0.006	0.014	0.005	0.002
CaO	1.350	1.690	1.790	4.130
CdO	0.039	0.053	0.083	0.026
Ce <sub>2</sub> O <sub>3</sub>	0.002	0.002	0.001	0.002
Co <sub>2</sub> O <sub>3</sub>	---	---	---	---
Cr <sub>2</sub> O <sub>3</sub>	0.174	0.215	0.240	0.067
Cs <sub>2</sub> O	0.500	0.574	0.559	0.064
CuO	0.001	---	0.012	0.015
Fe <sub>2</sub> O <sub>3</sub>	0.217	0.395	0.201	7.290
Gd <sub>2</sub> O <sub>3</sub>	---	---	---	0.008
K <sub>2</sub> O	14.500	16.000	17.300	2.450
Li <sub>2</sub> O	5.290	5.680	5.190	3.420
MgO	---	---	---	0.128
MnO	0.041	0.043	0.045	0.264
MoO <sub>3</sub>	0.002	0.004	0.004	0.007
Na <sub>2</sub> O	47.200	44.800	38.900	16.900
NiO	---	0.001	---	0.029
P <sub>2</sub> O <sub>5</sub>	0.222	---	0.330	0.257
PbO	---	---	0.078	0.077
RuO <sub>2</sub>	0.065	0.068	0.092	0.004
SO <sub>3</sub>	8.280	8.560	15.100	1.140
SiO <sub>2</sub>	---	---	---	44.400
SrO	0.010	0.008	0.008	0.003
TiO <sub>2</sub>	---	---	---	0.001
ZnO	---	---	0.006	0.023
ZrO <sub>2</sub>	---	---	---	0.002

Using the compositional information provided in Table 6.4, melter DFs associated with aerosol loss for individual elements can be calculated. These derived DF values are tabulated in Table 6.5. These tabular results reinforce the previous discussion that predicted low DFs for feed constituents that are volatile, or can form volatile or semivolatile compounds at melter-processing temperatures.

**Table 6.5. Elemental Melter DFs Associated With Aerosol Emissions**

Element	Melter Aerosol DFs @ %SBW Loading			
	30%	32%	35%	Ave
Al	---	---	---	---
B	36	30	20	29
Ba	37	21	138	65
Ca	133	116	75	108
Cd	34	28	11	24
Ce	---	---	---	---
Co	---	---	---	---
Cr	20	19	13	17
Cs	5	5	3	5
Cu	---	---	15	8
Fe	2010	1210	1450	1560
Gd	---	---	---	---
K	9	9	7	8
Li	44	39	30	38
Mg	---	---	---	---
Mn	372	402	302	359
Mo	253	89	126	156
Na	20	24	20	21
Ni	---	---	---	---
P	16	---	12	14
Pb	---	---	51	51
Ru	8	10	6	8
S	8	9	4	7
Si	---	---	---	---
Sr	37	54	40	44
Ti	---	---	---	---
Zn	---	---	157	157
Zr	---	---	---	---

The reproducibility of melter-feed component DFs are, overall, quite good. The magnitudes of nonvolatile DFs, such as Fe, however, are atypically high and should not be interpreted as representing average melter-performance behavior. On the other hand, the low DFs recorded for some of the volatiles/semivolatiles previously discussed may be atypically low. In particular, Cs and Ru DFs appear to be much lower than might normally be expected. A value of 10 to 20 is a much more typical DF value for Cs; however, unique chemical effects associated with SBW processing could be responsible for enhancing cesium loss rates. Similarly, Ru DF values also seem artificially depressed given the level of reductant used during all phases of testing. Analytical uncertainty is likely the cause of the latter DF anomaly, as detection limits are being approached in most samples. The lack of agreement between actual and target feed/glass data also support this proposition. Additional testing will, in time, resolve this apparent cesium-loss-rate anomaly, but higher levels of Ru will be needed in subsequent testing if reliable melter-performance values for this noble metal is to be obtained.

### 6.2.3 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 6.5 also represent total melter DFs for these elements. However, notable exceptions to this statement include B, Cl, F, N, P, and S, whose gaseous forms usually dominate melter off-gas system losses and determine their melter DFs. Table 6.6 summarizes the composition of the (0°C) chemical gas-scrubber solutions used to condense and/or remove melter (gaseous) effluents penetrating the upstream aerosol filter of the differential sampling system previously described (see Fig. 3.4). With the exception of NO<sub>x</sub>, these data show little evidence of gas-phase effluents. Moreover, the systematics of the distributions of nitrate and nitrite ion concentrations in these sequential scrubber vessels suggests a melter NO<sub>x</sub> source exhibiting a high NO:NO<sub>2</sub> ratio. Because of this apparently high ratio, gas-scrubber collection of NO<sub>x</sub> was incomplete and nonquantitative as evidenced by the high nitrite concentrations in the final caustic scrubber bath.

The only other feed components that exhibit any sort of systematic presence in these gas scrubber solutions are boron and calcium. Boron, as mentioned earlier, is an expected gaseous melter effluent, but calcium and its 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 produce a much stronger systematic trend across the acid baths, which it does not.

**Table 6.6. Off-Gas Sampler Scrub Solution Composition (ppm)**

Element	30% SBW Gas Scrub				32% SBW Gas Scrub				35% SBW Gas Scrub			
	H <sub>2</sub> SO <sub>4</sub>		NaOH		H <sub>2</sub> SO <sub>4</sub>		NaOH		H <sub>2</sub> SO <sub>4</sub>		NaOH	
Al	---	---	---	---	---	---	---	---	---	---	---	---
B	0.2	---	---	---	0.17	---	---	---	0.1	---	---	---
Ba	---	---	---	---	---	---	---	---	---	---	---	---
Ca	0.399	0.219	0.081	0.011	0.159	0.004	---	---	0.509	0.339	---	---
Cd	---	---	---	---	---	---	---	---	---	---	---	---
Ce	---	---	2.8	---	---	---	---	---	---	---	---	---
Co	---	---	---	---	---	---	---	---	---	---	---	---
Cr	---	---	---	---	---	---	---	---	---	---	---	---
Cu	---	---	0.21	0.03	---	---	---	---	---	---	---	---
Fe	0.67	---	---	---	---	0.21	---	---	0.06	0.22	---	---
Gd	---	---	---	---	---	---	---	---	---	---	---	---
I	---	---	---	---	---	---	---	---	---	---	---	---
K	---	---	---	---	---	---	---	---	---	---	---	---
Li	---	---	---	---	---	---	---	---	---	---	---	---
Mg	0.01	---	0.006	---	---	0.01	---	---	---	0.01	---	---
Mn	---	---	---	---	---	---	---	---	---	---	---	---
Mo	---	---	---	---	---	---	---	---	---	---	---	---
Na	6.866	4.166	---	---	2.866	5.716	---	---	3.666	5.966	---	---
Ni	---	---	---	---	---	---	---	---	---	---	---	---
P	---	---	---	---	---	---	---	---	---	---	---	---
Pb	---	---	---	---	---	---	---	---	---	---	---	---
Ru	---	---	---	---	---	---	---	---	---	---	---	---
S	---	---	---	---	---	---	---	---	---	---	---	---
Si	0.16	0.09	0.26	0.08	0.09	---	0.027	0.021	0.05	0.21	0.025	0.024
Sr	---	---	---	---	---	---	---	---	---	---	---	---
Ti	---	---	---	---	---	---	---	---	---	---	---	---
Zn	---	---	0.07	---	---	0.01	---	---	---	0.01	---	---
Zr	0.07	0.04	---	---	---	0.6	---	---	---	---	---	---
F <sup>-</sup>	0.22	0.04	0.044	0.045	0.03	0.03	0.03	0.04	0.03	0.08	0.04	0.04
NO <sub>2</sub> <sup>-</sup>	---	---	3600	2400	---	---	4500	3000	---	---	5800	4600
NO <sub>3</sub> <sup>-</sup>	350	900	84	32	360	1670	94	130	760	3000	310	150

However, apart from the nature and source of the calcium in these scrub solutions, its cumulative mass in all cases is <10% of the corresponding calcium particulate yield. Indeed, this is also true for all elements detected above scrub-solution background levels, except, of course, for nitrogen.

Table 6.7 summarizes total elemental melter DFs obtained by combining the scrub-solution composition with its corresponding particulate catch. Comparison of these total values with corresponding particulate DFs previously discussed (Table 6.5) clearly illustrate the dominant role played by the aerosol loss mechanism. The fact that greater volatility losses for boron and halogenated compounds was not observed during RSM testing is due to the low post film-cooler off-gas temperatures (~100°C). Unquenched exhaust temperatures of larger melters are typically at least twice this value.

Estimates of total melter DFs can also be extracted from off-gas system waste stream concentration data, provided the volatiles are efficiently scrubbed and that the effluent steams can be representatively sampled. These estimates will be discussed in the following Section.

**Table 6.7. Total Individual Elemental Melter DF Values**

Element	Total Melter DFs @ %SBW Loading			
	30%	32%	35%	Ave
Al	---	---	---	---
B	35	30	20	29
Ba	37	21	138	65
Ca	120	114	71	102
Cd	34	28	11	24
Ce	---	---	---	---
Co	---	---	---	---
Cr	20	19	13	17
Cs	5	5	3	5
Cu	---	---	15	8
Fe	1220	1100	1230	1180
Gd	---	---	---	---
K	9	9	7	8
Li	44	39	30	38
Mg	---	---	---	---
Mn	372	402	302	359
Mo	253	89	126	156
Na	19	23	19	21
Ni	---	---	---	---
P	16	---	12	14

**Table 6.7 (contd)**

Element	Total Melter DFs @ %SBW Loading			
	30%	32%	35%	Ave
Pb	---	---	51	51
Ru	8	10	6	8
S	8	9	4	7
Si	---	---	---	---
Sr	37	54	40	44
Ti	---	---	---	---
Zn	82	789	133	335
Zr	---	---	---	---

#### 6.2.4 Process-Waste-Stream Composition

The RSM's EVS acts as a single-stage sampler for process aerosols and condensable and/or reactive effluent gases. Many chemically reactive and condensable gases are generated during LFCM processing. Boron, chlorine, fluorine, phosphorous, and sulfur-feed components are all volatilized to some extent during LFCM processing, and some of the volatile species are efficiently removed (physically and/or chemically) by the aqueous off-gas system quencher—in this case, the EVS. Consequently, rough melter DFs can be estimated from EVS effluent concentration data if all chemical forms escaping the process are soluble. Elements that form insoluble compounds are of no practical analytical value for melter DF determinations since they cannot be representatively sampled from the EVS' condensate tank.

The time-dependent composition of the filtered SBS scrubbing liquor during RSM testing is summarized in Table 6.8. If the concentration information is examined for species whose compounds are thought to be totally soluble in samples RSM-01-1-28 and RSM-01-1-32, the impact of the 1/31/01 melter overfeeding event upon scrubbing liquor composition can be clearly distinguished. Since the feed composition is known and the volume of the EVS' condensate tank was approximately 117 L (31 gal) at the time of the event, one can attempt to estimate the feed volume directly lost to this tank. Using the most significant of the nominally soluble elements, B, K, Na, and S, the feed volume inadvertently lost is estimated to be 1.04, 0.97, 0.91 and 1.22 L, respectively. Since the Na value had to be compensated for NaOH additions, and volatile sulfur contributions were likely significant during this event, the average value of 1 L based on the B and K values is probably the most accurate estimate of the feed-loss volume to the EVS' condensate tank.

Using this feed-transfer information along with initial condensate tank composition, tank volume, and chemical additions made to the tank during testing, the accumulations of soluble process effluents in the condensate tank can be estimated from which the melter DF's can be derived. Table 6.9 presents these DF approximations and compares them to the reference average values derived from the off-gas sampling campaigns. Although the relative trends for related groups of elements are similar in both sets of data, corresponding DF values do not agree well. This result is not surprising insofar as representative

sampling of the EVS waste stream is limited to elements whose compounds are 100% soluble. The apparent melter DF will increase for any element that partitions to EVS insolubles or is otherwise lost to the process off-gas system.

**Table 6.8. EVS Condensate Soluble Effluent Composition**

Element	EVS Scrubbing Liquor Effluent Concentrations (ppm)								
	RSM -1	RSM -22	RSM -28	RSM -32	RSM -42	RSM -65	RSM -74	RSM -81	RSM -91
Al	0.073	0.5	0.49	1	2.2	1.8	1.4	1.5	1.4
B	1.53	29.5	35	89	92	100	100	97	98
Ba	0.004	---	---	0.01	---	---	---	---	---
Ca	7.6	2.59	4.6	3.5	2.4	2.45	3.3	3.2	4.7
Cd	---	---	0.01	---	---	---	---	---	0.046
Ce	---	---	---	---	---	---	---	---	---
Co	---	0.025	0.024	0.04	---	---	---	---	---
Cr	0.011	0.55	0.73	0.73	0.93	1.3	1.4	1.4	1.4
Cs	0.064	2.85	3.46	5.59	6.21	7.52	8.48	8.90	9.10
Cu	0.016	9	9.8	7.6	7.96	9.05	9.4	9.4	10
Fe	---	0.023	0.044	0.0125	---	---	---	---	0.1
Gd	---	---	---	---	---	---	---	---	---
I	---	0.2	0.3	0.3	---	---	---	---	---
K	1.7	47	60	120	130	140	150	160	160
Li	0.15	8.3	10	31	31	34	35	35	35
Mg	0.008	1.2	1.3	1.5	0.71	0.74	1	1.1	1.2
Mn	---	---	0.01	0.026	---	0.024	---	---	---
Mo	0.01	0.16	0.19	0.27	0.31	0.385	0.44	0.42	0.41
Na	280	1240	1400	2060	2430	2765	2700	2700	2700
Ni	---	0.13	0.14	0.3	0.3	0.4	0.4	0.4	0.4
P	---	0.52	0.53	0.51	0.8	1.05	0.8	0.8	1.5
Pb	---	---	---	---	---	---	---	---	---
Ru	---	0.05	0.07	0.1	0.16	0.225	0.25	0.24	0.25
S	36.4	52	53	70	70	74	80	76	86
Si	2.28	2.35	2.2	0.67	0.77	0.82	0.79	0.72	0.87
Sr	0.13	0.088	0.15	0.19	0.17	0.17	0.17	0.15	0.15
Ti	---	---	---	---	---	---	---	---	---
Zn	---	0.01	0.02	0.01	---	---	---	---	---
Zr	---	0.6	0.07	---	---	0.36	---	---	---
F <sup>-</sup>	0.45	9.74	11.9	20.7	20.5	23.6	23.2	23.2	26
Cl <sup>-</sup>	9.4	6.4	7.5	115	120	130	150	140	140
NO <sub>2</sub> <sup>-</sup>	---	1300	1700	2050	1700	3300	3300	3500	3500
NO <sub>3</sub> <sup>-</sup>	5.8	99	130	1400	980	1340	1360	1300	1250
SO <sub>4</sub> <sup>=</sup>	120	150	160	172.5	140	180	200	210	210



**Table 6.9. EVS and Off-Gas Sampler Total Melter DF Values**

Element	Total DF	
	EVS	OG Smp
B	310	28
Cr	309	17
Cs	28	4
Cu	4	2
K	61	8
Li	1040	37
Ru	64	8
S	28	7
Sr	284	44

Sulfur provides a classic example of the latter partitioning situation where the high-sulfur DF value derived from the EVS solution data is due to the low-efficiency removal of the primary effluent form(s) of sulfur: SO<sub>2</sub>, SO<sub>3</sub> and/or H<sub>2</sub>SO<sub>4</sub>. And indeed, the composition of aqueous run off from the downstream HEME, summarized in Table 6.10, strongly confirms this supposition. These tabular data when compared with Table 6.8 show that although the total HEME runoff (7.5 L [2 gal]) volume is only ~4% of EVS' condensate volume (193 L [51 gal]), it contains 3x as much sulfur.

**Table 6.10. HEME Aqueous Run-Off Composition**

Element	HEME Sol. Conc. (ppm)
Al	128
B	3770
Ba	4.3
Ca	820
Cd	28
Ce	---
Co	---
Cr	100
Cs	643
Cu	58
Fe	265
Gd	---

**Table 6.10 (contd)**

<b>Element</b>	<b>HEME Sol. Conc. (ppm)</b>
I	---
K	600
Li	100
Mg	79
Mn	19
Mo	22
Na	2200
Ni	53
P	106
Pb	51
Ru	0.05
S	7500
Si	39
Sr	7.2
Ti	1.5
Zn	26
Zr	11.3

The reason for the high-efficiency removal of sulfur is that the HEME's extremely high surface area is continually soaked by the aqueous mist carryover from the EVS. The high aqueous/gas-phase contacting that results from the wetting of this high-surface-area fiber bed provides efficient gas scrubbing capabilities for acid gases such as  $\text{NO}_x$  and  $\text{SO}_x$ . The very high nitrate ( $\text{HNO}_3$ ) content of the HEME run-off ( $\text{pH} \ll 1$ ) is primarily due to  $\text{NO}_x$  removal. The sulfur effluents responsible for the  $\text{SO}_4^{=}$  content of the HEME waste stream are not, however, as easily identified, as was the case for the  $\text{NO}_3^-/\text{NO}_x$  pairing.

Clearly, the HEME is specifically designed to efficiently coalesce liquid aerosols (droplets), including those associated with  $\text{SO}_3$  and  $\text{H}_2\text{SO}_4$ . Since droplets of  $\text{SO}_3$  and  $\text{H}_2\text{SO}_4$  do not quickly dissolve in water, low and high removal efficiencies would be predicted for the EVS and HEME, respectively, which is precisely what is observed. But on the other hand,  $\text{SO}_2$  is capable of being oxidized by nitric acid, which is the major chemical constituent of the fluid wetting the HEME fibers. Consequently, a melter  $\text{SO}_2$  effluent source could also explain the high concentration of sulfate in the HEME waste stream. Of course, any combination of these sources is also consistent with process-waste-stream observations. Historically,  $\text{SO}_3/\text{H}_2\text{SO}_4$  off-gas emissions have been the dominant sulfur loss mechanism associated with non-boosted melter operations. It is hoped that gas chromatographic monitoring of  $\text{SO}_2$  during future RSM testing will help to further define the nature of the RSM's sulfur off-gas source term.

## 7.0 Byproducts, Residuals, 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 constituents representative of  $\text{Na}_2\text{SO}_4$  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,  $\text{Na}_2\text{SO}_4$  was found, in all cases, to represent ~80% of the molten salt's mass with most of the remaining mass, 15 Wt%, being accounted for by the alkali halides. The presence of chromium in the salt layer, ~1 Wt%, is not alarmingly high, although its very presence may be indicative of molten-salt-induced corrosion of the refractories and/or electrodes. Indeed, the relative weight fractions of chromium to sodium in the molten-salt phases sampled are 3.5 to 5 times higher than its corresponding ratio in the glass. However, no unusual corrosion of the melter's components was visually obvious at the conclusion of RSM testing.

### 7.2 Off-Gas Line Deposits

A significant buildup of process debris in the off-gas line connecting the melter to the EVS quench scrubber occurred during RSM testing. Although the POG's film cooler is designed to prevent the buildup and growth of off-gas line obstructions, the overfeeding event previously mentioned in 6.2.4 introduced raw feed into the melter's off-gas jumper that, upon drying, created significant pipe-wall deposits. Once formed, these pipe-wall irregularities will inevitably build upon themselves, despite the film-cooler, by collecting off-gas debris generated by the melter. Although the buildup of off-gas line deposits did not impact the RSM's 5-day processing schedule, over time, they would have had to be removed.

Table 7.3 summarizes the compositions of deposit samples taken from the melter view port, the film cooler wall, and EVS' entry port. Of the samples taken, the film-cooler deposit most resembled fused material. Deposits taken from the view port and EVS entry area are best described as a mixture of dried and fused feed material laced with semivolatiles and distinguishing high-sulfur values.

**Table 7.1. Composition of Dissolved Molten Salt Samples**

Element	Molten Salt Solution Composition							
	RSM-1-01-18		RSM-1-01-31		RSM-1-01-41		RSM-1-01-63	
	ppm	mEq/L	ppm	mEq/L	ppm	mEq/L	ppm	mEq/L
Al	---	---	---	---	---	---	1.6	0.2
B	18.0	5.0	21.0	5.8	21.0	5.8	19.0	5.3
Ba	0.2	---	0.2	---	0.2	---	---	---
Ca	34.0	1.7	22.0	1.1	22.0	1.1	0.1	---
Cl	210.0	5.9	87.0	2.5	120.0	3.4	220.0	6.2
Cr	24.0	0.9	17.0	0.7	45.0	1.7	55.0	2.1
Cs	9.1	0.1	6.6	---	12.1	0.1	13.1	0.1
Cu	0.5	---	0.3	---	---	---	---	---
F	16.3	0.9	12.6	0.7	14.6	0.8	26.4	1.4
Fe	0.2	---	---	---	---	---	---	---
K	400.0	10.2	260.0	6.6	510.0	13.0	400.0	10.2
Li	110.0	15.8	64.0	9.2	100.0	14.4	66.0	9.5
Mg	3.4	0.3	0.2	---	0.1	---	13.0	1.1
Mn	0.1	---	---	---	---	---	---	---
Mo	12.0	0.4	7.7	0.2	15.0	0.5	---	---
Na	1900.0	82.6	1260.0	54.8	2350.0	102.2	1700.0	73.9
P	6.3	0.6	3.1	0.3	9.0	0.9	3.4	0.3
S	1420.0	88.6	930.0	58.0	1770.0	110.4	1220.0	76.1
Si	5.3	0.8	2.3	0.3	2.6	0.4	2.8	0.4
Sr	1.5	---	0.8	---	0.9	---	---	---
Ti	0.7	---	---	---	---	---	---	---

Photographs taken of the film cooler, the off-gas jumper and the EVS inlet are displayed in Appendix H. All off-gas lines and components downstream of the EVS' inlet section were found to be clean and unobstructed.

**Table 7.2. Assumed Chemical Forms and Weight Contributions of Alkali-Earths**

Assumed Form	Wt% Contributions Of Constituents			
	RSM-18	RSM-31	RSM-41	RSM-63
Na <sub>2</sub> SO <sub>4</sub>	77.31	79.37	79.90	77.15
K <sub>2</sub> SO <sub>4</sub>	7.83	6.54	9.03	3.16
KCl	7.70	6.79	5.05	12.37
KF	---	1.43	0.89	2.15
CsCl	0.28	---	---	---
LiCl	1.57	---	---	---
LiF	0.48	---	---	---
Total (%)	95.17	94.13	94.88	94.83

**Table 7.3. Unnormalized Off-Gas Deposit Compositions**

Oxide	Equivalent Oxide Wt%			
	Vw Port	Flm Cool	EVS	Trgt Gls
Al <sub>2</sub> O <sub>3</sub>	3.820	8.110	1.990	8.930
B <sub>2</sub> O <sub>3</sub>	4.150	4.420	6.930	10.300
BaO	0.010	0.013	0.003	0.002
CaO	1.490	8.190	1.230	4.110
CdO	0.014	0.169	0.041	0.026
Ce <sub>2</sub> O <sub>3</sub>	---	---	---	0.002
Co <sub>2</sub> O <sub>3</sub>	---	---	---	---
Cr <sub>2</sub> O <sub>3</sub>	0.087	0.179	0.105	0.067
Cs <sub>2</sub> O	0.037	0.171	0.205	0.064
CuO	---	0.010	0.003	0.015
Fe <sub>2</sub> O <sub>3</sub>	3.030	8.460	1.540	7.260
Gd <sub>2</sub> O <sub>3</sub>	---	---	---	0.008
K <sub>2</sub> O	1.990	1.700	6.080	2.430
Li <sub>2</sub> O	1.570	1.360	2.020	3.400
MgO	0.082	0.335	0.058	0.128
MnO	0.126	0.318	0.077	0.263
MoO <sub>3</sub>	---	---	0.026	0.007
Na <sub>2</sub> O	10.300	9.940	21.200	16.800
NiO	0.035	0.089	0.023	0.029
P <sub>2</sub> O <sub>5</sub>	---	0.331	---	0.256
PbO	0.058	0.231	0.046	0.077

**Table 7.3 (contd)**

<b>Oxide</b>	<b>Equivalent Oxide Wt%</b>			
	<b>Vw Port</b>	<b>Flm Cool</b>	<b>EVS</b>	<b>Trgt Gls</b>
RuO <sub>2</sub>	0.029	0.049	0.026	0.004
SO <sub>3</sub>	3.600	0.983	5.610	1.140
SiO <sub>2</sub>	17.700	45.100	8.690	44.200
SrO	0.043	0.023	0.006	0.003
TiO <sub>2</sub>	0.014	0.063	---	0.001
ZnO	0.009	0.042	0.009	0.023
ZrO <sub>2</sub>	0.046	---	---	0.002
<b>Total</b>	<b>48.2</b>	<b>90.3</b>	<b>55.9</b>	

### 7.3 Process Statistics

During the January 2001 melter-flowsheet evaluation studies, 168 L (44 gal) of SBW simulated waste having a total mass of 210 kg were processed by the RSM producing 22 L (5.8 gal) of glass having a total mass of 57 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<sub>2</sub>O and NO<sub>3</sub>) are nonvitrifiable, volatile species. On the other hand, since most of the hazardous and rad-waste SBW components can be incorporated and immobilized in the melter's vitreous product and tank waste volumes of SBW are a physical reality, volume reduction has important waste-disposal implications. During the current RSM test, an overall SBW waste-volume reduction factor of 7.6 was achieved.

### 7.4 Sulfur Balance

As discussed in the previous section, 167 L (44 gal) of SBW simulant having an equivalent elemental sulfur concentration of 1.635 g/L were fed to the melter, which produced 57 kg of glass having an average elemental sulfur loading of 0.265 Wt%. Off-gas system losses of sulfur were estimated from the concentrations of the soluble sulfur, 86 ppm and 7500 ppm, in the 193 L (51 gal) of EVS condensate and in the 7.5 L (2 gal) of HEME aqueous runoff, respectively. If the following sulfur residuals are neglected,

- the molten-salt-phase mass remaining (as glass inclusions) within the melter
- the feed deposits lining the walls of the off-gas jumper
- the insoluble forms of sulfur in the EVS condensate tank
- the aqueous inventory remaining within the HEME's fiber bed

then 80% of the sulfur fed to the melter can be accounted for by glass, EVS, and HEME process streams.

The ~55 g of sulfur that is unaccounted for can be used to estimate an upper bound to the average concentration of SO<sub>2</sub> gas that could have escaped detection by being released to the environment. If a

15-scfm noncondensable off-gas flow rate is assumed over an effective 4-day, steady-state processing period, the maximum possible SO<sub>2</sub> concentration is calculated to be <20 ppm. Previous melter tests have shown SO<sub>2</sub> emission rates to be very low (~ 1 ppm) when plenum temperatures are <600°C. Since the RSM's plenum temperature conforms with this low-temperature condition, it would be surprising if residual undetected sulfur residing on the HEME fiber bed and/or in EVS insoluble material could not account for a large fraction of the missing sulfur mass.

## 8.0 Conclusions and Recommendations

The results obtained from SBW melter-flowsheet evaluations have demonstrated that the processing characteristics of baseline melter-feed formulation for 30%, 32%, and 35% waste loadings are more than adequate and that the vitreous product formed is sufficiently durable to comply with all existing RCRA land-disposal limits. Moreover, area-normalized glass-production rates and specific process energy requirements for the SBW flowsheet are found, overall, to be in reasonably good agreement with generalized expectations for slurry-fed, Joule-heated ceramic melters.

Determining the fate of feed-component sulfur was an important objective during and after melter testing. Accessing melter accumulations of molten  $\text{Na}_2\text{SO}_4$  was an ongoing activity conducted during all phases of testing, as was  $\text{SO}_2$  off-gas monitoring. Post-test analyses of all process waste streams were used to help establish a mass balance for sulfur around the melter. The conclusions and results obtained from these evaluations are summarized by the following observations:

- A molten-salt phase was present for all waste loadings tested (30, 32, and 35 Wt%), but it never formed a contiguous surface layer.
- At the reference reductant (sugar) loading of 135 g/L-SBW, the inventory of molten salt noticeably increased when processing 35% SBW feeds.
- Increased reductant levels mitigated molten-salt accumulations at higher waste loadings, but it also affected the oxidation state of the product glass.
- 55% of the sulfur fed to the melter was incorporated within the melter's glass product, and 26% was condensed/collected as soluble sulfates by the RSM's off-gas system.
- Residual, uncollected  $\text{H}_2\text{SO}_4$  residing on the HEME fiber bed and/or unanalyzed insoluble sulfate material in EVS' condensate tank are believed to account for much of the remaining sulfur (~20%), as  $\text{SO}_2$  generation rates in nonboosted, cold-lid melters are usually quite low.
- If  $\text{SO}_2$  environmental releases are assumed responsible for all the unaccounted for sulfur, the average noncondensable off-gas concentration of this gas would have been <20 ppm, which is significantly less than the detection limit of the online instrument that failed to detect this effluent during RSM testing.

Given the importance of S in the SBW flowsheet, a dedicated, high-sensitivity  $\text{SO}_2$  monitor should be used in subsequent tests to help resolve ambiguities associated with sulfur off-gas behavior. Furthermore, since increased SBW reductant loading appears to have been effective in controlling molten salt accumulations when feed containing 140% of the baseline sulfur content was processed, a careful parametric study needs to be conducted to establish the reductant level that optimizes sulfate reduction while not overly reducing the melter's glass product.



Off-gas measurements revealed little evidence for the presence of gas-phase compounds of B, Cl, F, P and S due presumably to low ( $\sim 100^{\circ}\text{C}$ ) unquenched off-gas temperatures. This situation is not representative of full-scale operating conditions where temperatures exceeding  $200^{\circ}\text{C}$  are common; consequently, lower film-cooler injection rates should be considered when future RSM tests are conducted as the fate and behavior of reactive/condensable gases were not being properly represented.

Since only a very few feed components are typically lost to the off-gas processing system in the gaseous state, the low off-gas temperatures had little impact upon the majority of waste/feed constituents. Consequently, melter aerosol mass DFs ( $\sim 50$ ) were found to be consistent with previous small-scale melter flowsheet tests and did not appear to have been significantly affected by the SBW waste loading fraction. This was also true for most individual melter-feed component DFs, since aerosol losses normally dominate the melter source term.

However, atypically high off-gas partitioning coefficients were recorded for the radiologically important semivolatiles Cs and Ru. Although additional testing will resolve this apparent cesium loss rate anomaly, higher levels of Ru are recommended in subsequent RSM tests if reliable melter-performance values for this element is to be obtained. In addition, the method of preparing the aerosol samples for analysis (digestion with the quartz filter media) should be reexamined as it significantly reduced sensitivities for detecting minor effluent constituents and eliminates the possibilities of measuring trace constituents, such as the halogens.

## **Appendix A**

### **Test Plan for Vitrification Demonstration Tests of INEEL Sodium-Bearing Waste**

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

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

## **1. 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) 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 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 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 1 million gallons of SBW that was not calcined. The SBW is an aqueous, highly acidic (1-3 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. 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 tank farm facility 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 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 was selected by DOE in fiscal year 2000 as the preferred method. Beginning in fiscal year 2000, the INEEL High Level Waste (HLW) program embarked on a program for technology demonstration and development that will lead to conceptual design of a vitrification facility for the SBW. This program includes several separate activities that include, among others, demonstration testing of nonradioactive surrogate SBW in pilot facilities.

Demonstration tests are currently planned using two existing, nonradioactive pilot-scale melter test facilities. Small -scale tests will be performed using the Research Scale Melter (RSM) located at the Applied Process Engineering Laboratory (APEL) facility in Richland, Washington. Larger, pilot-scale tests are also planned using the EV-16 melter located at the Clemson Environmental Technologies Laboratory (CETL) at Clemson University in Anderson, South Carolina. Laboratory crucible tests are also included prior to and simultaneously with planned 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 RSM test series planned for late January 2001. A second RSM test series is planned for later in 2001.

## 2. OBJECTIVES

The objectives of the first RSM test series, in order of priority, are summarized as follows:

1. Determine feasibility of vitrifying surrogate SBW without pretreatment to produce a regulatorily acceptable borosilicate glass waste form.
2. Characterize the product glass, melter offgas, and particulate matter (PM, material volatilized or otherwise entrained in the offgas). This characterization includes determination of (a) amounts and elemental compositions of all products, (b) the fate of certain feed components (such as radionuclide surrogates, heavy metals, glass formers, carbon, nitrates, halogens, and sulfur), and (c) leachability (based on Product Consistency Test, PCT) and oxidation state (based on  $\text{Fe}^{+2}/\text{Fe}^{\text{total}}$  ratio) of the product glass, and (d) properties of the offgas and particulate matter.
3. Determine a maximum process rate based on stable operation at a maximum surrogate SBW loading and maximum feedrate.
4. Determine if the melt rate or melter processing conditions can be optimized by changing melter-operating conditions.
5. Determine surrogate SBW volume and mass reduction.
6. Determine power requirements versus surrogate SBW mass processed and versus glass produced.

Objective 1 is to verify the feasibility of SBW vitrification via direct slurry feeding of a surrogate SBW with added glass formers and an organic reducing agent. The added glass formers are used to produce an acceptable borosilicate glass waste form for the melter product. Glass waste form acceptability will be based on results of glass characterization summarized in Objective 2.

A reducing agent is expected to be needed to control the oxidation states of multivalent glass constituents, aid in the denitration of the feed, and to increase melt rate by reducing such conditions as melt foaming. Reductant may also help prevent formation or persistence of sulfate salts in the melter. Sucrose will be the first reductant that will be tested. Based on results of crucible tests and initial pilot tests, other reductants or varying concentrations of reductants may also be tested in the second RSM test series. This will demonstrate the ability to perform feed evaporation and denitration steps along with vitrification in the melter, thereby eliminating the need for any SBW pre-treatment technologies prior to vitrification.

The feasibility of vitrifying the surrogate SBW will be determined by establishing continuous surrogate SBW feeding at the maximum surrogate waste loading and feedrate, based on melter operating conditions such as cold cap, while avoiding the generation of sulfate salts in the melter in amounts that could impair melter performance. Establishing a maximum waste loading and feedrate will determine a maximum waste-processing rate and accomplish Objective 3.

To satisfy Objective 4, flowsheet reductant concentration will be varied to examine its influence upon feed rheology, melter processing rates, melt oxidation state and process off-gas conditions.

Objectives 5 and 6 are accomplished by recording and comparing the melter product masses and the melter power consumption to the surrogate waste feedrate and loading.

Not included in the scope of the first RSM test are:

- Determinations of offgas system or technology performance (beyond determinations used for waste characterization and mass balances)
- Determination the fate of Hg during the vitrification process (no Hg will be added in the feed)
- Evaluations of different reductants besides sucrose
- Measurements of sulfur and chlorine species in the offgas except as provided by the continuously operated mass spectrometer, which can quantify those species only to the extent that other species with identical masses are not also present in the offgas
- 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 of filter/bulk PM sample fractions)
- Particulate/metal sample train collection and analysis in excess of 3 separate sample trains
- Evaluations with sample collection and analysis to determine PM emissions for different melter conditions such as reduced cold cap.

### 3. PROJECT ORGANIZATION AND SCHEDULE

The RSM test program will be performed primarily by personnel from Pacific Northwest National Laboratory (PNNL) and INEEL. Project activities and performing organizations are listed as follows:

- INEEL: Provide (a) project definition and test objectives, (b) SBW composition and determination of reductant, (c) test plan preparation support, (d) project schedule definition, (e) onsite test team support of at least 1 engineer or technician on all shifts during testing, (f) scanning electron micrograph (SEM) analysis of PM samples, and (g) test report review and comment.
- PNNL (Battelle): Provide responsibility for the performance of the test, including (a) test facility (RSM system) and support infrastructure (utilities, electrician, mechanic, machine shop, etc), (b) feed procurement and preparation, (c) facility and test plan preparations for the test series, (d) sampling and analysis equipment preparation and operation, (e) all materials including sample containers, reagents, calibration gases, etc. associated with operating the RSM and collecting samples, (f) test team direction during the test series, (g) all sample analysis (except for SEM analyses performed at the INEEL), (h) all data reduction except for what can be done during test operation by INEEL personnel, and (i) draft and final test reports.

An approximate schedule is shown in Table 1. As this schedule shows, the soonest that draft report information could be available to help in planning subsequent EV-16 pilot tests is about March 16, 2001. These activities are all on the critical path for project completion. Any delays in these activities could extend this schedule beyond the estimated dates.

<b>Table 1. RSM Test Program Schedule</b>			
Activity	Start date	Completion date	Performer
1. Draft test plan	Dec 11, 2000	Dec 22, 2000	INEEL & PNNL
2. Procure feed components	Dec 2000	Jan 19, 2001	PNNL
3. Finalize test plan	---	Jan 19	INEEL & PNNL
4. Prepare test facility including preparation of RSM system, feed system, and sample collection and analysis equipment	Dec 2000	Jan 26	PNNL
5. Prepare feed mixture(s)	Jan 22	Jan 26	PNNL
6. Perform pretest shakedown	Jan 22	Jan 26	PNNL
7. Conduct test	Jan 29	Feb 2	PNNL w/ INEEL
8. Post-test inspections, facility cleanup, sample collection, and sample delivery to laboratory(ies)	Feb 2	Feb 9	PNNL
9. Laboratory analysis	Feb 9	Feb 23	PNNL (& INEEL for SEM analysis)
10. Continuous process monitor and continuous offgas monitor data reduction	Feb 5	Feb 23	PNNL
11. Laboratory data reduction	Feb 23	Mar 9	PNNL
12. Data evaluation and draft test report	Feb 23	Mar 16	PNNL
13. Project internal review and comment	Mar 16	Mar 30	PNNL & INEEL
14. Final report preparation/DOE review	Mar 30	Apr 13	PNNL
15. External report preparation/issuance	Apr 13	Apr 30	PNNL



## 4. TEST PROGRAM

The January 2001 test series is limited to 96 hours of continuous, 24 hr/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. The ability of this test series to accomplish all test objectives depends on how rapidly Objective 1 is satisfied. Establishing stable operation with surrogate SBW waste may require some trial and error operation. At a given set of operating conditions, some operating time is needed to (a) detect changes in some potentially slow-responding 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 4.5 hours of operation and tapping after a feed composition change to purge one volume of the old bath composition. Up to 3-bath volume changes (approximately 12 hours) are needed to approach a new composition equilibrium.

**Table 2. Target test matrix for the January 2001 RSM test series.**

Objective	Waste Loading, %	Feedrate	Reductant	Duration	Cumulative hours	Data/Sample Collection	Comments
Vitrification feasibility	A (a)	1.5 l/hr (b)	X (c)	24	24	Routine data recording; collect all samples & PM/metal train 1 at stable condition	Assuming that offgas measurements are continuous and relatively rapid, and that the response of the offgas to a step feed change is relatively rapid, extra time for feeding is not expected for offgas measurements except for the sample trains.
Waste loading maximization	B (d)	Highest optimum		24	48	Routine data; all samples & PM/metal train 2 at stable condition	
	C (d)	Highest optimum		24	72		
Reductant optimization	Highest optimum	Highest optimum	Y(c) (≠X)	12	84	Routine data; all samples & PM/metal train 3 at stable condition	
	Highest optimum	Highest optimum	Z(c) (≠X,Y)	12	96		
a. % of surrogate SBW in the feed mixture, 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 may be between 20-35%.							
b. The feedrate may be adjusted if necessary to try to achieve an optimum 90-95% cold cap.							
c. Ratio of reductant to nitrates in the surrogate SBW. Determined from crucible tests to be adequate to reduce the feed nitrates, reduce melt foaming, and help reduce any sulfate phase. This may need to vary during the vitrification feasibility determinations if the melt becomes too reduced (as determined by measured $Fe^{+2}/Fe^{total}$ ratios) or if there is too much melt foaming.							
d. Incrementally increased, if possible, to attempt to increase the melt rate and overall waste loading in the glass.							

If some conditions such as the generation of a separate molten sulfate salt phase occur, some time may be required to take corrective activities. Such corrective activities 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) temporarily decreasing the amount of sulfur in the feed and allowing time to 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, offgas, 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 extend to at least 6 hours.

## 4.1 Determine feasibility of vitrifying surrogate SBW

At the start of the test series, a slurry of the SBW surrogate, glass formers, and reductant (sucrose) will be fed to the RSM melter to verify the glass formulation. The feed mixture recipe will be based on determinations of reductant concentration and waste loading determined previously during crucible tests at PNNL. Target operating conditions for the melter system are shown in Table 3. The target melt temperature is 1,150°C, and the target feedrate is 1.5 l/hr. As shown in Table 2, this first feasibility evaluation may require 24 hours or more of operation (at least 3 melt turnovers, and added time for adjustments), depending on how many feedrate, temperature, or other adjustments or corrective actions are needed to enable operation at or near the target operating conditions. When stable and acceptable conditions are established, samples of the glass, offgas, offgas particulate/metals, and offgas system secondary wastes will be collected for analysis to satisfy Objective 2. Monitoring and recording of other operating conditions such as tap rates and power levels will satisfy Objectives 5 and 6.

Test conditions after the feasibility determination will be based on results of the feasibility determination. If the test leader determines that the waste loading can be increased above the loading established in the feasibility determination, time is allowed (48 hours) in Table 2 to incrementally increase the waste loading to determine a maximum loading that still provides acceptable system conditions. The waste loadings for the subsequent test iterations will be increased based on results of the previous iterations. If sulfate salts are not present following the previous test, the waste loading will be increased until a separate sulfate salt layer becomes apparent (providing values for other operating parameters are within acceptable ranges).

**Table 3. Target RSM operating conditions for the January 2001 test series.**

<b>Parameter</b>	<b>Target</b>
Feedrate, liters/hr	1.5
Feedrate, kg/hr (a)	2.0
Feed oxides, kg/liter	0.45
Glass melt temperature, °C	1,150
Glass melt volume, liters	1.4
Glass melt mass, kg (b)	3.6
Glass production rate, kg/hr	0.7
Glass production rate, kg/m <sup>2</sup> /day	890
Glass Fe <sup>+2</sup> /Fe <sup>total</sup> ratio	<0.3
Plenum temperature, °C	600
Plenum pressure, inches water	-0.5 to -1.5
Air inleakage rate, scfm	~1.0 (estimated)
Total offgas flowrate, liters/minute	173
Plenum residence time, sec	1.6
a. Assuming a feed mixture specific gravity of 1.34.	
b. Based on a glass specific gravity of 2.6.	

## **4.2 Waste Loading Maximization**

During the waste loading maximization tests, the reductant concentration will change in proportion with the surrogate SBW, so that the ratio of reductant to nitrates and other species in the surrogate waste stays constant. If/when a sulfate salt phase begins to form, corrective actions will be implemented to remove this phase. When stable operation is established at maximum waste loading and feedrate conditions, sample and data collection including offgas composition and particulate/metals will provide data to satisfy Objectives 2, 3, 5, and 6.

## **4.3 Reductant Optimization**

Following the maximum waste loading determination, and assuming adequate remaining time, additional test(s) will be conducted to optimize melter performance by changing the amount of reductant while maintaining (or increasing) the maximum melt rate. Decreasing the amount of reductant may be considered to optimize the melt oxidation state or reduce levels of products of incomplete oxidation in the melter offgas. Increasing the amount of reductant may be considered to improve nitrate reduction and melt rate, or improve the ability to prevent sulfate phase formation. 24 operating hours are available in Table 2 for this activity. If a new stable operating condition results from this activity, sample collection will be done to provide data for satisfying Objectives 2-6.

## 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 a data acquisition and control system that monitors and controls key system components and electronically logs key data. Process data that is not electronically logged by this system must be recorded manually on operator data sheets.

Table 4 shows process data that is electronically logged by the data logging system and manually logged on operator data sheets. These data document data about melter conditions, electrode power, the kiln that encloses the melter, glass tapping, and the offgas system.

Visual observations of the operating behavior of the feed system and the melter will be very important during these trials. Any foaming, corrosion, or salt formation will be noted. Accurate records of feed rate will be made so reaction times and associated glass oxidation conditions can be documented. Any operational problems or potential optimizations will also be recorded.

At the completion of each test, the melter 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 4. RSM process data that is 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 LRB	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 4. RSM process data that is electronically or manually logged.**

Parameter	Units	Range	Electronic log	Manual log
Melt Resistance	Ω		X	X
Melt (Electrode) Setpoint Temp	° C		X	X
Parameter	Units	Range	Electronic log	Manual log
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	scfm	reg OGT (1-10)	---	X
Melter Vacuum-Magnehelic	in. H2O	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 for this test series include the feed slurry, glass product, accretions of material on the inside of the melter plenum or offgas pipe (if present), and scrubber solution. Table 6 shows these sample matrices, frequency of sample collection, and planned analyses. In general, process samples (except for plenum or offgas 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.

Glass sample analysis will include not only the elemental composition, but also leachability, density, crystalline morphology, and viscosity. Leachability will be measured using both the Product Consistency Test (PCT), ASTM C-1285-94<sup>[1]</sup>, and the Toxicity Characteristic Leaching Procedure (TCLP). 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.

Beyond compositional characteristics of the glass product, a limited PNNL budget (~\$4K) remains for conducting morphology, viscosity and regulatory-based tests mentioned above. Consequently, PNNL evaluations of noncompositional properties of glasses will, by necessity, be limited unless additional analytical scope is authorized. Otherwise, samples will be transmitted to INEEL for characterization.

Table 6. Process sample collection and analysis for the RSM January 2001 test series.				
Sample Matrix	Size	Frequency	Analysis	Comments
Glass product	≥200 g?	During each pour (~ every 1 to 4 hours) or every two hours whichever is less frequent.	Elemental (a)	Full metals and other analyses may only be done on those glass samples that represent test conditions that satisfy test objectives; full sample analysis will not be done on all collected glass samples.
			Fe ratio	
			PCT leachability	
			TCLP leachability	
			Density	
			Crystalline morphology	
	Viscosity			
Plenum or offgas pipe accretions	≥200 g	At end of test series or when system is shut down and opened up	Elemental (a)	
Scrubber solution	≥50 ml	Once per shift and/or at the conclusion of every test condition considered to be stable and satisfactory of test objectives	Elemental (a)	
a. Elemental analysis includes 25 elements – 7 RCRA metals (Ag, Ba, Be, Cd, Cr, Pb, Se), 10 glass formers (Al, B, Ca, Fe, K, Li, Mg, Mn, Na, and Si), 2 nonradioactive surrogates of radioactive elements (Ce and Cs), 3 halogens (Cl, F and I), and P, S, and C.				

### 5.3 Offgas Sample Collection and Analysis

Offgas sampling and analysis will include online, practically online, and off-line measurements. True online measurements include offgas temperature and NO, NO<sub>2</sub>, and total hydrocarbon (THC) measurements. Measurements of the offgas 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 ( $\leq 75$ s) of sample collection, and so are still as useful as online measurements.

Table 7 shows the offgas and particulate/metals sampling and analysis for this test series. All sampling will be done near the outlet of the melter, just downstream of the film cooler, but upstream of the offgas processing system. These measurements, when corrected for dilution from the film cooler gas, will best represent the actual uncontrolled melter emissions for melter mass balance evaluations and offgas system design.

The Stanford Research System's 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<sub>2</sub> (with molecular weight of 28) and CO<sub>2</sub> and N<sub>2</sub>O (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.

**Table 7. Melter offgas sample collection and analysis for the RSM January 2001 test series.**

Offgas Measurement (a)	Analytical instrument/ no. of samples (b)			Comments
	GC (c)	MS	Other	
Offgas flowrate/ Velocity	X (He)	X		Use He fed into the melter plenum as a tie element (tracer); subtract film cooler gas flowrate
% sample gas dilution	X (Ar)	X		Use Ar in the dilution gas supply as a tie element (tracer) to calculate sample gas dilution
O <sub>2</sub>	X	X		
CO <sub>2</sub>	X			
CO	X			
N <sub>2</sub>	X			
NO			X	Chemiluminescent analyzer
NO <sub>2</sub>			X	Chemiluminescent analyzer
N <sub>2</sub> O	X			Which column/detector?
HNO <sub>3</sub>		X		Sampling temp below BP/moisture reactive, data may not be valid
SO <sub>2</sub>		X		
COS		X		
CS <sub>2</sub>		X		Sampling temp below BP
H <sub>2</sub> S		X		
SO <sub>3</sub>		X		Sampling temp below BP
THC			X	FID, Total Hydrocarbon Analyzer
CH <sub>4</sub>		X	X	FID
VOCs		X		Specific species need to be identified
H <sub>2</sub>	X			
HCl		X		Moisture reactive
Cl <sub>2</sub>		X		
HF		X		
F <sub>2</sub>		X		
PM and metals (d)			3 trains total	Single-point, isokinetic Method 5 sample train; filtration without size discrimination; INEEL will do SEM analysis of aliquots of filter.
<p>a. Off-gas measurements not included or deferred to later RSM or EV-16 tests include HCl/Cl<sub>2</sub>/F/nitrates sample train, HCl &amp; SO<sub>2</sub> continuous monitoring, sulfur species sample train (total reduced sulfur, TRS sample train), and cascade impactor particle sizing, and all Hg feed and analysis. To the extent that the MS analysis can provide measurements of sulfur species SO<sub>2</sub>, H<sub>2</sub>S, COS, and CS<sub>2</sub>, Cl species HCl and Cl<sub>2</sub>, F species HF and F<sub>2</sub> and HNO<sub>3</sub> these will be reported.</p> <p>b. GC = Microtechnologies Incorporated (MTI) gas chromatograph calibrated for the specified species. The GC measurements are continuous only when the GC sample system and GC are online. MS = mass spectrometer calibrated for quantitative analysis based on height of peaks that are qualitatively identified based on molecular weight.</p> <p>c. The GC has two separate columns and detectors. He is a carrier gas for one column/detector for measuring CO<sub>2</sub> and N<sub>2</sub>O. Ar is the carrier gas for the other column/detector for measuring H<sub>2</sub>, He, N<sub>2</sub>, CO NO, and O<sub>2</sub>.</p> <p>d. Elemental analysis includes 25 elements – 7 RCRA metals (Ag, Ba, Be, Cd, Cr, Pb, Se), 10 glass formers (Al, B, Ca, Fe, K, Li, Mg, Mn, Na, and Si), 2 nonradioactive surrogates of radioactive elements (Ce, and Cs), 3 halogens (Cl, F and I), and P, S, and C.</p>				



## 5.4 Sample Analysis Procedures and Equipment

Process and offgas 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 8 shows the briefly describes the different analyses that will be included in this test program.

Table 8. Sample analysis methods used in the January 2001 RSM test series.					
Analysis	Sample Matrix	Sampling/Analysis Method*	Detection limits	Sample Preparation (solids)	Analysis Description
Cations	Solid or liquid	EPA 6010B, Inductively-coupled argon plasma atomic emission spectroscopy (ICP-AES). AA for Cs	Variable	Na fusion followed by total digestion (EPA 3050)	Analysis of total amount of element, regardless of speciation
			Variable	K fusion followed by total digestion (EPA 3050)	
Anions		IC	≥1ppm	Leachate (solids)	Cl, F, I, S and P
Organic		TOC	TBD	Feed samples only	Combustion, CO <sub>2</sub> detection
Leach	Glass	TCLP, PCT, ASTM C-1285-94,	By Reg	Crush and size-segregate	By PNNL if requested, otherwise by INEEL.
Particulate matter (PM)	Method 5/0060 probe & filter catches	EPA 5	1 mg/dscm	Dry per EPA 5	Gravimetric
PM size distribution		Scanning electron micrograph (SEM)	0.1 micron	Mount on stages, coat with gold	Visually count particles and estimate size in SEM fields magnified up to 10,000x
O <sub>2</sub> , CO <sub>2</sub> , CO, and Ar H <sub>2</sub> and He	Gaseous Effluents	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, drying, and filtration; injection of discrete <1 ml sample aliquots into GC	
NO, NO <sub>2</sub>		EPA 7E		Filter, dilute and cool.	Rosemount continuous emissions monitoring (CEM) using chemiluminescence
THC		EPA 25A		Filter dilute keep warm	CEM using flame ionization detection (FID)
Gaseous species <300 molecular weight		quadrupole mass spectrometer		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.	

\*Analytical/sampling approaches identified, as applicable, to similar EPA methodologies.

## 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 (sucrose).

### 6.1 SBW Description

Liquid SBW contained in the INTEC TFF 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 and potassium, some toxic metals, and radionuclides. Due to its relatively high concentration of sodium, this waste is referred to as sodium-bearing waste (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 or 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 as closely as reasonably possible. Table 9 shows the target composition of the surrogate SBW, based on the chemical composition of the actual SBW contained in INTEC TFF tank WM-180 <sup>[2]</sup>. A procedure for preparing the surrogate based on specific compounds and sequences of mixing that will enable the different components to become dissolved in the acid matrix was also recommended in this reference.

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 in the January RSM Test are summarized below.

Radio Nuclide	Stable / Surrogate
H-3	X
CO60	---
Sr-90	---
TC99	---
I129	X
CS134	X
CS137	X
EU154	X (Ce?)
U234	---
U235	---
U236	---
U238	---
NP237	---
PU238	---
PU239	---
AM241	---

The surrogate recipe does not include Hg to avoid system contamination and increased administrative and engineered protection against Hg environmental releases and worker exposure. Other elements that are in the SBW but that are excluded from the surrogate recipe are indicated in Table 9.

**Table 9. INTEC TFF Tank WM-180 Waste and Simulant Compositions**

INEEL Waste Definition*		PNNL Simulant	Comment
Element	Reagent		
Aluminum	$\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	X	---
Arsenic	$\text{As}_2\text{O}_3$	---	Chem hazard, excluded, no impact on glass product
Barium	$\text{Ba}(\text{NO}_3)_2$	X	---
Beryllium	$\text{BeF}_2$	---	Chem hazard, excluded, no impact on glass product
Boron	$\text{H}_3\text{BO}_3$	X	---
Cadmium	$\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$	X	---
Calcium	$\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$	X	---
Cerium	$\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$	X	---
Cesium	---	X	Added as $\text{CsNO}_3$
Chromium	$\text{Cr}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$	X	---
Cobalt	$\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	X	---
Copper	$\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$	X	---
Gadolinium	$\text{Gd}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$	X	---
Iron	$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	X	---
Lead	$\text{Pb}(\text{NO}_3)_2$	X	---
Lithium	$\text{LiNO}_3$	X	---
Magnesium	$\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	X	---
Manganese	$\text{Mn}(\text{NO}_3)_2$	X	---
Mercury	$\text{Hg}(\text{NO}_3)_2 \cdot \text{H}_2\text{O}$	---	Chem hazard, excluded, no impact on glass product
Molybdenum	Mo in $\text{HNO}_3$	X	$\text{H}_2\text{MoO}_4$ used.
Nickel	$\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	X	---
Potassium	$\text{KNO}_3$	X	---
Ruthenium	$\text{RuCl}_3$	X	---
Sodium	$\text{NaNO}_3$	X	---
Strontium	$\text{Sr}(\text{NO}_3)_2$	X	---
Titanium	$\text{TiCl}_4$	X	---
Uranium	$\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	---	No available nonradioactive surrogate
Zinc	$\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	X	---
Zirconium	$\text{ZrF}_4$	X	---
Chloride	$\text{HCl}$	X	---
Fluoride	$\text{HF}$	X	---
Iodide	$\text{KI}$	X	---
Nitrate	$\text{HNO}_3$	X	---
Phosphate	$\text{H}_3\text{PO}_4$	X	---
Sulfate	$\text{H}_2\text{SO}_4$	X	---

\* Unstable elements of insignificant mass not included.

## 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 10.

**Table 10. Target Glass Composition.**

Oxide	Wt%
B <sub>2</sub> O <sub>3</sub>	10.53
CaO	4.13
Fe <sub>2</sub> O <sub>3</sub>	7.37
Li <sub>2</sub> O	3.47
Na <sub>2</sub> O	15.64
SiO <sub>2</sub>	45.16

## 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 may 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  $\text{Fe}^{+2}/\text{Fe}^{\text{total}}$  ratio, which should be maintained under 0.3. The maximum concentration for sucrose, based on crucible tests, is 141 g of sugar per liter of surrogate SBW. This maximum value is about 64% of the amount needed to stoichiometrically react C with nitrates to reduce the nitrates to N<sub>2</sub>. While initial reductant concentrations may target values less than this, the results of initial testing early in the test series should provide information to enable adjusting the reductant concentration to maximize the melt rate while staying within the acceptance limit for the glass oxidation state.

Different waste loadings also need to be investigated during the test series. Batches need to be prepared that can enable increasing or decreasing the waste loading according to Table 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 11 shows the composition of the glass-forming chemicals used as a feed additive to produce a regulatorily acceptable product glass.

**Table 11. Frit Composition For The January 2001 RSM Test Series.**

<b>Frit</b>	<b>Oxide</b>		<b>Glass Former</b>		<b>Mass Ratio</b>
<b>Oxide</b>	<b>Wt%</b>	<b>MW</b>	<b>Chemical</b>	<b>MW</b>	<b>(g-Chem /g-frit)</b>
B <sub>2</sub> O <sub>3</sub>	12	69.6	H <sub>3</sub> BO <sub>3</sub>	61.8	0.266
Fe <sub>2</sub> O <sub>3</sub>	12	159.7	Fe <sub>2</sub> O <sub>3</sub>	160	0.100
Li <sub>2</sub> O	4.0	29.9	LiOH*H <sub>2</sub> O	42.0	0.140
SiO <sub>2</sub>	70	60.1	SiO <sub>2</sub>	60.1	0.650
CaO	<u>2.0</u>	56.1	Ca(OH) <sub>2</sub>	74.1	0.066

## 7. RSM FACILITY DESCRIPTION

The Research Scale Melter (RSM) facility is located in the Applied Process Engineering Laboratory (APEL) building in Richland, Washington. Figure 1 shows the RSM system. Table 12 shows RSM dimensions and other operational features.

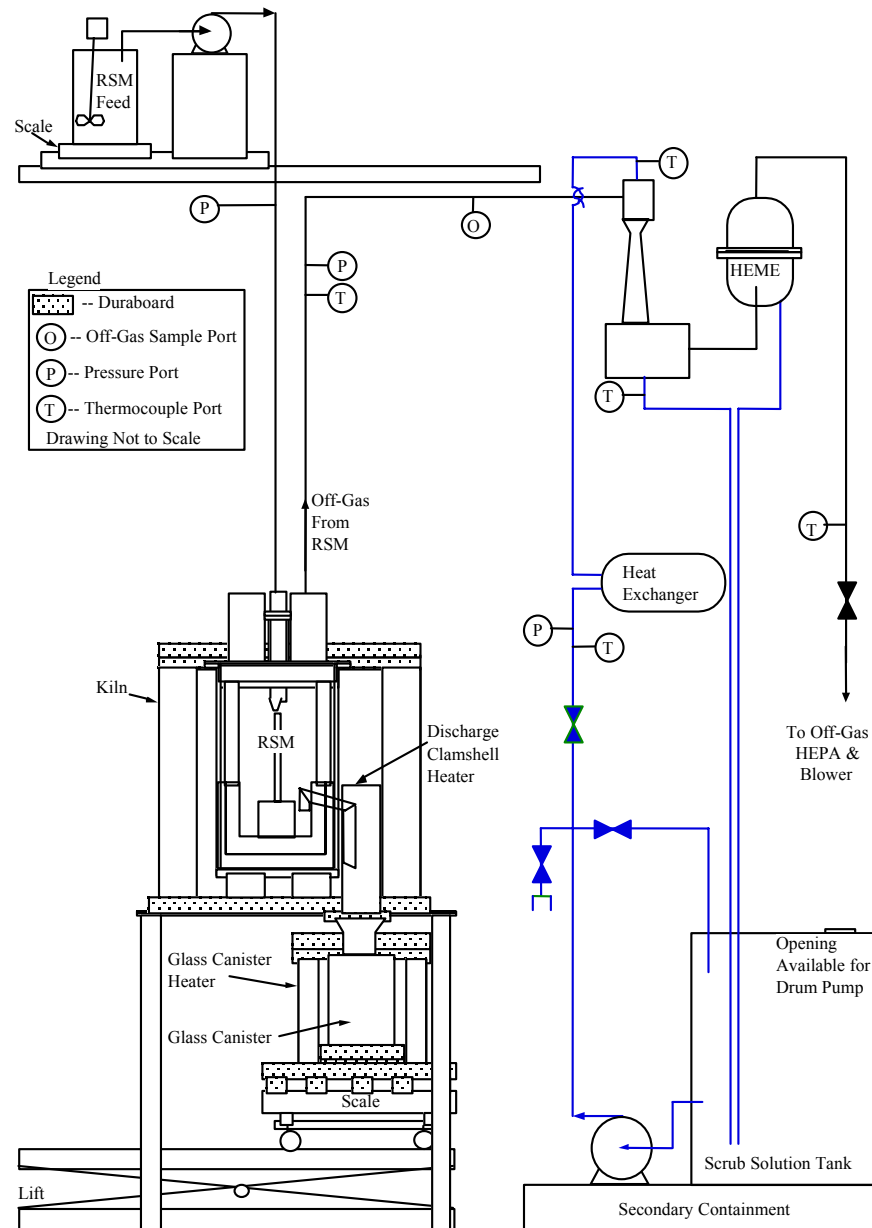


Figure 1. Research-Scale Melter Test Apparatus (Not shown is a HEPA filter that was installed for this test downstream of the HEME)

**Table 12. RSM dimensions and operational features**

Parameter	Value
Melter cavity diameter	15 cm
Melter cavity height	17 cm
Melter inside volume	4.5 liters
Glass pool surface area	182 cm <sup>2</sup>
Nominal glass depth	7.6 cm
Melter inside volume	1.4 liters
Nominal molten glass mass	3.6 kg
Glass turnover rate @ nominal feedrate of 1.5 liters/hr of feed with 0.6 kg/liter oxides	4.5 hr
Maximum operating temperature	1,200°C
Nominal operating Temperature	1,150°C
Electrode Dimensions	7.6 cm x 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 <sup>2</sup>

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 offgas 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 is displayed at more frequent intervals to assist the operators.



## 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 work place copy. Changes may be entered by the 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 13 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 13. Data Quality Objectives for RSM Test Objectives**

Objective#	C/NC	Quant.	Qual.	DQOs
1	C	x		By using standard with established procedures, develop an optimized SBW melter process flowsheet that doesn't produce a separate salt phase, and results in a glass waste product that maximizes waste loading and meets regulatory repository requirements,
2	C	x		Analyses of product streams by standard methods with established QA/QC procedures; replicates performed to obtain statistical confidence intervals; accuracies of calibrated M&T are summarized in Appendix A
3 & 4	C	x	x	Characterize maximum stable processing rates of the SBW baseline flowsheet as a function of waste loading and reductant composition using M&TE listed in Appendix A. Analyze vitreous waste products by standard methods with established QA/QC procedures, to establish compliance with regulatory standards. Process observations
5 & 6	NC	x		Recorded process measurements using the M&TE listed in Appendix A will be use to accomplish these test objectives.

### 8.1.2 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  $\pm 2\%$  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 using the user calibrated instruments previously described in Table 7. These gas analyzers will also be used to characterize total unquenched melter flow rates downstream of the melter's film cooler 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. The quantified off-gas flow rate along with off-gas temperature and piping dimensions will allow isokinetic, particulate sampling flow rate conditions to be established for a sampling probe of fixed cross sectional area.

### 8.1.3 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. All attempts will be made to conduct operations 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

The melter feed stream that will be thermally processed by the RSM contains the reductant sucrose (sugar). The purpose for feed component sugar is to control the overall oxidation state of the product glass. The unmodified melter feed stream is extremely oxidizing due to its high acidity ( $\text{pH} \approx 0$ ) and  $\text{NO}_3^-$  (5M) content. Because of the high melter oxidizing conditions,  $\text{CO}_2$  and  $\text{H}_2\text{O}$  will be the primary thermal effluent gases generated by feed component sugar. Historically, these primary combustion products have always been observed to dominate the melter reductant source term whenever sugar has been used.

Although  $\text{CO}_2$  and  $\text{H}_2\text{O}$  will be the dominant thermal byproducts of sugar, the off-gases generated could possibly contain pyrolytic and incomplete products of combustion such as  $\text{CO}$ ,  $\text{H}_2$ , and volatile organic compounds (VOCs). However, the highly oxidizing conditions expected in the plenum will virtually eliminate the flammability risks posed by pyrolytic byproducts such as  $\text{H}_2$ , and VOCs.  $\text{CO}$ , on the other hand, is expected to be present at measurable concentrations ( $\sim 0.1$  Vol%).

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  $300^\circ\text{C}$  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%. However, the worst-case off-gas system concentration of  $\text{CO}$  (where all available carbon is converted to  $\text{CO}$ ) that could occur under maximum melter feeding and sugar loading conditions and minimum film-cooler flow rate (6 scfm) is: 1.2% (9.4% of LFL). Since, as mentioned above, flammability issues involving VOCs are expected to be inconsequential, baseline-operating conditions preclude hazardous flammability-based test conditions. Nevertheless, in order to assure and maintain safe operating conditions, active monitoring of combustible off-gas constituents will be continuously conducted through use of a total hydrocarbon analyzer for VOCs and a gas chromatograph for  $\text{H}_2$  and  $\text{CO}$ .

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). VOC 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. Similarly, 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, latest version
2. The Research Scale Melter Test Plan
3. All applicable test instructions (see Appendix A)
4. Applicable Material Safety Data Sheets

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 offgas line, etc.). Prior to opening the melter, feeding will be stopped and the cold cap will allowed to be fully incorporated into the 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.

### **9.2.2 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 high-bay experimental area hosting the RSM. The various operational scenarios that could lead to a process exhaust failure and the emergency responses required are detailed in RSM-SOP-80. However it is useful to estimate the maximum possible gas release condition expected from a worse case scenario.

Under worst-case conditions (facility power failure) all off-gas functions would be lost. If under these conditions, a 100% cold cap is assumed that is:

- 6" in diameter by 1" thick,
- devoid of water (1.6 g/cc) and

- totally unreacted,

the maximum possible volumetric releases of all anticipated noxious, room-temperature gases are summarized in Table 14.

Table 14. Worst Case RSM Noxious Gas Release Estimates Accompanying Off-Gas System Failure

<b>Noxious Volatiles</b>	<b>Max Vol (Std.liters)</b>	<b>Vol (%)</b>	<b>Max Mixed Conc. (ppm)</b>
NO, NO <sub>2</sub> , N <sub>2</sub> O	127.4	97.61	5.80
SO <sub>2</sub> , H <sub>2</sub> S	1.3	0.97	0.06
HF, F <sub>2</sub>	1.1	0.87	0.05
HCl, Cl <sub>2</sub>	0.7	0.54	0.03
HI, I <sub>2</sub>	0.003	0.002	0.0001

It is clear from this tabular data that NO<sub>x</sub> is the dominant (98%) gas generated. If it is further assumed that these released gases were allowed to mix uniformly within the confines of the high-bay without the benefit of outside ventilation that would normally result from required emergency responses, the total resultant concentration of all released gases, (excluding steam) would be bounded at 10ppm. However, the cross ventilation that will result from opening of the (5) 14-ft roll-up doors located on all but one side of the high-bay will provide quick and effective recovery from any uncontrolled release of process gases, should such an event occur.

### 9.2.3 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 the caustic off-gas condensate. Because the simulated melter waste and feed streams are highly acidic (pH~0) and contain small concentrations of HF, a full-face shield, goggles, acid resistant over garments and HF-rated, protective (non-latex) 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 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 the corrosive, acidic feed conditions. 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. Appendix A contains additional HF handling precautions. Equipment needed for other activities are discussed in SOP #80.

### 9.2.4 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. Details concerning the probing of the melter with an alumina rod are provided in RSM test instructions (see Appendix A); 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.

#### **9.2.5 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.

#### **9.2.6 Medical Requirements**

Since the waste and feed streams will be treated with the same precautions required for HF solutions, skin exposure to either waste or feed solutions will require medical attention, after the exposed area is thoroughly flushed with water and application of calcium glutonate, as prescribed by the MSDS, has been accomplished.

#### **9.2.7 Confined Space**

Not Applicable.

#### **9.2.8 Respiratory Protection Requirements**

There are no respiratory protection requirements for operation of the RSM unit.

### **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

1. Standard Test Methods for Determining Chemical Durability of Nuclear Waste Glasses: The Product Consistency Test (PCT), **ASTM C1285-94**, 1994.
2. Christian, Jerry D., BBWI 4121, INEEL, private communication, November 2000.



## **Appendix A.1**

## Research-Scale Melter Measurement and Testing Equipment - DRAFT

DESCRIPTION	M&TE DESCRIPTION	LOCATION	CALIBRATION	CALIBRATION
			BASIS	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	Offgas 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 Offgas 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
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-01-1 TEST INSTRUCTION

This test instruction provides additional guidance to testing staff during the execution of the INEEL SBW RSM test, RSM-01-1.

### Objectives

Waste Loading Determination: The main test objective is to establish a waste loading that does not result in the formation of a sulfate salt phase on the glass surface. An initial waste loading will be recommended by John Vienna prior to the test start and concurred upon by INEEL staff. It is expected that a waste oxide loading between 28 and 33% will be recommended. A sugar addition level to achieve an acceptable redox level will also be defined by John Vienna prior to the test. Based on testing to date, the sugar addition level is expected to be between 110 and 141 grams of sugar per liter of SBW simulant.

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 and 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 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 can not 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 cornstarch slurry will be made and feed using the feed pump to meter the slurry into the RSM. There is some consideration to introducing the cornstarch into the RSM in a dry form. This may be attempted with small batches dropped through an insulated piece of tubing and determining if the cornstarch is reacting with the sulfate or simply burning in the plenum space.

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 skimmed to reduce the waste loading and melter feeding resumed. The waste oxide loading will be defined by the shift leader based on guidance provided at the start of the test and recorded in this test instruction (see Attachment 1: Run Guidance)

No Salt Layer Formation: For planning purposes it has been established that at least 24 hours of melter feeding will be required as a minimum to confirm an acceptable waste loading, i.e., no visible salt phase accumulation. At an assumed feed rate of 1.5 L/h, 450 g-oxide per liter of feed and a 1.4 liter glass tank volume approximately 4.5 tank turnovers can be achieved. If feed rates are significantly different than 1.5 L/h the lead test engineer will define the minimum run time in Attachment 1: Run Guidance. If no salt formation has been observed at the end of the minimum run time the shift leader will confer with the INEEL shift member and agree to suspend this feeding phase. Glass and glass surface samples should be obtained (again using a ceramic alumina probe unless lockout/tagout is applied) as described above after the cold cap has melted. The feed tank contents will then be adjusted or replaced with a batch of feed containing the next higher waste loading target (see Attachment 1 Run Guidance).

Reductant Level: The initial reductant level will have been established prior to starting the run (see Attachment 1). If the waste loading test objectives have been accomplished within the 96-hr feeding period; a second redox level will be tested to assess its effect on sulfate salt formation. Based on earlier test results and measurements of the glass  $\text{Fe}^{+3}/\text{Fe}^{\text{total}}$  redox values a second and possibly a third reductant level will be selected for testing. It is assumed that if two levels may be tested, reductant levels will be progressively increased to avoid the need for having to change out the melter feed. That is, more reductant will be added to the feed rather than having to replace the feed with a feed batch containing less reductant. The duration of each test will depend on the initial conditions of the melter, i.e., if a salt layer is already present, if the redox analyses performed by an INEEL staff member can be performed quickly enough to observe a stable redox level prior to changing to the next redox level.

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 approximately 12 hours of operation. 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 quantity of glass formers (here glass formers includes the sugar reductant). There is expected to be a 10% to 20% volume expansion in feed volume when the glass formers are added to the SBW waste simulant. However, for

batching purposes, melter feed volume will be assumed to be equivalent the SBW waste simulant volume. The attached Feed Batching Sheet (Attachment 2) 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-01-1-1")
    - 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 tank will be used to transfer feed to the make-up tank. It is required that splash protection, coat and overalls, full-face shield, safety goggles and gloves suitable for HF be donned.
  - 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 and enter into the Batch Sheet the glass former weights.
  - 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: 2 staff are required for this activity):
- a. Place two buckets next to the feed station. One should have approx. one 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 coat and overalls).
  - 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. Please the copus blower intake hose at the feed station.
  - g. Reduce the melter feed tank agitator speed 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 valve use 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 THAT 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 melter lid and slid 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 log book.**
3. Off-gas condensate tank pH adjustment: The pH of the condensate will be measured hourly. If the pH drops to 9, add 50 grams of NaOH to the condensate tank. This should increase the pH to above 11. Record the amount of NaOH added in the laboratory record book and the NaOH log sheet.
4. Sample Identification: Mark sample containers with the following:
- a. RSM-01-1
  - b. Sequential Sample Number
  - c. Date & Military Time
  - d. Sample description, e.g., *Feed, Condensate, Glass Sample*
  - e. Initial of operations staff obtaining sample
5. Routine data sheets and sample logs are attached for information. Data sheets #1 & #2 are to be filled out every hour on the hour.

## ATTACHMENT 1: RUN GUIDANCE

(Guidance to be initialed and dated by lead test engineer)

Initial Waste Loading:

---

Second Waste Loading if initial waste loading **IS NOT** Acceptable:

---

Second Waste Loading if initial waste loading **IS** Acceptable:

---

Third Waste Loading if second waste loading **IS NOT** Acceptable:

---

Third Waste Loading if second waste loading **IS** Acceptable:

---

Initial Sugar Addition Level:

---

Second Sugar Addition Level:

Third Sugar Addition Level:

---

Minimum Run Time to achieve 4.5 turnovers (based on feed rate):

---

Additional Guidance:

---

1. \_\_\_\_\_
2. \_\_\_\_\_
3. \_\_\_\_\_
4. \_\_\_\_\_
5. \_\_\_\_\_
6. \_\_\_\_\_
7. \_\_\_\_\_
8. \_\_\_\_\_
9. \_\_\_\_\_
10. \_\_\_\_\_



## ATTACHMENT 2: FEED BATCHING CALCULATION SHEET

Batch Sheet No. RSM-01-1-: \_\_\_\_\_

Prepared by: \_\_\_\_\_

Date: \_\_\_\_\_ Time: \_\_\_\_\_

SBW target volume, Liters: \_\_\_\_\_

SBW transfer tank gross wt: \_\_\_\_\_ kg  
 SBW transfer tank tare wt: 2.30 kg  
 SBW transfer tank net wt: \_\_\_\_\_ kg  
 SBW Density: 1.25 kg/L

Calculated volumed  
 transferred: Net wt. / density = \_\_\_\_\_

Target glass waste oxide  
fraction:

### 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: 114.6 = A  
 Fraction waste oxide loading: \_\_\_\_\_ = B  
 Fraction glass former loading: \_\_\_\_\_ = C  
 SBW Volume, liters \_\_\_\_\_ = D  
  
 (A/B) \* C \* D = \_\_\_\_\_ = E

Glass Formers	F		Batch totals: (E * F)	Initial after weighing
gm silica / gm total GFO =	<u>0.7</u>	=>	_____ grams	_____
gm boric acid / gm total GFO =	<u>0.213</u>	=>	_____ grams	_____
gm Fe2O3 / gm total GFO =	<u>0.12</u>	=>	_____ grams	_____
gm LiOH / gm total GFO =	<u>0.064</u>	=>	_____ grams	_____
gm Ca(OH)2 / gm total GFO =	<u>0.026</u>	=>	_____ 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: \_\_\_\_\_

# Data Sheet #1: Priority & Electrical Data

<b>RSM-01-1</b>										
	By:									
	Date:									
	Time									
DESCRIPTION	UNITS	Range								
<b>Priority Data</b>										
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 LRE	y or n									
Glass Pouring	y or n									
<b>Electrical Data</b>										
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 Output	%									
Time	hh:mm									

## Data Sheet #2: Routine Status Sheet

**RSM-01-1**

		By:								
		Date:								
		Time:								
<b>DESCRIPTION</b>	<b>units</b>	<b>range</b>								
Feed Nozzle Cooling Flow	gpm	.5 - 1(reg FNT)								
Film Cooler Air Supply	scfm	1-10 (reg OGT)								
Melter Vacuum-Magnehelic	in. H <sub>2</sub> O	0.1 - 2.0								
EVS ? P	in. H <sub>2</sub> O									
Film Cooler ? P	in. H <sub>2</sub> O									
HEME ? P	in. H <sub>2</sub> O									
System ? P	in. H <sub>2</sub> O									
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									

## Sample Log Sheet

[illegible]

## Safety Precautions Working with SBW Feed

Hydrofluoric acid (HF) is present in the SBW simulant at a concentration of approximately 0.03 moles per liter. At a density of 1.25 g/ml the weight percent HF in the SBW is only 0.05 wt.%. Although this concentration is very low, MSDS documentation on 1 wt.% recommends treating skin contact in the same manner as if the solution were concentrated HF. HF acid is a health hazard to the respiratory and digestive systems, and an extreme irritant to the skin. Hydrofluoric acid is especially dangerous as it anesthetizes the nerves as it makes contact with the body. Major and potential health hazards are:

- Major health hazards: No significant target effects reported.
- Potential health effects:
  - Inhalation:
    - Short term exposure: no information on significant adverse effects
    - Long term exposure: no information on significant adverse effects
  - Skin contact:
    - Short term exposure: delayed tissue destruction
    - Long term exposure: no information on significant adverse effects
  - Eye contact:
    - Short term exposure: no information on significant adverse effects
    - Long term exposure: no information on significant adverse effects
  - Ingestion:
    - Short term exposure: no information on significant adverse effects
    - Long term exposure: no information on significant adverse effects

If any feed spills occur, even a drop of solution, clean up the area carefully with a damp towel.

**A container of calcium gluconate is available at the sink along the north wall.** This is an antidote that helps neutralize the HF acid burns on the skin. The quickest and best remedy for HF acid burns to the skin are copious amounts of water, intermingled with scrubbing with soap, repeated numerous rinsings and scrubbing followed by applications of the calcium gluconate which is to be applied repeatedly while medical help is being obtained. Do not breathe fumes of HF acid even for a very short time, the fumes can cause severe damage to the respiratory system.

Fumes from the HF acid are very harmful to the respiratory system. This is not expected to be a factor because of the very dilute concentration in the feed. However, melter off-gas emissions are also a source of HF fumes. As a result, except when changing the viewport glass, no access into the melter should occur without first turning the feed off and allowing any liquid in the melter to completely evaporate. When changing the viewport window other staff should remain clear of the immediate area and positive vacuum must be maintained on the melter. This may require the melter off-gas control to be temporarily switched from auto to manual control (if not already on manual control) to prevent the controller from over-reacting to the approx. one second period when the viewport glass is removed.

Full personal protection equipment are to be worn when handling the SBW simulant or melter feed (feed make-up and feed sampling) or when working on any part of the feed mixing or RSM feeding equipment. Minimum PPE includes:

1. *Chemical safety goggles, at a minimum, are worn when handling the corrosive chemicals that may splash. Chemical splash shields, in combination with safety glasses or safety goggles, are worn when working with these chemicals.*
2. *Operations that pose a skin exposure while working with chemicals require hand gloves to be worn. Specific types include: nitrile or neoprene gloves*
3. *Protective coat and bib overalls are to be worn when performing feed makeup and transfer operations.*

## **Appendix B**

### **Test Instruction for Preparing 200l of Sodium Bearing Waste Simulant**

## **TEST INSTRUCTION FOR PREPARING 200L OF SODIUM BEARING WASTE SIMULANT**

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

Start Date:

End Date:

Laboratory Record Book Used \_\_\_\_\_, pp. \_\_\_\_\_

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

Author: \_\_\_\_\_

Responsible Scientist: .

Test Operator(s): \_\_\_\_\_

\_\_\_\_\_

Balance Calibration Record:

Balance Calibration # \_\_\_\_\_ Last Cal. \_\_\_\_\_ Due \_\_\_\_\_

Balance Calibration # \_\_\_\_\_ Last Cal. \_\_\_\_\_ Due \_\_\_\_\_

Chemical Inventory List:

[illegible]



1. Add 952.80 g of 50%  $\text{Mn}(\text{NO}_3)_2$  solution and 57.11 L of 2.2M  $\text{Al}(\text{NO}_3)_3$  to an appropriate sized vessel.

Amount of  $\text{Mn}(\text{NO}_3)_2$  solution added: \_\_\_\_\_

Amount of 2.2M  $\text{Al}(\text{NO}_3)_3$  added: \_\_\_\_\_

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

<u>Chemical</u>	<u>Target Wt.</u>	<u>Actual Wt.</u>	<u>Initial</u>	<u>Date</u>
$\text{NaNO}_3$	33.01 kg	_____	_____	_____
$\text{KNO}_3$	3744.4 g	_____	_____	_____
$\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$	2139.0 g	_____	_____	_____
$\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$	44.00 g	_____	_____	_____
$\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	80.80 g	_____	_____	_____
$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	1656.4 g	_____	_____	_____
$\text{ZrF}_4$	2.00 g	_____	_____	_____
$\text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	253.2 g	_____	_____	_____
$\text{Ba}(\text{NO}_3)_2$	2.80 g	_____	_____	_____
$\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$	3.80 g	_____	_____	_____
$\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	1.00 g	_____	_____	_____
$\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$	31.80 g	_____	_____	_____
$\text{Gd}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$	14.60 g	_____	_____	_____
$\text{Pb}(\text{NO}_3)_2$	81.60 g	_____	_____	_____
$\text{LiNO}_3$	4.40 g	_____	_____	_____
$\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	582.60 g	_____	_____	_____
$\text{RuCl}_3$	4.80 g	_____	_____	_____

		_____	_____	_____
Sr(NO <sub>3</sub> ) <sub>2</sub>	4.80 g	_____	_____	_____
TiO <sub>2</sub>	0.80 g	_____	_____	_____
Zn(NO <sub>3</sub> ) <sub>2</sub> @6H <sub>2</sub> O	59.00 g	_____	_____	_____
KI	4.20 g	_____	_____	_____
CsNO <sub>3</sub>	63.38 g	_____	_____	_____

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

Actual amount of DI water added: \_\_\_\_\_

3. Add 143.6 g H<sub>3</sub>BO<sub>3</sub>. Dissolve as much as possible by heating and stirring as necessary.

Amount of H<sub>3</sub>BO<sub>3</sub> added: \_\_\_\_\_

4. Discontinue heating the simulant. Add 234 mL of 28.9 M HF. Stir well. **Wear acid protective clothing, goggles, and gloves. Be extremely careful in handling HF as it is very corrosive and dangerous.**

Amount of HF added: \_\_\_\_\_

5. In a separate vessel, add about 1800 mL of DI water. Slowly add 566 mL of 18M H<sub>2</sub>SO<sub>4</sub> to the water and mix thoroughly.

Amount of DI water added: \_\_\_\_\_ Amount of 18M H<sub>2</sub>SO<sub>4</sub> added: \_\_\_\_\_

6. Slowly add the H<sub>2</sub>SO<sub>4</sub> 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 170 L.

Amount of DI water added: \_\_\_\_\_

8. In a separate vessel, combine 464 mL 12M HCl, 178 mL 14.6M H<sub>3</sub>PO<sub>4</sub> and 11.41 L 15.4M HNO<sub>3</sub>.

Amount of HCl added: \_\_\_\_\_ Amount of HNO<sub>3</sub> added: \_\_\_\_\_

Amount of H<sub>3</sub>PO<sub>4</sub> added: \_\_\_\_\_

9. Slowly add this mixture of acids to 20 L of DI water.

Amount of DI water added: \_\_\_\_\_

10. Slowly add the diluted acid mixture to the 170 L solution.

11. Add 5.89 g  $\text{H}_2\text{MoO}_4$  to the solution.

Amount of  $\text{H}_2\text{MoO}_4$  added: \_\_\_\_\_

12. Add DI water (approximately 10 L) to the combined solution, bringing the final volume to 200 L.

Amount of DI water added: \_\_\_\_\_

Final slurry volume: \_\_\_\_\_

## **Appendix C**

### **RSM Feed Batching and Manual Data Sheets**

## **Feed Batching Sheets**

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

Batch Sheet No. RSM-01-1-: 1

Prepared by: Renee Russell

Date: 1-29-01

Time: 3:35pm

SBW target volume, Liters: 40

Note: shaded areas  
require value to be input

SBW transfer tank gross wt: 52.1 kg

SBW transfer tank tare wt: 2.30 kg

SBW transfer tank net wt: 49.80 kg

SBW Density: 1.25 kg/L

Calculated volumed  
transferred: Net wt. / density = 39.84 Liters

Target glass waste oxide  
fraction: 0.3

### 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: 114.6 = A  
Fraction waste oxide loading: 0.3 = B  
Fraction glass former loading: 0.7 = C  
SBW Volume, liters 39.84 = D

(A/B) \* C \* D = 10653.22 = E

Glass formers	F	Batch totals: (E * F)	Initial after weighing
gm silica / gm total GFO =	0.65 =>	6924.6 grams	<u>6925.1g</u> PR
gm boric acid / gm total GFO =	0.2664 =>	2838.0 grams	<u>2838.0g</u> PR
gm Fe2O3 / gm total GFO =	0.10 =>	1065.3 grams	<u>1065.2</u> HDS
gm LiOH / gm total GFO =	0.1404 =>	1495.7 grams	<u>1495.9</u> HDS
gm Ca(OH)2 / gm total GFO =	0.0661 =>	704.2 grams	<u>704.2g</u> PR
gm sugar/L SBW	135 =>	5378.4 grams	<u>5378.4g</u> HDS

Initial melter feed tank weight: 50.0 kg

Time: 16:18

Melter feed tank wt. after SBW  
addition: 50.0 kg

Time: 16:18

Melter feed tank wt. after glass  
formers addition: 68.0 kg

Time: 17:18

Completed by (sign & data): [Signature] 1/29/01

Reviewed & Approved by: [Signature] 1/29/01

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

Batch Sheet No. RSM-01-1-: 2

Prepared by: Renée Russell

Date: 1-30-01

Time: 9:15 am

SBW target volume, Liters: 15

Note: shaded areas  
require value to be input

SBW transfer tank gross wt: 20.95 kg  
SBW transfer tank tare wt: 2.30 kg  
SBW transfer tank net wt: 18.65 kg  
SBW Density: 1.25 kg/L

Calculated volumed

transferred: Net wt. / density = 14.92 Liters

Target glass waste oxide  
fraction: 0.3

## Glass Former Addition Calculation

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

Waste oxide loading, g/L: 114.6 = A  
Fraction waste oxide loading: 0.3 = B  
Fraction glass former loading: 0.7 = C  
SBW Volume, liters 14.92 = D

$(A/B) * C * D =$  3989.61 = E

Glass formers	F	Batch totals: (E * F)	Initial after weighing
gm silica / gm total GFO =	0.65 =>	<u>2593.2</u> grams	<u>2593.1</u> g HPS
gm boric acid / gm total GFO =	0.2664 =>	<u>1062.8</u> grams	<u>1062.8</u> g PR
gm Fe2O3 / gm total GFO =	0.10 =>	<u>399.0</u> grams	<u>399.0</u> g PR
gm LiOH / gm total GFO =	0.1404 =>	<u>560.1</u> grams	<u>560.1</u> g PR
gm Ca(OH)2 / gm total GFO =	0.0661 =>	<u>263.7</u> grams	<u>263.7</u> g PR
gm sugar/L SBW	135 =>	<u>2014.2</u> grams	<u>2014.2</u> g HPS

Initial melter feed tank weight: Not RECORDED kg <sup>RW 9</sup> <sub>18.65 kg</sub> Time: \_\_\_\_\_  
Melter feed tank wt. after SBW addition: Not RECORDED kg <sup>RW 9</sup> <sub>PER FAIRBANKS LOAD CELL</sub> Time: \_\_\_\_\_  
Melter feed tank wt. after glass formers addition: 46 kg Time: 11:45

Completed by (sign & data): RW 9

Reviewed & Approved by: Bry 2/1/01



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

1 of 2  
DMS 1/30/01

Batch Sheet No. RSM-01-1-: 3

Prepared by: D. Bennett

Date: \_\_\_\_\_

Time: \_\_\_\_\_

SBW target volume, Liters: 4.52

Note: shaded areas  
require value to be input

SBW transfer tank gross wt: 17 kg

SBW transfer tank tare wt: 2.30 kg

SBW transfer tank net wt: 14.70 kg

SBW Density: 1.25 kg/L

Calculated volumed  
transferred: Net wt. / density = 11.76 Liters

Target glass waste oxide  
fraction: 0.3

## ADJUSTMENT CALCULATION

① DETERMINE SBW IN REMAINING  
20.1 kg of FEED

② DETERMINE ADDED SBW TO  
BRING FEED TO 32% W.L.

5433g GLASS FORMERS  
14700g SBW  
20,133g FEED  
@ 30% W.O. LOADING

## Glass Former Addition Calculation

Glass former weights:  $114.6 \text{ gm WO/L} \times (1 \text{ gm glass} / X \text{ gm WO}) \times (\text{gm total GFO} / \text{gm glass}) \times Y \text{ L SBW} \times (\text{gm GF chemical} / \text{gm total GFO})$

Waste oxide loading, g/L: 114.6 = A

Fraction waste oxide loading: 0.3 = B

Fraction glass former loading: 0.7 = C

SBW Volume, liters 11.76 = D

$(A/B) \times C \times D = 3144.62 = E$

Glass formers	F	Batch totals: (E * F)	Initial after weighing
gm silica / gm total GFO =	0.65 =>	2044.0 grams	
gm boric acid / gm total GFO =	0.2664 =>	837.7 grams	
gm Fe2O3 / gm total GFO =	0.10 =>	314.5 grams	
gm LiOH / gm total GFO =	0.1404 =>	441.5 grams	
gm Ca(OH)2 / gm total GFO =	0.0661 =>	207.9 grams	
gm sugar/L SBW	135 =>	1587.6 grams	

5433.2g

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: [Signature] 2/1/01



2002

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

Batch Sheet No. RSM-01-1-: 3

ADJUSTMENT CALCULATION  
1/30/01 20:45 (AM)

Prepared by: P. Burt

Date: \_\_\_\_\_ Time: \_\_\_\_\_

SBW target volume, Liters: 4.52

Note: shaded areas  
require value to be input

SBW transfer tank gross wt: 18 kg

SBW transfer tank tare wt: 2.30 kg

SBW transfer tank net wt: 15.70 kg

SBW Density: 1.25 kg/L

Calculated volumed

transferred: Net wt. / density = 12.56 Liters

5436.1 g GLASS FORMERS  
15700 g SBW  
21,136.1

Target glass waste oxide  
fraction: 0.32

EXISTING IN TANK - 20,133 g  
 $\Delta = 1003 \text{ g} = .8 \text{ L}$

## 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: 114.6 = A

Fraction waste oxide loading: 0.32 = B

Fraction glass former loading: 0.68 = C

SBW Volume, liters 12.56 = D

(A/B) \* C \* D = 3058.67 = E

0.8 L SBW @  
135 g sugar/L SBW  
= 108 g sugar.

Glass formers	F	Batch totals: (E * F)	Initial after weighing
gm silica / gm total GFO =	0.65 =>	1988.1 grams	
gm boric acid / gm total GFO =	0.2664 =>	814.8 grams	
gm Fe2O3 / gm total GFO =	0.10 =>	305.9 grams	
gm LiOH / gm total GFO =	0.1404 =>	429.4 grams	
gm Ca(OH)2 / gm total GFO =	0.0661 =>	202.2 grams	
gm sugar/L SBW	135 =>	1695.6 grams	
			5436.1 g GLASS FORMERS

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: Sherry 2/01/01

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

Batch Sheet No. RSM-01-1-: 4

Prepared by: H. D. Smith / D. Bennett

Date: 1/30/01

Time: 21:59

SBW target volume, Liters: 30

Note: shaded areas  
require value to be input

SBW transfer tank gross wt: 37.5 kg

SBW transfer tank tare wt: 2.30 kg

SBW transfer tank net wt: 35.20 kg

SBW Density: 1.25 kg/L

TRANS-1 20L - 27.10 - 2.3  
= 24.8 kg / 19.8 L

TRANS 2 - TARGET 10.4 kg / 8.32 L  
12.7 - 2.3 = 10.4 kg

Calculated volumed

transferred: Net wt. / density = 28.16 Liters

Target glass waste oxide

fraction: 0.32

## Glass Former Addition Calculation

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

Waste oxide loading, g/L: 114.6 = A

Fraction waste oxide loading: 0.32 = B

Fraction glass former loading: 0.68 = C

SBW Volume, liters 28.16 = D

(A/B) \* C \* D = 6857.66 = E

Glass formers	F	Batch totals: (E * F)	Initial after weighing
gm silica / gm total GFO =	0.65 =>	4457.5 grams	<u>4457.5</u> HDS
gm boric acid / gm total GFO =	0.2664 =>	1826.9 grams	<u>1826.9</u> HDS
gm Fe2O3 / gm total GFO =	0.10 =>	685.8 grams	<u>685.8</u> HDS
gm LiOH / gm total GFO =	0.1404 =>	962.8 grams	<u>962.8</u> HDS
gm Ca(OH)2 / gm total GFO =	0.0661 =>	453.3 grams	<u>453.3</u> HDS
gm sugar/L SBW	135 =>	3801.6 grams	<u>3801.6</u> HDS

Initial melter feed tank weight: 45.5 / 15.4 kg  
Melter feed tank wt. after SBW addition: TRANS-1 40.1 kg  
Melter feed tank wt. after glass formers addition: TRANS-2 50.4 kg

Time: 23:22

Time: 23:48

Time: 00:12

12,188.1

Completed by (sign & data): JM Davis 1/31/01

Reviewed & Approved by: [Signature] 1/31/01

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

Batch Sheet No. RSM-01-1-: 5

Prepared by: Renee Russell

Date: 1-31-01

Time: 2:20pm

SBW target volume, Liters: 14

Note: shaded areas  
require value to be input

SBW transfer tank gross wt: 19.7 kg

SBW transfer tank tare wt: 2.35 kg

SBW transfer tank net wt: 17.35 kg

SBW Density: 1.25 kg/L

Calculated volumed

transferred: Net wt. / density = 13.88 Liters

Target glass waste oxide  
fraction: 0.32

### Glass Former Addition Calculation

Glass former weights:  $114.6 \text{ gm WOIL} * (1 \text{ gm glass} / X \text{ gm WO}) * (\text{gm total GFO} / \text{gm glass}) * Y \text{ L SBW} * (\text{gm GF chemical} / \text{gm total GFO})$

Waste oxide loading, g/L: 114.6 = A

Fraction waste oxide loading: 0.32 = B

Fraction glass former loading: 0.68 = C

SBW Volume, liters 13.88 = D

$(A/B) * C * D =$  3380.13 = E

Glass formers	F	Batch totals: (E * F)	Initial after weighing
gm silica / gm total GFO =	0.65 =>	<u>2197.1</u> grams	<u>2196.8g RR</u>
gm boric acid / gm total GFO =	0.2664 =>	<u>900.5</u> grams	<u>900.5g RR</u>
gm Fe2O3 / gm total GFO =	0.10 =>	<u>338.0</u> grams	<u>338.0g RR</u>
gm LiOH / gm total GFO =	0.1404 =>	<u>474.6</u> grams	<u>474.5g RR</u>
gm Ca(OH)2 / gm total GFO =	0.0661 =>	<u>223.4</u> grams	<u>223.3g RR</u>
gm sugar/L SBW	135 =>	<u>1873.8</u> grams	<u>1873.8g RR</u>

Initial melter feed tank weight: 13.5 kg

Time: 1503

Melter feed tank wt. after SBW  
addition: 31.0 kg

Time: 15:15

Melter feed tank wt. after glass  
formers addition: 37.0 kg

Time: 15:25

Completed by (sign & data): RWR

Reviewed & Approved by: [Signature] 1/31/01



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

Batch Sheet No. RSM-01-1-: 6

Prepared by: J. M. Perez

Date: 1/31/01

Time: 22:25

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: 0.35

### Glass Former Addition Calculation

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

Waste oxide loading, g/L: 114.6 = A

Fraction waste oxide loading: 0.35 = B

Fraction glass former loading: 0.65 = C

SBW Volume, liters 19.84 = D

$(A/B) * C * D =$  4222.52 = E

Glass formers	F	Batch totals: (E * F)	Initial after weighing
gm silica / gm total GFO =	0.65 =>	<u>2744.6</u> grams	<u>2744.6</u>
gm boric acid / gm total GFO =	0.2664 =>	<u>1124.9</u> grams	<u>1124.9</u>
gm Fe <sub>2</sub> O <sub>3</sub> / gm total GFO =	0.10 =>	<u>422.3</u> grams	<u>422.3</u>
gm LiOH / gm total GFO =	0.1404 =>	<u>592.8</u> grams	<u>592.8</u>
gm Ca(OH) <sub>2</sub> / gm total GFO =	0.0661 =>	<u>279.1</u> grams	<u>279.1</u>
gm sugar/L SBW	135 =>	<u>2678.4</u> grams	<u>2678.4</u>

Initial melter feed tank weight: 9.0 kg

Time: 23:17

Melter feed tank wt. after SBW

addition: 33.8 kg

Time: 23:31

Melter feed tank wt. after glass

formers addition: 42.5 kg

Time: 23:58

Completed by (sign & data): J. M. Perez 2/1/01

Reviewed & Approved by: [Signature] 2/1/01

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

Batch Sheet No. RSM-01-1-: 7

Prepared by: D. Bennert

Date: 1/31/01

Time: ~22:30 LB

SBW target volume, Liters: 6

Note: shaded areas  
require value to be input

SBW transfer tank gross wt: 9.7 kg

SBW transfer tank tare wt: 2.30 kg

SBW transfer tank net wt: 7.40 kg

SBW Density: 1.25 kg/L

BALANCE IN TANK = 10.0 kg

= 7.40 kg SBW

+ 2562 g GLASS FORMERS

9962 g FEED

Calculated volume

transferred: Net wt. / density = 5.92 Liters

ADDED SBW TO INCREASE TO  
35% W.L.

$7.40 \text{ kg} \cdot \frac{35}{32} = 8.09$

$8.09 - 7.40 = 0.69 \text{ kg ADDED}$

$0.69 \text{ kg} \cdot 1.25 / 1.25 \text{ kg} = 0.552 \text{ L}$

$0.552 \text{ L} \cdot 135 \text{ g SUGAR} = 74.5 \text{ g}$

SUGAR

## 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: 114.6 = A

Fraction waste oxide loading: 0.32 = B

Fraction glass former loading: 0.68 = C

SBW Volume, liters 5.92 = D

(A/B) \* C \* D = 1441.67 = E

Glass formers	F	Batch totals: (E * F)	Initial after weighing
gm silica / gm total GFO =	0.65 =>	937.1 grams	
gm boric acid / gm total GFO =	0.2664 =>	384.1 grams	
gm Fe2O3 / gm total GFO =	0.10 =>	144.2 grams	
gm LiOH / gm total GFO =	0.1404 =>	202.4 grams	
gm Ca(OH)2 / gm total GFO =	0.0661 =>	95.3 grams	
gm sugar/L SBW	135 =>	799.2 grams	
Initial melter feed tank weight:	33.8 kg	Time: 23:34	} 2562 g GLASS FORMERS (for calculation purposes only - not added)
Melter feed tank wt. after SBW addition:	34.9 kg	Time: 23:35	
Melter feed tank wt. after glass formers addition:	42.5 kg	Time:	

Completed by (sign & data): D. Bennert 2/1/01 LB

Reviewed & Approved by: [Signature] 2/1/01 LB

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

Batch Sheet No. RSM-01-1-: 8

Prepared by: Renee Russell

Date: 2-01-01

Time: 10:00 am

SBW target volume, Liters: 20

Note: shaded areas  
require value to be input

SBW transfer tank gross wt: 29.75 kg

SBW transfer tank tare wt: 2.30 kg

SBW transfer tank net wt: 27.45 kg

SBW Density: 1.25 kg/L

Calculated volumed

transferred: Net wt. / density = 21.96 Liters

Target glass waste oxide  
fraction: 0.35

### Glass Former Addition Calculation

Glass former weights:  $114.6 \text{ gm WO/L} \times (1 \text{ gm glass} / X \text{ gm WO}) \times (\text{gm total GFO} / \text{gm glass}) \times Y \text{ L SBW} \times (\text{gm GF chemical} / \text{gm total GFO})$

Waste oxide loading, g/L: 114.6 = A

Fraction waste oxide loading: 0.35 = B

Fraction glass former loading: 0.65 = C

SBW Volume, liters 21.96 = D

$(A/B) \times C \times D =$  4673.72 = E

Glass formers	F	Batch totals: (E * F)	Initial after weighing
gm silica / gm total GFO =	0.65 =>	3037.9 grams	<u>3037.9 g RR</u>
gm boric acid / gm total GFO =	0.2664 =>	1245.1 grams	<u>1245.1 g RR</u>
gm Fe <sub>2</sub> O <sub>3</sub> / gm total GFO =	0.10 =>	467.4 grams	<u>467.4 g RR</u>
gm LiOH / gm total GFO =	0.1404 =>	656.2 grams	<u>656.4 g RR</u>
gm Ca(OH) <sub>2</sub> / gm total GFO =	0.0661 =>	308.9 grams	<u>309.0 g RR</u>
gm sugar/L SBW	135 =>	2964.6 grams	<u>2964.8 g RR</u>

Initial melter feed tank weight: 10.5 kg Time: 10:40

Melter feed tank wt. after SBW  
addition: 38.5 kg Time: 10:58

Melter feed tank wt. after glass  
formers addition: 46.5 kg Time: 11:40

Completed by (sign & data): RW Gals 2/1/01

Reviewed & Approved by: [Signature] 2/1/01



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

Batch Sheet No. RSM-01-1-: 9

Prepared by: J. M. Perez

Date: 2/1/01

Time: 23:44

SBW target volume, Liters: 20

Note: shaded areas  
require value to be input

SBW transfer tank gross wt: 27.35 kg

SBW transfer tank tare wt: 2.30 kg

SBW transfer tank net wt: 25.05 kg

SBW Density: 1.25 kg/L

Calculated volumed

transferred: Net wt. / density = 20.04 Liters

Target glass waste oxide  
fraction: 0.35

## Glass Former Addition Calculation

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

Waste oxide loading, g/L: 114.6 = A

Fraction waste oxide loading: 0.35 = B

Fraction glass former loading: 0.65 = C

SBW Volume, liters 20.04 = D

$(A/B) * C * D = 4265.08 = E$

Glass formers	F	Batch totals: (E * F)	Initial after weighing
gm silica / gm total GFO =	0.65 =>	2772.3 grams	2772.3
gm boric acid / gm total GFO =	0.2664 =>	1136.2 grams	1136.2
gm Fe2O3 / gm total GFO =	0.10 =>	426.5 grams	426.5
gm LiOH / gm total GFO =	0.1404 =>	598.8 grams	598.8
gm Ca(OH)2 / gm total GFO =	0.0661 =>	281.9 grams	281.9
gm sugar/L SBW	145 =>	2905.8 grams	2905.8

Initial melter feed tank weight: 10.5 kg

Time: 23:15

Melter feed tank wt. after SBW

addition: 35.5 kg

Time: 23:25

Melter feed tank wt. after glass

formers addition: 43.5 kg

Time: 2/2/01 - 01:05

Completed by (sign & data): J. M. Perez 2/2/01

Reviewed & Approved by: [Signature] 2/2/01

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

Batch Sheet No. RSM-01-1-: 10

Prepared by: Renee Russell

Date: 2-2-01

Time: 8:10 am

SBW target volume, Liters: 20

Note: shaded areas  
require value to be input

SBW transfer tank gross wt: 27.5 kg

SBW transfer tank tare wt: 2.30 kg

SBW transfer tank net wt: 25.20 kg

SBW Density: 1.25 kg/L

Calculated volumed

transferred: Net wt. / density = 20.16 Liters

Target glass waste oxide  
fraction: 0.35

## Glass Former Addition Calculation

Glass former weights:  $114.6 \text{ gm WO/L} \times (1 \text{ gm glass} / X \text{ gm WO}) \times (\text{gm total GFO} / \text{gm glass}) \times Y \text{ L SBW} \times (\text{gm GF chemical} / \text{gm total GFO})$

Waste oxide loading, g/L: 114.6 = A

Fraction waste oxide loading: 0.35 = B

Fraction glass former loading: 0.65 = C

SBW Volume, liters 20.16 = D

$(A/B) \times C \times D =$  4290.62 = E

Glass formers	F	Batch totals: (E * F)	Initial after weighing
gm silica / gm total GFO =	0.65	=> <u>2789.9</u> grams	<u>2789.3g</u> RR
gm boric acid / gm total GFO =	0.2664	=> <u>1143.0</u> grams	<u>1143.0g</u> RR
gm Fe2O3 / gm total GFO =	0.10	=> <u>429.1</u> grams	<u>429.3g</u> RR
gm LiOH / gm total GFO =	0.1404	=> <u>602.4</u> grams	<u>602.7g</u> RR
gm Ca(OH)2 / gm total GFO =	0.0661	=> <u>283.6</u> grams	<u>283.6g</u> RR
gm sugar/L SBW	145	=> <u>2923.2</u> grams	<u>2923.5g</u> RR

Initial melter feed tank weight: 15.0 kg

Time: 0850

Melter feed tank wt. after SBW

addition: 40.0 kg

Time: 0907

Melter feed tank wt. after glass

formers addition: 48.0 kg

Time: 0925

Completed by (sign & data): R. Russell

Reviewed & Approved by: M. D. 2/2/1

NOTE THIS RS WILL AS ISL HREFL WERE ADJUSTED FOR A 40% INCREASE  
IN SO<sub>2</sub> AND 165 1/2 SUGAR (WASTE BAK) SEE LRB



**Data Sheet #1: Priority & Electrical Data**

Data Sheet #1: Priority &amp; Electrical Data

RSM-01-1									
	By:		Jme	AS	AS	AS	AS	AS	
	Date:		1/24	1/29/01	1/29/01	1/29/01	1/29/01	1/29/01	
	Time		1752	1705	2005	2110	2206	2308	
DESCRIPTION	UNITS	Range							
<b>Priority Data</b>									
Melt Temperature (T1, control)	°C	1125 - 1175	1149	1149	1147	1143	1145	1134	
Melt Temperature (T2)	°C	1125 - 1175	1149	1145	1155	1157	1156	1146	
Plenum Temperature	°C	400 - 600	662	557	554	519	559	538	
Feed pump setting	%		0	15	15	20	20	15	
Cold Cap Coverage	%	> 75	0	70	60	70	90	70	
Slurry Pool Coverage of Cap	%		0	90	90	70	70	70	
Cold Cap Flexibility Visible	y or n		—	Y	Y	N	?	N	
Number of Vents	#		—	0	0	0	0	0	
Cold Cap Thickness	inch		—	1	1.5	1	?	1/2	
Phase Separation (Note in LRB)	y or n		—	N	N	N	N	N	
Glass Pouring	y or n		N	N	N	N	Y	N	
<b>Electrical Data</b>									
Electrode Potential	Volts		28	5.5/2.3	7.7/29.2	7.4/26.5	7.4/26.3		
Electrode Current	Amps		94	107/106	132/107	132/109	131/106		
Electrode Power	kW		2.6 calc	2.63 <sup>①</sup>	3.12 <sup>②</sup>	2.89 <sup>②</sup>	2.79 <sup>②</sup>		
Melt Resistance	Ω		0.3 calc	0.27 <sup>①</sup>	0.27 <sup>②</sup>	0.24 <sup>②</sup>	0.25 <sup>②</sup>		
Melt (Electrode) Setpoint Temp	°C		1150	1150	1150	1150	1150		
(Electrode Power) Output	%		47	45	52	46	46	55	
(Electrode) Control Mode	A or M		A	A	A	A	A	A	
Kiln Power	kW		0.9	0.3	0.1	0.3	0.0	0.0	
Kiln Temp Setpoint	°C		850	850	850	850	800	800	
Kiln Actual (Middle) Temp	°C		851	850	850	850	850	825	
Kiln Power Output	%		60	34	19	34	0	0	
Kiln Control Mode	A or M		A	A	A	A	A	A	
Discharge Can Power	kW		0.1	0.2	0.2	0.2	0.2	0.2	
Discharge Can Temp Setpoint	°C		600	750	750	750	750	750	
Discharge Can Actual Temp	°C		603	747	749	749	750	750	
Discharge Can Power Output	%		50	72	69	62	62	69	
Overflow Heater Power	kW		1.2	0.0	0.0	0.0	0	0	
Overflow Heater Setpoint	°C		1000	1050	1050	1050	1050	1050	
Overflow Heater Power Output	%		52	44	0	0	0	0	
Time hh:mm			1759	1945	2014	2115	2209	2313	

① calculated from V &amp; A fluke readings

Reviewed and Approved: AS/ASDate: 4/23/01sheet 1 of 13

## Data Sheet #1: Priority &amp; Electrical Data

RSM-01-1	By:		1/30/01	1/30/01	1/30/01	1/30/01	1/30/01	1/30/01	1/30/01	1/30/01
	Date:		1/30/01	1/30/01	1/30/01	1/30/01	1/30/01	1/30/01	1/30/01	1/30/01
	Time		00:48	01:30	02:15	03:02	04:10	05:03	06:12	7:02
DESCRIPTION	UNITS	Range								
<b>Priority Data</b>										
Melt Temperature (T1, control)	°C	1125 - 1175	1146	1150	1147	1148	1147	1149	1151	1147
Melt Temperature (T2)	°C	1125 - 1175	1155	1156	1153	1161	1169	1166	1166	1164
Plenum Temperature	°C	400 - 600	528	522	521	495	523	499	486	506
Feed pump setting	%		15	17	19%	20%	22	22	21	21
Cold Cap Coverage	%	> 75	60-40	50	50	50	60	70	60	60
Slurry Pool Coverage of Cap	%		50	50	75	60	80	80	80	80
Cold Cap Flexibility Visible	y or n		N	N	N	Y	N	Y	N	N
Number of Vents	#		2	3	2	1/2	4	0	3	0
Cold Cap Thickness	inch		1/2	1/2	1/2	1	1/4	1/2	1	1/2
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	N
<b>Electrical Data</b>										
Electrode Potential	Volts		230	270	272	27	26	25	27	26
Electrode Current	Amps		1290	1230	130	125	127	130	129	129
Electrode Power	kW		3.60	3.30	3.50	3.4	3.3	3.25	3.50	3.4
Melt Resistance	Ω		0.220	0.220	0.210	0.220	0.200	0.19	0.210	0.200
Melt (Electrode) Setpoint Temp	°C		1150	1150	1150	1150	1150	1150	1150	1150
(Electrode Power) Output	%		64	64	65	62	65	64	64	64
(Electrode) Control Mode	A or M		A	A	A	A	A	A	A	A
Kiln Power	kW		0	0	0	1.0	0.3	0.3	0.4	0.3
Kiln Temp Setpoint	°C		800	800	800	850	850	850	850	850
Kiln Actual (Middle) Temp	°C		810	809	810	862	851	850	850	850
Kiln Power Output	%		0	0	0	62	37	36	40	38
Kiln Control Mode	A or M		A	A	A	A	A	A	A	A
Discharge Can Power	kW		0.2	0.3	0.1	0.1	0.2	0.2	0.1	0.2
Discharge Can Temp Setpoint	°C		750	750	750	750	750	750	750	750
Discharge Can Actual Temp	°C		752	756	750	754	752	748	750	750
Discharge Can Power Output	%		66	78	46	43	75	63	46	62
Overflow Heater Power	kW		1.7	1.8	1.8	1.8	1.6	1.6	1.6	1.6
Overflow Heater Setpoint	°C		1050	1050	1050	1050	1050	1050	1050	1050
Overflow Heater Power Output	%		63	65	62	59	61	61	61	61
Time hh:mm			01:10	1:43	2:37	3:11	4:19	05:06	06:24	7:07

① Calculated from Fluke readings

Reviewed and Approved: 

Date: 1/30/01

sheet 2 of 13

Data Sheet #1: Priority & Electrical Data

MR. [unclear]  
 Tank  
 Start starting  
 feed again  
 feed line  
 plugging  
 problems

RSM-01-1										
By:			Sam	Sam	Sam	Sam	Sam	Sam	Sam	Sam
Date:			1/30/01	1/30/01	1/30/01	1/30/01	1/30/01	1/30/01	1/30/01	1/30/01
Time			10:00	11:20	12:14	13:18	13:58	15:05	16:01	
DESCRIPTION	UNITS	Range	09:10							
Priority Data										
Melt Temperature (T1, control)	°C	1125 - 1175	1132	1140	1140	1132	1131	1137	1140	1141
Melt Temperature (T2)	°C	1125 - 1175	1151	1156	1159	1161	1157	1154	1154	1152
Plenum Temperature	°C	400 - 600	537	509	654	691	608	632	531	513
Feed pump setting	%		20	20	20	20	20	20	20	20
Cold Cap Coverage	%	> 75	80	75	0	0	60	20	90	45
Slurry Pool Coverage of Cap	%		15	—	NA	NA	10	25	10	50
Cold Cap Flexibility Visible	y or n		Y	Y	NA	NA	Y	Y	Y	Y
Number of Vents	#		3	3	NA	NA	4	3	2	2
Cold Cap Thickness	inch		1/32	1/2	NA	NA	4/16	1/4	1/4	1/2
Phase Separation (Note in LRB)	y or n		N	N	N	N	N	N	N	N - sides
Glass Pouring	y or n		N	Y	N	N	N	N	N	N - 60s
Electrical Data										
Electrode Potential	Volts		20	20	25	28	28.7	24.5	24.9	23.6
Electrode Current	Amps		129	130	130	132	132	129	129	130
Electrode Power	kW		—	—	—	—	—	—	—	—
Melt Resistance	Ω		0.553	0.555	0.558	0.965	0.318	0.497	0.558	—
Melt (Electrode) Setpoint Temp	°C		1150	1150	1150	1150	1150	1150	1150	1150
(Electrode Power) Output	%		59	65	65	69	68	65	67	65
(Electrode) Control Mode	A or M		A	A	A	A	A	A	A	A
Kiln Power	kW		0.3	0.3	0.2	0.0	0	0.3	0.3	.3
Kiln Temp Setpoint	°C		850	850	850	850	850	850	850	850
Kiln Actual (Middle) Temp	°C		850	850	858	860	843	858	851	850
Kiln Power Output	%		34	37	31	0	11	32	38	37
Kiln Control Mode	A or M		A	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	.1
Discharge Can Temp Setpoint	°C		750	750	750	750	760	750	750	750
Discharge Can Actual Temp	°C		738	741	749	750	747	754	756	752
Discharge Can Power Output	%		67	62	71	71	70	63	80	57
Overflow Heater Power	kW		1.5	1.2	1.2	1.2	1.8	1.6	1.6	1.6
Overflow Heater Setpoint	°C		1050	1050	1050	1050	1050	1050	1050	1050
Overflow Heater Power Output	%		59	65	65	65	69	61	63	61
Time hh:mm			09:24	10:16	11:28	12:22	13:22	14:14	15:09	16:07

Reviewed and Approved: 

Date: 2/1/01

sheet 3 of 4



Data Sheet #1: Priority &amp; Electrical Data

RSM-01-1	By:		OMS	OMS	AMS	AMS	AMS	AMS	AMS	AMS
	Date:		1/30	1/30	1/30	1/30	1/30	1/30	1/30	1/30
	Time		16:24	15:54	16:56	17:54	18:57	20:02	21:00	22:02
DESCRIPTION	UNITS	Range								
<b>Priority Data</b>										
Melt Temperature (T1, control)	°C	1125 - 1175	1142	1134	1145	1144	1145	1143	1132	1142
Melt Temperature (T2)	°C	1125 - 1175	1158	1162	1174	1161	1157	1163	1153	1178
Plenum Temperature	°C	400 - 600	570	542	540	510	492	526	690	613
Feed pump setting	%		20%	20%	21%	21%	22%	20%	20%	20%
Cold Cap Coverage	%	> 75	~60%	80%	80%	85	90	25%	60%	20%
Slurry Pool Coverage of Cap	%		~50%	50%	50%	60	50	70%	70%	70%
Cold Cap Flexibility Visible	y or n		Y	Y	Y	Y	Y	Y	Y	Y
Number of Vents	#		> 5	> 5	> 5	4	2	1	0	0
Cold Cap Thickness	inch		0.5	0.5	0.5	.5	.5	.25	.25	.25
Phase Separation (Note in LRB)	y or n		Y	STABLE	STABLE	Stable	STABLE	Not Visible	NO NOT V.S.	NO NOT V.S.
Glass Pouring	y or n		N	Y	Y	N	N	N	N	N
<b>Electrical Data</b>										
Electrode Potential	Volts		24.7	28.0	27.5	24.5	24.0	27.7	37.1	28.9
Electrode Current	Amps		121.9	141.6	140.4	137.4	139.6	138.8	138.6	126.5
Electrode Power	kW		3.00	3.96	3.86	3.37	3.35	3.84	5.84	3.66
Melt Resistance	Ω		0.20	0.20	0.20	0.18	0.17	0.20	0.27	0.23
Melt (Electrode) Setpoint Temp	°C		1150	1150	1150	1150	1150	1150	1150	1140
(Electrode Power) Output	%		70	59	69	69	69	69	76	57
(Electrode) Control Mode	A or M		A	A	A	A	A	A	A	A
Kiln Power	kW		0.3	0.3	0.3	0.3	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		849	848	850	849	852	848	858	848
Kiln Power Output	%		35	34	34	33	30	31	30	27
Kiln Control Mode	A or M		A	A	A	A	A	A	A	A
Discharge Can Power	kW		0.2	0.1	0.1	0.1	0.2	0.1	0.2	0.2
Discharge Can Temp Setpoint	°C		750	750	750	750	750	750	750	750
Discharge Can Actual Temp	°C		752	747	733	747	749	761	747	753
Discharge Can Power Output	%		60	57	37	51	70	41	69	67
Overflow Heater Power	kW		1.6	1.6	1.6	1.6	1.6	1.3	1.8	1.6
Overflow Heater Setpoint	°C		1050	1050	1050	1050	1050	1050	1050	1050
Overflow Heater Power Output	%		1050	1052	1049	1050	1050	1053	1054	1052
Time	hh:mm		16:28	16:54	17:57	19:00	20:24	21:06	22:04	23:05

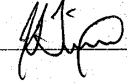
Reviewed and Approved:

Date: 2/01/01

sheet 4 of 12

Data Sheet #1: Priority &amp; Electrical Data

RSM-01-1									
	By:		100	020	000/11	020	111	020	WCB
	Date:		1/31/01	1/31/01	1/31/01	1/31/01	1/31/01	1/31/01	1/31/01
	Time		00:15	01:01	02:09	03:03	~402	5:00	6:20
DESCRIPTION	UNITS	Range							
<b>Priority Data</b>									
Melt Temperature (T1, control)	°C	1125 - 1175	1159	1142	1158	1144	1150	1105	1145
Melt Temperature (T2)	°C	1125 - 1175	1147	1169	1161	1160	1166	1131	1164
Plenum Temperature	°C	400 - 600	751	702	516	458	493	638	611
Feed pump setting	%		0	20	25	25	25	10%	20
Cold Cap Coverage	%	> 75	0	0	100	100	0 cold cap buried	0 - feed line plugged	40
Slurry Pool Coverage of Cap	%		0	0	20	20	0	100	100
Cold Cap Flexibility Visible	y or n		-	-	Y	N	-	-	Y
Number of Vents	#		-	-	106	3	-	-	2
Cold Cap Thickness	inch		-	-	1/4 - 1/2	casted	-	-	1/4"
Phase Separation (Note in LRB)	y or n		Y small amt	Y	N	-	-	-	N
Glass Pouring	y or n		N	N	drips	Y	N	-	N
<b>Electrical Data</b>									
Electrode Potential	Volts		26	28	25	26	25	27	25
Electrode Current	Amps		125	132	138	141	137	119	132.3
Electrode Power	kW		3.25	3.70	3.45	3.67	3.15	3.21	3.31
Melt Resistance	Ω		0.21	0.21	0.18	0.18	0.17	0.23	0.19
Melt (Electrode) Setpoint Temp	°C		1140	1140	1150	1150	150	1140	1140
(Electrode Power) Output	%		60	66	69	69	69	60	65
(Electrode) Control Mode	A or M		A	A	A	A	A	A	A
Kiln Power	kW		0	0	0.5	0.3	0.3	0	0.2
Kiln Temp Setpoint	°C		850	850	850	850	850	850	850
Kiln Actual (Middle) Temp	°C		858	860	852	850	850	846	852
Kiln Power Output	%		0	0	44	36	36	13	28
Kiln Control Mode	A or M		A	A	A	A	A	A	A
Discharge Can Power	kW		0.2	0.2	0.2	0.1	0.2	0.2	0.2
Discharge Can Temp Setpoint	°C		750	750	750	750	750	750	750
Discharge Can Actual Temp	°C		750	749	742	741	747	749	749
Discharge Can Power Output	%		67	67	62	54	71	63	68
Overflow Heater Power	kW		1.6	1.6	1.6	1.3	1.6	1.6	1.6
Overflow Heater Setpoint	°C		1050	1050	1050	1050	1050	1050	1050
Overflow Heater Power Output	%		61	61	61	56	61	62	62
Time hh:mm			00:25	01:06	02:15	03:07		6:34	7:22


Reviewed and Approved: 

Date: 1/31/01

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Data Sheet #1: Priority &amp; Electrical Data

RSM-01-1										
	By:		54	58	54	54	54	54	Sam	54
	Date:		1/31/01	1/31/01	1/31/01	1/31/01	1/31/01	1/31/01	1/31/01	1/31/01
	Time		0823	0919	1006	1103	1229	1305	1355	1455
DESCRIPTION	UNITS	Range								
Priority Data										
Melt Temperature (T1, control)	°C	1125 - 1175	1138	1150	1150	1150	1149	1145	1149	1151
Melt Temperature (T2)	°C	1125 - 1175	1155	1164	1166	1161	1168	1164	1156	1164
Plenum Temperature	°C	400 - 600	545	532	530	529	691	613	568	537
Feed pump setting	%		20	20	20	20	20	20	20	20
Cold Cap Coverage	%	> 75	75	80	75	60	25	60	50	75
Slurry Pool Coverage of Cap	%		50	50	30	30	25	90	50	50
Cold Cap Flexibility Visible	y or n		y	y	y	y	y	y	y	y
Number of Vents	#		4	7-8	4-5	4-7	—	6-7	6	6
Cold Cap Thickness	inch		1/2	1/2	1/2	1/2	—	1/2	<1/2	1/2
Phase Separation (Note in LRB)	y or n		No	No	No	No	No	No	no	No
Glass Pouring	y or n		No	No	No	No	No	No	no	No
Electrical Data										
Electrode Potential	Volts		25.4	24.2	24.5	24.3	26.2	25.4	25.5	24.6
Electrode Current	Amps		136.2	145.9	145.0	142.3	148.4	148.6	143.0	139.2
Electrode Power	kW		43.9	3.5	3.6	3.46	3.89	3.77	3.64	3.42
Melt Resistance	Ω		0.166	0.166	0.164	0.171	0.177	0.171	0.178	0.177
Melt (Electrode) Setpoint Temp	°C		1150	1150	1150	1150	1150	1150	1150	1150
(Electrode Power) Output	%		74	73	72	70	74	74	71	69
(Electrode) Control Mode	A or M		A	A	A	A	A	A	A	A
Kiln Power	kW		0.2	0.2	0.2	0.2	0.2	0	0.3	0.3
Kiln Temp Setpoint	°C		850	850	850	850	850	850	850	850
Kiln Actual (Middle) Temp	°C		850	850	850	850	855	848	851	850
Kiln Power Output	%		31	28	31	30	3	5	33	32
Kiln Control Mode	A or M		A	A	A	A	A	A	A	A
Discharge Can Power	kW		0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Discharge Can Temp Setpoint	°C		750	750	750	750	750	750	750	750
Discharge Can Actual Temp	°C		750	750	747	749	749	750	750	750
Discharge Can Power Output	%		67	63	72	63	64	65	61	66
Overflow Heater Power	kW		1.6	1.6	1.6	1.6	1.6	1.6	1.6	1.6
Overflow Heater Setpoint	°C		1050	1050	1050	1050	1050	1050	1050	1050
Overflow Heater Power Output	%		62	60	61	61	62	61	61	62
Time	hh:mm		0814	0926	1008	1107	1230	1309	1405	1458

Reviewed and Approved: 

Date: 2/1/01

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Data Sheet #1: Priority &amp; Electrical Data

RSM-01-1										
	By:		SL	LC	am	LO	AS	DM	BS	BS
	Date:		1/31/01	1/31/01	1/31/01	1/31/01	1/31/01	1/31/01	1/31/01	1/31/01
	Time		1554	1700	1756	1904	2007	2101	2203	2306
DESCRIPTION	UNITS	Range								
<b>Priority Data</b>										
Melt Temperature (T1, control)	°C	1125 - 1175	1142	1151	1151	1149	1147	1149	1150	1143
Melt Temperature (T2)	°C	1125 - 1175	1166	1164	1156	1161	1158	1158	1160	1159
Plenum Temperature	°C	400 - 600	676	566	522	509	514	515	510	630
Feed pump setting	%		22	22	22	22	22	22	22	0
Cold Cap Coverage	%	> 75	75	65	75	70	75	880	70	0
Slurry Pool Coverage of Cap	%		60	70	60	50	60	60	60	—
Cold Cap Flexibility Visible	y or n		Y	Y	Y	Y	Y	Y	Y	—
Number of Vents	#		3	Edgewise	2	2	3	2	2	—
Cold Cap Thickness	Inch		1/2	~ 1/2	~ 1/2	1/2	1/2	1/2	1/2	—
Phase Separation (Note in LRB)	y or n		N	small	N	settled	N	N.D.	N.D.	Y
Glass Pouring	y or n		N	N	N	N	N	N	N	N
<b>Electrical Data</b>										
Electrode Potential	Volts		24.4	22.8	24.0	24.4	24.3	24.3	24.3	24.8
Electrode Current	Amps		147.1	139	143.1	146	147	145	147	141
Electrode Power	kW		3.7	3.3	3.4	3.6	3.6	3.5	3.6	3.5
Melt Resistance	Ω		0.173	0.171	0.17	0.17	0.17	0.17	0.16	0.18
Melt (Electrode) Setpoint Temp	°C		1150	1150	1150	1150	1150	1150	1150	1150
(Electrode Power) Output	%		73	70	71	73	74	72	72	66
(Electrode) Control Mode	A or M		A	A	A	A	A	A	A	A
Kiln Power	kW		0	0.3	0.2	0.3	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		855	851	850	850	850	850	850	856
Kiln Power Output	%		0	36	30	33	32	31	31	29
Kiln Control Mode	A or M		A	A	A	A	A	A	A	A
Discharge Can Power	kW		0.2	0.2	0.2	0.2	0.2	0.2	0.2	0
Discharge Can Temp Setpoint	°C		750	750	750	750	750	750	750	750
Discharge Can Actual Temp	°C		623	752	751	750	747	749	749	764
Discharge Can Power Output	%		70	75	61	69	72	66	61	5
Overflow Heater Power	kW		1.7	1.6	1.6	1.6	1.6	1.6	1.6	1.6
Overflow Heater Setpoint	°C		1050	1050	1052	1051	1050	1050	1050	1050
Overflow Heater Power Output	%		63	62	61	61	62	1051	61	62
Time hh:mm			1605	17:06	17:59	19:10	2009	2101	2212	2316

Reviewed and Approved: 

Date: 2/1/01

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## Data Sheet #1: Priority &amp; Electrical Data

RSM-01-1										
	By:		1508	1400	141	1500	1508	1500	1500	1500
	Date:		2/1/01	2/1/01	2/1/01	2/1/01	2/1/01	2/1/01	2/1/01	2/1/01
	Time		0:09	0:00	0:03	3:01	3:02	05:00	06:07	07:11
DESCRIPTION	UNITS	Range								
Priority Data										
Melt Temperature (T1, control)	°C	1125 - 1175	1149	1081	1149	1151	1149	1150	1150	1091
Melt Temperature (T2)	°C	1125 - 1175	1165	1105	1158	1150	1152	1168	1157	1095
Plenum Temperature	°C	400 - 600	761	723	573	495	460	480	492	600
Feed pump setting	%		0.0	off	25	25	25	25	25	25
Cold Cap Coverage	%	> 75	0	0	40%	70%	90%	100	100	60
Slurry Pool Coverage of Cap	%				50	50	50	20	conf	90
Cold Cap Flexibility Visible	y or n				Y	Y	Y	N	conf	Y
Number of Vents	#				105	No	None	0	0	4
Cold Cap Thickness	inch				1"	1	1	1	1	1/2
Phase Separation (Note in LRB)	y or n		Y	Feed off	Y	Y	Y	N	conf	N
Glass Pouring	y or n		N		N	N	N	Y	N	N
Electrical Data										
Electrode Potential	Volts		23.7	27.1	25	24.9	22.9	22.1	24.3	29.2
Electrode Current	Amps		127.4	130.3	144	147	137	143	141	149
Electrode Power	kW		3.02	3.53	3.60	3.67	3.14	3.16	3.43	4.3
Melt Resistance	Ω		0.19	0.21	0.174	0.169	0.17	0.16	0.17	0.2
Melt (Electrode) Setpoint Temp	°C		1150	1050	1050	1150	1150	1150	1150	1150
(Electrode Power) Output	%		64	62	72	74	70	70	70	80
(Electrode) Control Mode	A or M		A	A	A	A	A	A	A	A
Kiln Power	kW		0.0	0.0	0.0	0.3	0.3	0.3	0.3	0.0
Kiln Temp Setpoint	°C		850	850	850	850	850	850	850	850
Kiln Actual (Middle) Temp	°C		865	857	844	851	850	850	850	848
Kiln Power Output	%		0	0	8	34	36	37	38	1
Kiln Control Mode	A or M		A	A	A	A	A	A	A	A
Discharge Can Power	kW		0.2	0.2	0.2	0.2	0.1	0.2	0.2	0.2
Discharge Can Temp Setpoint	°C		750	750	750	750	750	750	750	750
Discharge Can Actual Temp	°C		749	750	750	751	745	752	751	750
Discharge Can Power Output	%		67	67	66	63	49	59	67	66
Overflow Heater Power	kW		1.5	1.6	1.6	1.6	1.6	1.6	1.6	1.7
Overflow Heater Setpoint	°C		1050	1050	1050	1050	1050	1050	1050	1050
Overflow Heater Power Output	%		61	62	62	61	62	62	62	63
Time	hh:mm		0:15	1:03	—	3:10	4:10	5:07	06:10	07:14

Reviewed and Approved:

Date: 2/1/01

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Data Sheet #1: Priority &amp; Electrical Data

shut down  
for part  
addition  
new  
batch,  
feed  
back  
in

RSM-01-1										
	By:		JAM	JAM	JAM	JAM	JAM	JAM	JAM	JAM
	Date:		2/1/01	2/1/01	2/1/01	2/1/01	2/1/01	2/1/01	2/1/01	2/1/01
	Time		8:29	8:04	10:29	10:59	12:22	13:00	15:13	16:01
DESCRIPTION	UNITS	Range								
<b>Priority Data</b>										
Melt Temperature (T1, control)	°C	1125 - 1175	1149	1150	1150	1149	1150	1149	1149	1149
Melt Temperature (T2)	°C	1125 - 1175	1159	1157	1160	1163	1163	1152	1157	1152
Plenum Temperature	°C	400 - 600	626	563	547	727	691	558	489	499
Feed pump setting	%		25	25	20	25	25	23	23	23
Cold Cap Coverage	%	> 75	60	90	60	00	50	70	80	90
Slurry Pool Coverage of Cap	%		50	25	—	—	50	30	70	50
Cold Cap Flexibility Visible	y or n		Y	Y	—	—	Y	Y	Y	Y
Number of Vents	#		~5	3-4	—	—	0	4-5	4-5	3
Cold Cap Thickness	inch		< 1/2	~ 1/2	—	—	1/2	1/2	1/2	3/4
Phase Separation (Note in LRB)	y or n		Y	Y	Y	Y	Y	Y	Y	Y
Glass Pouring	y or n		N	N	N	N	N	N	N	N
<b>Electrical Data</b>										
Electrode Potential	Volts		24.7	24	22.7	22.5	24.2	23.2	22.7	23.6
Electrode Current	Amps		152	157	155	153	149	140	150	158.5
Electrode Power	kW		3.75	3.8	3.5	3.4	3.6	3.3	3.4	3.7
Melt Resistance	Ω		0.16	0.15	0.15	0.15	0.16	0.17	0.15	0.15
Melt (Electrode) Setpoint Temp	°C		1150	1150	1150	1150	1150	1150	1150	1150
(Electrode Power) Output	%		75	75	77	76	73	69	72	74
(Electrode) Control Mode	A or M		A	A	A	A	A	A	A	A
Kiln Power	kW		0	0.2	0.2	0	0	0.1	0.3	-3
Kiln Temp Setpoint	°C		850	850	850	850	850	850	850	850
Kiln Actual (Middle) Temp	°C		850	844	851	860	867	841	850	850
Kiln Power Output	%		8	26	29	3	0	16	33	32
Kiln Control Mode	A or M		A	A	A	A	A	A	A	A
Discharge Can Power	kW		0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Discharge Can Temp Setpoint	°C		750	750	750	750	750	750	750	750
Discharge Can Actual Temp	°C		754	752	752	747	750	750	749	749
Discharge Can Power Output	%		63	70	71	67	71	70	69	68
Overflow Heater Power	kW		1.6	1.6	1.6	1.6	1.6	1.7	1.6	1.6
Overflow Heater Setpoint	°C		1050	1050	1050	1050	1050	1050	1050	1050
Overflow Heater Power Output	%		62	61	62	61	62	63	61	61
Time hh:mm			8:42	09:13	10:34	11:03	12:26	13:11	15:20	16:03

Reviewed and Approved: ASDate: 2/1/01sheet 9 of 13



Data Sheet #1: Priority &amp; Electrical Data

RSM-01-1										
	By:		MT	MT	LO	PD	MT	PD	MT	
	Date:		2/1/01	2/1/01	2/1/01	2/1/01	2/1/01	2/1/01	2/1/01	
	Time		1705	1800	1856		2100	2158	2300	
DESCRIPTION	UNITS	Range								
<b>Priority Data</b>										
Melt Temperature (T1, control)	°C	1125 - 1175	1027	1075	1149	1149	1150	1154	1150	
Melt Temperature (T2)	°C	1125 - 1175	1037	1079	1149	1145	1147	1150	1145	
Plenum Temperature	°C	400 - 600	592	687	609	592	556	539	54501	
Feed pump setting	%		0	0	20	20	21	21	21	
Cold Cap Coverage	%	> 75	0	0	50	50	50	60	60	
Slurry Pool Coverage of Cap	%		—	—	?	~100	100	100	100	
Cold Cap Flexibility Visible	y or n		—	—	Y	Y	Y	Y	Y	
Number of Vents	#		—	—	2	2	SIDES	None	MANY BUBBLES	
Cold Cap Thickness	inch		—	—	< 1/2	~1/2	1/2	1/2	1/2	
Phase Separation (Note in LRB)	y or n		Y	Y	Y	Y	YES	decreasing	YES	
Glass Pouring	y or n		N	N	N	N	NO	No	N	
<b>Electrical Data</b>										
Electrode Potential	Volts		24.1	24.5	24.7	24.4	23.4	22.5	23	
Electrode Current	Amps		29.8	29	159	153	146	125	142	
Electrode Power	kW		4.2	4.3	3.9	3.7	3.4	2.8	3.3	
Melt Resistance	Ω		0.21	0.193	0.16	0.16	0.16	0.18	0.16	
Melt (Electrode) Setpoint Temp	°C		1150	1150	1150	1150	1150	1150	1150	
(Electrode Power) Output	%		70	75	78	77	71	64	72	
(Electrode) Control Mode	A or M		M	M	A	A	A	A	A	
Kiln Power	kW		9.7	0.1	0	0.1	0.2	0.3	0.3	
Kiln Temp Setpoint	°C		850	850	850	850	850	850	850	
Kiln Actual (Middle) Temp	°C		851	848	851	850	848	848	849	
Kiln Power Output	%		41	18	11	22	26	32	34	
Kiln Control Mode	A or M		A	A	A	A	A	A	A	
Discharge Can Power	kW		0.2	2.7	0.2	0.2	0.2	0.2	2.7	
Discharge Can Temp Setpoint	°C		750	750	750	750	750	750	750	
Discharge Can Actual Temp	°C		756	749	748	745	750	747	749	
Discharge Can Power Output	%		63	66	68	76	64	64	66	
Overflow Heater Power	kW		1.7	1.7	1.6	1.7	1.7	1.7	1.7	
Overflow Heater Setpoint	°C		1050	1050	1050	1050	1050	1050	1050	
Overflow Heater Power Output	%		64	64	63	63	63	63	63	
Time	hh:mm		1708	1805	1906	2007	2105	2202	2302	

Reviewed and Approved: RV [Signature]Date: 4/23/01sheet 10 of 13

Data Sheet #1: Priority &amp; Electrical Data

RSM-01-1										
	By:		ONS	ONS	MS	MS	MS	MS	MS	MS
	Date:		2/2/01	2/2/01	2/2/01	2/2/01	2/2/01	2/2/01	2/2/01	2/2/01
	Time		00:22	00:10	04:00	03:05	04:03	05:05	06:05	07:17
	UNITS	Range								
Priority Data										
Melt Temperature (T1, control)	°C	1125 - 1175	1147	1149	1156	1149	1150	1154	1159	1148
Melt Temperature (T2)	°C	1125 - 1175	1147	1148	1152	1144	1149	1152	1147	1147
Plenum Temperature	°C	400 - 600	717	756	667	535	483	422	490	495
Feed pump setting	%		0	0	22	25	25	25	25	25
Cold Cap Coverage	%	> 75	None	Feed	23	40	90%	90%	100%	100%
Slurry Pool Coverage of Cap	%		Feed	off.	90	60	20	20	25%	20%
Cold Cap Flexibility Visible	y or n		off	maxing	y	y	y	y	y	y
Number of Vents	#		for feed	max	3	2	0	4	4	2
Cold Cap Thickness	inch		mechanical	Feed	1/4	1	1-4 in	1	1/4 - 2"	1/4 - 2"
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		23.1	23.8	25	24.4	24	22.2	23	25.2
Electrode Current	Amps		136.3	134.33	140	149.8	157	142.5	143	153
Electrode Power	kW		3.1	3.2	3.2	3.7	3.6	3.1	3.3	2.9
Melt Resistance	Ω		0.17	0.18	0.16	0.16	0.16	0.16	0.16	0.16
Melt (Electrode) Setpoint Temp	°C		1150	1150	1150	1150	1150	1150	1150	1150
(Electrode Power) Output	%		68	67	70	74	75	70	76	79
(Electrode) Control Mode	A or M		A	A	A	A	A	A	A	A
Kiln Power	kW		0	0	0	0.3	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		860	867	851	848	850	851	850	851
Kiln Power Output	%		13	0	13	36	32	32	31	30
Kiln Control Mode	A or M		A	A	A	A	A	A	A	A
Discharge Can Power	kW		0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.7
Discharge Can Temp Setpoint	°C		750	750	750	750	750	750	750	750
Discharge Can Actual Temp	°C		750	750	750	752	750	750	749	749
Discharge Can Power Output	%		65	67	68	71	63	62	72	71
Overflow Heater Power	kW		1.6	1.5	1.7	1.7	1.6	1.6	1.6	1.6
Overflow Heater Setpoint	°C		1050	1050	1050	1050	1050	1050	1050	1050
Overflow Heater Power Output	%		62	61	63	63	61	62	62	62
Time	hh:mm		00:25	01:03	02:12	03:10	04:05	05:09	06:07	07:23

Reviewed and Approved: Re. G. G.Date: 4/23/01sheet 11 of 13

Data Sheet #1: Priority &amp; Electrical Data

RSM-01-1	By:		Jan	183	Jan	Jan	Jan	Jan	Jan	Jan
	Date:		2/2/01	2/2/01	2/2/01	2/2/01	2/2/01	2/2/01	2/2/01	2/2/01
	Time		8:12	8:16	10:05	11:11	12:10	1300	1400	16:05
DESCRIPTION	UNITS	Range								
<b>Priority Data</b>										
Melt Temperature (T1, control)	°C	1125 - 1175	1145	1154	1149	1152	1150	1150	1148	1147
Melt Temperature (T2)	°C	1125 - 1175	1149	1156	1154	1154	1150	1149	1150	1145
Plenum Temperature	°C	400 - 800	500	705	761	780	575	507	991	991
Feed pump setting	%		25	0	0	0	30 *	28	27.5	27.5
Cold Cap Coverage	%	> 75	20	0	0	0	70	100	90	90
Slurry Pool Coverage of Cap	%		30	0	—	—	30	25	30	25
Cold Cap Flexibility Visible	y or n		Y	—	—	—	Y	Y	Y	Y
Number of Vents	#		4-6	—	—	—	5-6	0	6-7	6-7
Cold Cap Thickness	inch		1/2	—	—	—	1/4	1/4-3'	1/4-1/2	1/4-1/2
Phase Separation (Note in LRB)	y or n		N	Y	Y	Y	Y	N	N	N
Glass Pouring	y or n		N	N	—	—	N	N	N	N
<b>Electrical Data</b>										
Electrode Potential	Volts		15825	24	23	22.6	25.1	24	29.2	29.4
Electrode Current	Amps		153	144	143	136	145	143	154	153
Electrode Power	kW									
Melt Resistance	Ω									
Melt (Electrode) Setpoint Temp	°C		1150	1150	1150	1150	1150	1150	1150	1150
(Electrode Power) Output	%		79	71	71	68	67	69	77	77
(Electrode) Control Mode	A or M		A	A	A	A	A	A	A	A
Kiln Power	kW		0.2	0.2	0	0	0	0.3	0.2	0.3
Kiln Temp Setpoint	°C		850	850	850	850	850	850	850	850
Kiln Actual (Middle) Temp	°C		851	853	872	879	866	844	850	851
Kiln Power Output	%		30	17	0	0	0	35	30	34
Kiln Control Mode	A or M		A	A	A	A	A	A	A	A
Discharge Can Power	kW		0.2	0.2	0.2	0.2	0.2	0.2	0.1	0.2
Discharge Can Temp Setpoint	°C		750	750	750	750	750	750	750	750
Discharge Can Actual Temp	°C		755	740	750	750	751	752	747	750
Discharge Can Power Output	%		72	71	70	73	67	66	59	65
Overflow Heater Power	kW		1.6	1.6	1.5	1.5	1.6	1.7	1.6	1.6
Overflow Heater Setpoint	°C		1050	1050	1050	1050	1050	1050	1050	1050
Overflow Heater Power Output	%		61	62	61	61	61	61	62	62
Time hh:mm			8:19	8:18	10:02	11:13	12:17	1302	14:04	16:10

Reviewed and Approved: *RW*Date: 4/23/01  
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sheet 12 of 13



Data Sheet #1: Priority & Electrical Data

feed just  
turned back  
on

RSM-01-1									
	By:		16	16	JAM	16			
	Date:		2/2/01	2/2/01	2/2/01	2/2/01			
	Time		17:22	18:03	18:59	20:03			
DESCRIPTION	UNITS	Range							
<b>Priority Data</b>									
Melt Temperature (T1, control)	°C	1125 - 1175	1151	1151	1147	1153			
Melt Temperature (T2)	°C	1125 - 1175	1153	1149	1147	1154			
Plenum Temperature	°C	400 - 600	471	606	491	472			
Feed pump setting	%		27.5	25	25	25			
Cold Cap Coverage	%	> 75	> 95	5.1	95	> 95			
Slurry Pool Coverage of Cap	%		~ 70	100	50	too dark			
Cold Cap Flexibility Visible	y or n		not visible	Y	Y	not sure			
Number of Vents	#		0-1	lots	6-7	0			
Cold Cap Thickness	inch		~ 1/2	0	1/2 - 1/2	unknown			
Phase Separation (Note in LRB)	y or n		Y	Y	?	?			
Glass Pouring	y or n		drips	N	N	N			
<b>Electrical Data</b>									
Electrode Potential	Volts		23.0	23.5	22.8	20.4			
Electrode Current	Amps		154	152	156	134			
Electrode Power	kW		3.5	3.6	3.6				
Melt Resistance	Ω		0.15	0.15					
Melt (Electrode) Setpoint Temp	°C		1150	1150	1150	1150			
(Electrode Power) Output	%		76	76	75	66			
(Electrode) Control Mode	A or M		A	A	A	A			
Kiln Power	kW		0.3	0.2	0.3	0.3			
Kiln Temp Setpoint	°C		850	850	850	850			
Kiln Actual (Middle) Temp	°C		850	853	849	850			
Kiln Power Output	%		32	30	36	37			
Kiln Control Mode	A or M		A	A	A	A			
Discharge Can Power	kW		700	0.1	0.2	0.2			
Discharge Can Temp Setpoint	°C		750	750	750	750			
Discharge Can Actual Temp	°C		751	750	750	746			
Discharge Can Power Output	%		60	48	66	66			
Overflow Heater Power	kW		1.6	1.7	1.6	1.6			
Overflow Heater Setpoint	°C		1050	1050	1050	1050			
Overflow Heater Power Output	%		61	63	62	62			
Time hh:mm			17:22	18:06	19:06	20:05			

C.26

Reviewed and Approved: RW Gault

Date: 4/23/01

sheet 13 of 13

## **Data Sheet #2: Routine Status Sheets**

## Data Sheet #2: Routine Status Sheet

RSM-01-1

			By:	HBS	BT	MT	MT	MT	MT	MT	MT	
			Date:	1/29/01	1/29/01	1/29/01	1/29/01	1/29/01	1/29/01	1/29/01	1/30/01	
			Time:	17:52	19:13	19:56	20:00	22:00	23:00	23:00	20:24	
DESCRIPTION	units	range										
Feed Nozzle Cooling Flow	gpm	.5 - 1 (reg FNT)	0.5	0.45	0.45	0.45	0.45	0.45	0.45	0.45	0.45	
Film Cooler Air Supply	scfm	1-10 (reg OGT)	8	7.6	7.5	7.5	7.5	7.5	7.4	7.4	7.4	
Melter Vacuum-Magnehelic	in. H <sub>2</sub> O	0.1 - 2.0	1.0	0.96	0.95	0.97	0.97	0.92	0.99	0.99	0.7	
EVS ΔP	in. H <sub>2</sub> O		-0.023	-0.0156	0.02	0.01	0.01	0.01	0.004	0.004		
Film Cooler ΔP	in. H <sub>2</sub> O		0.01	0.047	0.039	0.05	0.05	0.05	0.09	0.09		
HEME ΔP	in. H <sub>2</sub> O		0.60	0.64	0.609	0.67	0.66	0.66	0.78	0.78		
System ΔP	in. H <sub>2</sub> O		0.98	1.05	1.04	1.13	1.09	1.09	1.15	1.15		
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	3.7	3.7	3.7	3.7	3.7	3.7	3.7	3.7		
EVS Scrub Tank Volume	gallon	35 - 50	22	22	22	22	22	23	24	24		
EVS Nozzle Pressure	psi	50 - 55	55	55	55	55	55	55	55	55		
EVS Scrub Solution pH	pH	> 9	12	12	12	12	11	11	11	11		
OG Control Valve position	% closed		50	50	50	50	55	55	55	55		
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	0-10		5	5	5	5	5	5	5	5		
Blower Cooling Flow	gpm	1 - 1.5	1.15	1.15	1.15	1.15	1.15	1.15	1.15	1.15		
Feed Nozzle Temp (FNT)	°C	< 40	20	20	22	20	21	20	20	20		
Off-Gas Temp (OGT)	°C	< 250	79	55	88	88	83	83	80	80		
Post EVS Off-Gas Temp	°C	< 50	25	25	30	27	26	25	25	25		
Scrub Liquid Temp (SLT)	°C	< 40	16	18	17	18	17	17	17	17		
Heat Xer Temp	°C	< 30	15	15	17	16	15	15	15	15		
Feed Pump Setting	%		15	15	15	20	20	20	15	15		
Feed Pump Control Mode	A or M		A	A	A	A	A	A	A	A		
Feed Pressure	psi	< 1.0	0.6	0	0	0	0.3	0.1	0.1	0.1		
Feed Tank Wt	Kg	decreasing	67.8	65.5	65.5	61.0	60.0	57.0	57.0	57.0		
Overflow Temp	°C	1000 - 1100	1000	1050	1082	1083	1084	1069	1069	1069		
Discharge Can Temp	°C	750 - 850	602	752	752	752	752	748	748	748		
Glass Scale	Kg	< 10	—	—	—	—	—	—	—	—		
Alarm Condition	On/Off	off		OFF	OFF	OFF	OFF	OFF	OFF	OFF		
time hh:mm												

Starts new parts

Reviewed and Approved: Red InkDate: 4/23/01sheet 1 of 13



## Data Sheet #2: Routine Status Sheet

RSM-01-1

		By:	OK	1/30/01	1/30/01	1/30/01	1/30/01	1/30/01	1/30/01	1/30/01
		Date:	1/30/01	1/30/01	1/30/01	1/30/01	1/30/01	1/30/01	1/30/01	1/30/01
		Time:	50:24	1:11	2:06	3:00	4:00	5:03	6:01	7:03
DESCRIPTION	units	range								
Feed Nozzle Cooling Flow	gpm	.5 - 1 (reg FNT)	0.45	0.47	0.47	0.47	0.47	0.47	0.47	0.47
Film Cooler Air Supply	scfm	1-10 (reg OGT)	7.4	7.3	7.3	7.3	7.3	7.3	7.3	7.3
Melter Vacuum-Magnehelic	in. H <sub>2</sub> O	0.1 - 2.0	0.7	0.6	0.7	0.7	0.7	0.7	0.7	0.7
EVS ΔP	in. H <sub>2</sub> O		0	0	0	0	0	0	0	0
Film Cooler ΔP	in. H <sub>2</sub> O		0.070	0.074	0.062	0.082	0.086	0.094	0.129	0.098
HEME ΔP	in. H <sub>2</sub> O		0.73	0.73	0.72	0.76	0.77	0.78	0.81	0.77
System ΔP	in. H <sub>2</sub> O		1.2	1.17	1.20	1.23	1.23	1.23	1.25	1.24
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	3.7	3.7	3.7	3.7	3.7	3.7	3.7	3.7
EVS Scrub Tank Volume	gallon	35 - 50	25	25	25	25	25	25	25	26
EVS Nozzle Pressure	psi	50 - 55	48	55	55	55	55	55	55	55
EVS Scrub Solution pH	pH	> 9	16	16	9.5	9	10	10	10	9
OG Control Valve position	% closed		50	50	50	50	50	50	45	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	0-10		5	5	5	5	5	5	5	5
Blower Cooling Flow	gpm	1 - 1.5	1.15	1.15	1.15	1.15	1.15	1.15	1.15	1.15
Feed Nozzle Temp (FNT)	°C	< 40	22	21	20	21	19	20	22	21
Off-Gas Temp (OGT)	°C	< 250	77	89	84	86	70	86	86	92
Post EVS Off-Gas Temp	°C	< 50	26	28	27	29	27	30	30	29
Scrub Liquid Temp (SLT)	°C	< 40	18	18	17	18	18	18	18	18
Heat Xer Temp	°C	< 30	15	16	15	15	15	17	17	17
Feed Pump Setting	%		15	20	19	20	20	22	21	21
Feed Pump Control Mode	A or M		A	A	A	A	A	A	A	A
Feed Pressure	psi	< 1.0	0.2	0.0	0.0	0.0	0.0	0.0	0.1	0.3
Feed Tank Wt	Kg	decreasing	52.8	51.5	48.8	45.2	42.9	39.5	35.8	32.2
Overflow Temp	°C	1000 - 1100	1042	1050	1048	1048	1051	1051	1048	1049
Discharge Can Temp	°C	750 - 850	754	747	749	752	745	749	749	750
Glass Scale	Kg	< 10	2.82	3.19	3.16	4.63	5.36	5.77	6.65	7.43
Alarm Condition	On/Off	off	OFF	OFF	OFF	OFF	OFF	OFF	OFF	OFF
time hh:mm				1:22	2:15	3:14	4:08	5:15	6:11	6:12

Reviewed and Approved:

Date: 1/30/01

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C.29

## Data Sheet #2: Routine Status Sheet

Just turned  
off feed to add/mix  
more feed.

Just started  
feeding  
as com.

RSM-01-1

			By:	J. McCreary	RP	J. McCreary	S. McCreary	SD	RP	RP
			Date:	11/30/01	1/30/01	11/30/01	11/30/01	1/30/01	1/30/01	1/30/01
			Time:	9:00	10:00	11:00	12:22	13:11	14:08	15:04
DESCRIPTION	units	range								
Feed Nozzle Cooling Flow	gpm	.5 - 1 (reg FNT)		0.45	0.50	0.5	0.5	0.5	0.5	0.5
Film Cooler Air Supply	scfm	1-10 (reg OGT)		7.0	8.8	6.8	6.8	6.8	7.2	7.2
Melter Vacuum-Magnehelic	in. H <sub>2</sub> O	0.1 - 2.0		0.6	0.6	0.4	0.7	2.3	0.7	1.1
EVS ΔP	in. H <sub>2</sub> O			-0.016	-0.195	-0.0390	-0.0195	-0.195	-0.04	-0.008
Film Cooler ΔP	in. H <sub>2</sub> O			0.9	0.90	0.056	0.1210	0.102	0.148	0.141
HEME ΔP	in. H <sub>2</sub> O			0.715	0.707	0.7	0.703	0.703	0.785	0.785
System ΔP	in. H <sub>2</sub> O			1.18	1.17	1.0+2	1.179	1.14	1.308	1.332
EVS HX Cooling Flow	gpm	1-5 (reg SLT)		3.7	3.7	3.7	3.7	3.7	3.7	3.7
EVS Scrub Tank Volume	gallon	35 - 50		26	26	27	27	27	27	28
EVS Nozzle Pressure	psi	50 - 55		55	55	55	55	55	55	55
EVS Scrub Solution pH	pH	> 9		11	10	10	~10	9	11	11
OG Control Valve position	% open			50	55	55	55	55	55	50
OG Control Valve mode	A or M				A	A	A	A	A	A
Feed Pump (tubing) Condition				OK	OK	OK	OK	OK	OK	OK
Agitator Setting	0-10 %			5	5	5	5	5	5	5
Blower Cooling Flow	gpm	1 - 1.5		1.1	1.15	1.15	1.15	1.15	1.15	1.15
Feed Nozzle Temp (FNT)	°C	< 40		22	22	18	20	21	20	21
Off-Gas Temp (OGT)	°C	< 250			93	57	80	100	92	92
Post EVS Off-Gas Temp	°C	< 50		29	29	22	25	28	29	29
Scrub Liquid Temp (SLT)	°C	< 40		18	18	18	18	18	19	19
Heat Xer Temp	°C	< 30		16	17	15	17	17	17	17
Feed Pump Setting	%			20	20	0	20	20	20	20
Feed Pump Control Mode	A or M			A	A	A	A	A	A	A
Feed Pressure	psi	< 1.0		0.1	0.1	0.6	1.1	0.2	0.2	0.0
Feed Tank Wt	Kg	decreasing		27.0	24.0	—	44.9	43.8	41.5	38.8
Overflow Temp	°C	1000 - 1100		1051	1052	1048	1036	1050	1051	1050
Discharge Can Temp	°C	750 - 850		750	752	751	749	751	749	754
Glass Scale	Kg	< 10		0.24	1.07	1.83	1.84	2.24	2.66	3.51
Alarm Condition	On/Off	off		OFF	OFF	OFF	OFF	OFF	OFF	OFF
time hh:mm					10:08	11:20	12:34	13:18	14:14	15:10

Reviewed and Approved:

RW

Date:

4/23/01  
1/30/01

sheet 3 of 13

## Data Sheet #2: Routine Status Sheet

RSM-01-1

			By:	Jam	DMS	DMS	DMS	DMS	DMS	DMS
			Date:	1/30/01	1/30/01	1/30/01	1/30/01	1/30/01	1/30/01	1/30/01
			Time:	15:59	16:59	17:57	1900	2024	21:06	22:03
DESCRIPTION	units	range								
Feed Nozzle Cooling Flow	gpm	.5 - 1 (reg FNT)	0.5	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	7.2	7.2	7.2	7.2	7.2
Melter Vacuum-Magnehelic	in. H <sub>2</sub> O	0.1 - 2.0	0	2.0	2.0	1.5	1.4	0.2	1.3	1.0
EVS ΔP	in. H <sub>2</sub> O		0.0078	0.016	0.023	0.023	0.023	0.0195	0.043	0.031
Film Cooler ΔP	in. H <sub>2</sub> O		0.1601	0.187	0.172	0.188	0.195	0.183	0.273	0.195
HEME ΔP	in. H <sub>2</sub> O		0.2203	0.79	0.801	0.828	0.821	0.863	0.895	0.863
System ΔP	in. H <sub>2</sub> O		1.290	1.35	1.40	1.4	1.4	1.2	1.59	1.49
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	3.7	3.7	3.7	3.7	3.7	3.7	3.7	3.7
EVS Scrub Tank Volume	gallon	35 - 50	29	29	29	30	30	30	30	30
EVS Nozzle Pressure	psi	50 - 55	55	55	55	55	55	55	55	55
EVS Scrub Solution pH	pH	> 9	10	10	10	10	9	9	9	10
OG Control Valve position	%		50	54	55	56	84	49	0	0
OG Control Valve mode	A or M		A	A	A	A	A	A	M	M
Feed Pump (tubing) Condition			OK	OK	OK	OK	OK	OK	OK	OK
Agitator Setting	0-10%		5	5	5	5	5	5	5	5
Blower Cooling Flow	gpm	1 - 1.5	1.15	1.15	1.15	1.2	1.2	1.2	1.2	1.2
Feed Nozzle Temp (FNT)	°C	< 40	20	20	20	19	20	18	20	22
Off-Gas Temp (OGT)	°C	< 250	84	85	88	80	84	69	138	109
Post EVS Off-Gas Temp	°C	< 50	29	29	30	29	29	25	29	31
Scrub Liquid Temp (SLT)	°C	< 40	19	19	18	19	18	18	18	18
Heat Xer Temp	°C	< 30	16	17	17	15	17	15	12	17
Feed Pump Setting	%		20	20	21	21	20	0	20	23
Feed Pump Control Mode	A or M		A	A	A	A	A	A	A	A
Feed Pressure	psi	< 1.0	0.0	0.0	0.0	0.0	0	0.5	0.1	0.1
Feed Tank Wt	Kg	decreasing	35.2	32.4	28.6	25.4	21.4	20.4	19.3	16.3
Overflow Temp	°C	1000 - 1100	1051	1051	1050	1050	1050	1041	1004	1050
Discharge Can Temp	°C	750 - 850	749	749	738	750	750	764	749	754
Glass Scale	Kg	< 10	4.33	4.83	5.46	5.82	6.40	8.94	8.96	0.02
Alarm Condition	On/Off	off	off	off	off	off	off	off	off	off
time hh:mm			16:11	17:12	18:03	1710	2030	21:17	2210	2302

Reviewed and Approved:

*[Signature]*

Date:

4/23/01  
1/30/01

sheet 4 of 13

## Data Sheet #2: Routine Status Sheet

RSM-01-1

			By:	AKS/MD	AKS	AKS	AKS	AKS	AKS	AKS	AKS
			Date:	1/31/01	1/31/01	1/31/01	1/31/01	1/31/01	1/31/01	1/31/01	1/31/01
			Time:	0013	01:10	02:07	03:12	04:00	06:30	07:10	07:10
DESCRIPTION	units	range									
Feed Nozzle Cooling Flow	gpm	.5 - 1 (reg FNT)	0.5	0.5	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	7.2	7.2	7.2	5.6	5.7	5.7
Melter Vacuum-Magnehelic	in. H <sub>2</sub> O	0.1 - 2.0	2.0	1.4	1.4	1.4	1.4	1.6	0.8	0.9	0.9
EVS ΔP	in. H <sub>2</sub> O		0.039	0.019	0.031	0.027	0	0	2.2	3.03	3.03
Film Cooler ΔP	in. H <sub>2</sub> O		0.066	0.21	0.25	0.26	0.25	0	-0.79	-0.86	-0.86
HEME ΔP	in. H <sub>2</sub> O		0.765	0.836	0.875	0.875	0.863	0	0.65	0.69	0.69
System ΔP	in. H <sub>2</sub> O		1.23	1.48	1.56	1.56	1.52	0	2.6	3.26	3.26
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	3.7	3.7	3.7	3.6	3.65	0	3.6	3.6	3.6
EVS Scrub Tank Volume	gallon	35 - 50	26	26	26	31	31	0	34	35	35
EVS Nozzle Pressure	psi	50 - 55	55	55	55	55	55	0	55	55	55
EVS Scrub Solution pH	pH	> 9	10	10	10	9	10	0	8.5	9	9
OG Control Valve position	% closed		0	0	0	0	0	0	0	0	0
OG Control Valve mode	A or M		M	M	M	M	M	0	M	M	M
Feed Pump (tubing) Condition			OK	OK	OK	OK	SPRt	0	fixed	fixed	fixed
Agitator Setting	0-10		2.5	5	5	5	5	0	5	5	5
Blower Cooling Flow	gpm	1 - 1.5	1.2	1.2	1.2	1.2	1.2	0	1.2	1.2	1.2
Feed Nozzle Temp (FNT)	°C	< 40	19	20	20	19	18	0	22	20	20
Off-Gas Temp (OGT)	°C	< 250	99	114	98	86	46	0	74	81	81
Post EVS Off-Gas Temp	°C	< 50	20	31	32	33	22	0	29	30	30
Scrub Liquid Temp (SLT)	°C	< 40	18	18	18	20	19	0	19	19	19
Heat Xer Temp	°C	< 30	14	17	17	13	14	0	18	17	17
Feed Pump Setting	%		0	25	25	25	0	0	20	20	20
Feed Pump Control Mode	A or M		A	A	A	A	OFF	0	A	A	A
Feed Pressure	psi	< 1.0	0.7	0.2	0.0	2.2	2.8	0	0.1	0.2	0.2
Feed Tank Wt	Kg	decreasing	62.4	61.5	56.9	56.4	50.0	0	41.3	39.7	39.7
Overflow Temp	°C	1000 - 1100	1050	1051	1051	1049	1056	0	1050	1051	1051
Discharge Can Temp	°C	750 - 850	749	750	742	754	787	0	749	749	749
Glass Scale	Kg	< 10	0	0	0.43	0.88	2.23	0	4.03	3.79	3.79
Alarm Condition	On/Off	off	OFF	OFF	OFF	OFF	OFF	0	OFF	OFF	OFF
time hh:mm			00:21	01:20	02:17	3:22	04:09	0	06:41	07:21	07:21

Reviewed and Approved:

*R. W. Galt*

Date: 4/22/01

sheet 5 of 13

## Data Sheet #2: Routine Status Sheet

RSM-01-1

			By:	WCB	54	54	54	54	54	54	54
			Date:	1/31/01	1/31/01	1/31/01	1/31/01	1/31/01	1/31/01	1/31/01	1/31/01
			Time:	0808	0905	1008	10:53	1231	13:00	1404	14:51
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	0.5
Film Cooler Air Supply	scfm	1-10 (reg OGT)	5.9	5.8	5.9	5.8	5.8	5.8	5.8	5.8	5.8
Melter Vacuum-Magnehelic	in. H <sub>2</sub> O	0.1 - 2.0	1.2	1.2	0.8	0.9	0.8	0.7	0.5	0.7	0.7
EVS ΔP	in. H <sub>2</sub> O		2.04	1.0724	1.66	1.81	1.94	1.61	1.48	1.75	1.75
Film Cooler ΔP	in. H <sub>2</sub> O		0.93	1.01	0.64	0.7460	0.91	0.48	0.21	0.547	0.547
HEME ΔP	in. H <sub>2</sub> O		0.79	0.79	0.86	0.93	0.76	0.797	0.84	0.852	0.852
System ΔP	in. H <sub>2</sub> O		1.87	1.95	2.00	1.96	1.93	2.10	2.23	2.27	2.27
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	3.5	3.6	3.5	3.6	3.6	3.7	3.6	3.6	3.6
EVS Scrub Tank Volume	gallon	35 - 50	37	37	37	37	37	37	37	37	37
EVS Nozzle Pressure	psi	50 - 55	55.2	55	55	55	55	55	55	55	55
EVS Scrub Solution pH	pH	> 9	10	10	9	10	10	10	9	9	9
OG Control Valve position	% down		100	100	100	100	100	100	100	100	100
OG Control Valve mode	A or M		M	M	M	M	M	M	M	M	M
Feed Pump (tubing) Condition			OK	OK	OK	OK	OK	OK	OK	OK	OK
Agitator Setting	0-10		5.5	5.5	5.5	5.5	5.5	5.5	5.5	5.5	5.5
Blower Cooling Flow	gpm	1 - 1.5	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2
Feed Nozzle Temp (FNT)	°C	< 40	20	19	20	20	22	20	20	19	19
Off-Gas Temp (OGT)	°C	< 250	102	98	93	91	112	114	105	107	107
Post EVS Off-Gas Temp	°C	< 50	31	30	31	30	28	30	32	32	32
Scrub Liquid Temp (SLT)	°C	< 40	20	20	20	20	18	19	20	20	20
Heat Xer Temp	°C	< 30	18	18	17	17	18	17	18	18	18
Feed Pump Setting	%		20	20	20	20	20	20	20	22	22
Feed Pump Control Mode	A or M		A	A	A	A	A	A	A	A	A
Feed Pressure	psi	< 1.0	0.1	0.1	0.1	0.2	0.1	0.2	0.1	0.1	0.1
Feed Tank Wt	Kg	decreasing	36.5	31.0	27.5	24.5	21.5	20.0	16.5	13.4	13.4
Overflow Temp	°C	1000 - 1100	1050	1051	1050	1050	1050	1051	1050	1051	1051
Discharge Can Temp	°C	750 - 850	750	750	753	749	749	750	750	750	750
Glass Scale	Kg	< 10	8.87	4.50	5.53	6.10	6.66	6.87	7.76	8.10	8.10
Alarm Condition	On/Off	off	off	off	off	off	off	off	off	off	off
time hh:mm			0815	0917	1016	1103	1236	13:09	1409		

Reviewed and Approved: RW [Signature]Date: 4/23/01sheet 6 of 13

C.33

## Data Sheet #2: Routine Status Sheet

RSM-01-1

			By:	JRM	hrs	20	hrs	20	20	20	20	20
			Date:	1/31/01	1/31/01	1/31/01	1/31/01	1/31/01	1/31/01	1/31/01	1/31/01	1/31/01
			Time:	16:00	17:00	17:59	19:00	20:00	21:04	22:00	23:13	
DESCRIPTION	units	range										
Feed Nozzle Cooling Flow	gpm	.5 - 1 (reg FNT)	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Film Cooler Air Supply	scfm	1-10 (reg OGT)	5.7	5.7	5.7	5.7	5.7	5.7	5.7	5.7	5.7	5.7
Melter Vacuum-Magnehelic	in. H <sub>2</sub> O	0.1 - 2.0	0.9	1.2	1.3	1.2	1.3	1.3	1.2	1.2	1.4	1.4
EVS ΔP	in. H <sub>2</sub> O		2.09	0.85	0.32	0.29	0.40	0.43	0.50	0.50	0.36	0.36
Film Cooler ΔP	in. H <sub>2</sub> O		0.977	0.14	-0.32	-0.37	-0.33	-0.35	-0.38	-0.42	-0.42	-0.42
HEME ΔP	in. H <sub>2</sub> O		0.793	0.86	0.92	0.90	0.94	0.91	0.94	0.94	0.97	0.97
System ΔP	in. H <sub>2</sub> O		2.30	1.7	1.7	1.68	1.81	0.81	2.0	1.84	1.84	1.84
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	3.6	3.6	3.6	3.6	3.6	3.6	3.6	3.6	3.6	3.6
EVS Scrub Tank Volume	gallon	35 - 50	37	37	36	36	37	37	37	37	38	38
EVS Nozzle Pressure	psi	50 - 55	55	55.5	55	55	55	55	55	55	55	55
EVS Scrub Solution pH	pH	> 9	9	9	~9 1/2	9.5	12	12	10	10	10	10
OG Control Valve position	% close		100	100	100	100	100	100	100	100	100	100
OG Control Valve mode	A or M		M	M	M	M	M	M	M	M	M	M
Feed Pump (tubing) Condition			OK	OK	OK	OK	OK	OK	OK	OK	OK	OK
Agitator Setting	6-10%		5.2	5.2	5.2	5.2	5.2	5.2	5.2	4	4	4
Blower Cooling Flow	gpm	1 - 1.5	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2
Feed Nozzle Temp (FNT)	°C	< 40	20	19	18	19	18	19	18	18	18	18
Off-Gas Temp (OGT)	°C	< 250	121	110	103	98	97	97	98	98	76	76
Post EVS Off-Gas Temp	°C	< 50	32	32	32	32	32	32	32	32	22	22
Scrub Liquid Temp (SLT)	°C	< 40	19	20	25	20	20	20	20	20	20	20
Heat Xer Temp	°C	< 30	18	18	18	18	18	18	18	18	15	15
Feed Pump Setting	%		22	22	22	22	22	22	22	22	0	0
Feed Pump Control Mode	A or M		A	A	A	A	A	A	A	A	A	A
Feed Pressure	psi	< 1.0	0.1	0.2	0.3	0.3	0.1	0.3	0.1	0.1	0.4	0.4
Feed Tank Wt	Kg	decreasing	35.4	32.9	29.0	25.9	22.5	19.5	15.0	8.8	8.8	8.8
Overflow Temp	°C	1000 - 1100	1049	1050	1049	1050	1048	1051	1051	1050	1050	1050
Discharge Can Temp	°C	750 - 850	681	754	749	748	743	749	751	731	731	731
Glass Scale	Kg	< 10	0.00	—	1.02	—	—	—	—	—	5.11	5.11
Alarm Condition	On/Off	off	off	off	off	off	off	off	off	off	off	off
time	hh:mm		16:11	15:09	18:15	19:06	20:06	21:10	22:08	23:23		

Reviewed and Approved: *RW*

Date: 4/23/01

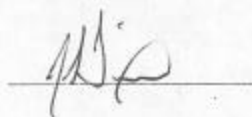
sheet 7 of 13

## Data Sheet #2: Routine Status Sheet

RSM-01-1

			By:	SPB	1/1	SPB	SPB	SPB	SPB	SPB	SPB
			Date:	2/1/01	2/1/01	2/1/01	2/1/01	2/1/01	2/1/01	2/1/01	2/1/01
			Time:	0:00	0:00	2:02	3:27	4:11	05:00	06:04	7:03
DESCRIPTION	units	range									
Feed Nozzle Cooling Flow	gpm	.5 - 1 (reg FNT)	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Film Cooler Air Supply	scfm	1-10 (reg OGT)	5.8	5.8	5.7	5.7	5.8	5.8	5.8	5.8	6.4
Melter Vacuum-Magnehelic	in. H <sub>2</sub> O	0.1 - 2.0	1.4	1.4	0.9	1.2	2.5	1.2	1.5	1.2	
EVS ΔP	in. H <sub>2</sub> O		0.43	0.42	0.68	0.69	0.82	0.92	1.01	0.695	
Film Cooler ΔP	in. H <sub>2</sub> O		-0.32	-0.31	-0.40	-0.44	-0.48	-0.48	-0.57	-0.86	
HEME ΔP	in. H <sub>2</sub> O		0.94	0.92	0.98	0.99	0.99	1.161	1.166	1.05	
System ΔP	in. H <sub>2</sub> O		1.83	1.76	2.18	2.27	2.24	2.92	3.70	2.63	
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	3.7	3.7	3.7	3.7	3.7	3.7	3.7	3.6	
EVS Scrub Tank Volume	gallon	35 - 50	38	38	39	39	40	40	40	40	
EVS Nozzle Pressure	psi	50 - 55	55	55	55	55	55	55	55	55	
EVS Scrub Solution pH	pH	> 9	9	9	9	9	9	12	12	10	
OG Control Valve position	% <sub>cbv</sub>		0	0	0	0	0	0	0	0	
OG Control Valve mode	A or M		M	M	M	M	M	M	M	M	
Feed Pump (tubing) Condition			OK		OK	OK	OK	OK	OK	OK	
Agitator Setting	0-10%		6		6	6	6	6	6	6	
Blower Cooling Flow	gpm	1 - 1.5	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	
Feed Nozzle Temp (FNT)	°C	< 40	19	18	18	18	18	17	18	20	
Off-Gas Temp (OGT)	°C	< 250	29	76	120	100	102	81	101	116	
Post EVS Off-Gas Temp	°C	< 50	21	20	32	33	32	24	34	32	
Scrub Liquid Temp (SLT)	°C	< 40	18	17	20	20	21	21	20	20	
Heat Xer Temp	°C	< 30	15	15	18	18	18	16	19	18	
Feed Pump Setting	%		0	0	25	25	25	25	25	25	
Feed Pump Control Mode	A or M		A	A	A	A	A	A	A	A	
Feed Pressure	psi	< 1.0	0.4	0.5	0.2	0.3	0.0	0.0	0.0	0.0	
Feed Tank Wt	Kg	decreasing	42.2	42.4	38.3	32.7	29.9	26.3	22.5	20.9	
Overflow Temp	°C	1000 - 1100	1050	1050	1051	1051	1051	1055	1058	1050	
Discharge Can Temp	°C	750 - 850	752	750	751	750	746	750	750	750	
Glass Scale	Kg	< 10	5.05	5.11	5.16	6.04	6.70	7.52	8.15	8.47	
Alarm Condition	On/Off	off	OFF	OFF	OFF	OFF	OFF	OFF	OFF	OFF	
time hh:mm			0:07	0:09	2:10	3:34	04:18	05:06	06:10	07:14	

Reviewed and Approved:



Date: 2/1/01

sheet 8 of 13

## Data Sheet #2: Routine Status Sheet

new  
feed  
batch  
just  
started

RSM-01-1

		By: <i>BRUC</i>	<i>BRUC</i>	<i>JAM</i>	<i>JAM</i>	<i>HJ</i>	<i>JAM</i>	<i>JAM</i>	<i>BRUC</i>	<i>JAM</i>
		Date:	<i>2-1-01</i>	<i>2/1/01</i>	<i>2/1/01</i>	<i>2/1/01</i>	<i>2/1/01</i>	<i>2/1/01</i>	<i>2-1-01</i>	<i>2/1/01</i>
		Time:	<i>08:25</i>	<i>09:04</i>	<i>10:22</i>	<i>11:01</i>	<i>12:12</i>	<i>13:12</i>	<i>13:56</i>	<i>15:09</i>
DESCRIPTION	units	range								
Feed Nozzle Cooling Flow	gpm	.5 - 1 (reg FNT)	<i>0.5</i>	<i>0.5</i>	<i>0.5</i>	<i>0.5</i>	<i>0.5</i>	<i>0.5</i>	<i>0.5</i>	<i>0.5</i>
Film Cooler Air Supply	scfm	1-10 (reg OGT)	<i>6.5</i>	<i>6.5</i>	<i>6.5</i>	<i>6.6</i>	<i>6.4</i>	<i>6.3</i>	<i>6.2</i>	<i>6.2</i>
Melter Vacuum-Magnehelic	in. H <sub>2</sub> O	0.1 - 2.0	<i>1.1</i>	<i>1.1</i>	<i>1.4</i>	<i>1.9-2.0</i>	<i>1.0</i>	<i>.7</i>	<i>0.5</i>	<i>1.2</i>
EVS ΔP	in. H <sub>2</sub> O		<i>0.68</i>	<i>0.66</i>	<i>0.62</i>	<i>0.42</i>	<i>0.53</i>	<i>.57</i>	<i>0.64</i>	<i>0.69</i>
Film Cooler ΔP	in. H <sub>2</sub> O		<i>0.51</i>	<i>0.543</i>	<i>0.56</i>	<i>0.30</i>	<i>0.61</i>	<i>0.52</i>	<i>0.61</i>	<i>0.801</i>
HEME ΔP	in. H <sub>2</sub> O		<i>1.06</i>	<i>1.11</i>	<i>1.09</i>	<i>1.07</i>	<i>0.945</i>	<i>1.04</i>	<i>1.03</i>	<i>1.07</i>
System ΔP	in. H <sub>2</sub> O		<i>2.7</i>	<i>2.64</i>	<i>2.63</i>	<i>2.14</i>	<i>2.30</i>	<i>2.54</i>	<i>2.63</i>	<i>2.93</i>
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	<i>2.7</i>	<i>3.6</i>	<i>3.6</i>	<i>3.6</i>	<i>3.7</i>	<i>3.6</i>	<i>3.7</i>	<i>3.6</i>
EVS Scrub Tank Volume	gallon	35 - 50	<i>40</i>	<i>41</i>	<i>42</i>	<i>41 1/2</i>	<i>41 1/2</i>	<i>N/A 40</i>	<i>42</i>	<i>42</i>
EVS Nozzle Pressure	psi	50 - 55	<i>55</i>	<i>55</i>	<i>55</i>	<i>55</i>	<i>55</i>	<i>55</i>	<i>55</i>	<i>55</i>
EVS Scrub Solution pH	pH	> 9	<i>10.5</i>	<i>10</i>	<i>10</i>	<i>10</i>	<i>10</i>	<i>9.5</i>	<i>10.5</i>	<i>10</i>
OG Control Valve position	% <i>close</i>		<i>0%</i>	<i>100</i>	<i>100</i>	<i>0% close</i>	<i>100</i>	<i>100</i>	<i>100</i>	<i>100</i>
OG Control Valve mode	A or M		<i>M</i>	<i>M</i>	<i>M</i>	<i>M</i>	<i>M</i>	<i>M</i>	<i>M</i>	<i>M</i>
Feed Pump (tubing) Condition			<i>Good</i>	<i>OK</i>	<i>OK</i>	<i>changing</i>	<i>OK</i>	<i>OK</i>	<i>OK</i>	<i>OK</i>
Agitator Setting	<i>0-10%</i>		<i>4</i>	<i>4</i>	<i>4</i>	<i>4</i>	<i>4.5</i>	<i>4.5</i>	<i>4.5</i>	<i>4.5</i>
Blower Cooling Flow	gpm	1 - 1.5	<i>1.2</i>	<i>1.2</i>	<i>1.2</i>	<i>1.2</i>	<i>1.2</i>	<i>1.2</i>	<i>1.2</i>	<i>1.2</i>
Feed Nozzle Temp (FNT)	°C	< 40	<i>20</i>	<i>20</i>	<i>18</i>	<i>20</i>	<i>20</i>	<i>19</i>	<i>18</i>	<i>18</i>
Off-Gas Temp (OGT)	°C	< 250	<i>100</i>	<i>98</i>	<i>99</i>	<i>55</i>	<i>91</i>	<i>95</i>	<i>91</i>	<i>90</i>
Post EVS Off-Gas Temp	°C	< 50	<i>32</i>	<i>33</i>	<i>32</i>	<i>21</i>	<i>29</i>	<i>34</i>	<i>34</i>	<i>33</i>
Scrub Liquid Temp (SLT)	°C	< 40	<i>19</i>	<i>20</i>	<i>21</i>	<i>20</i>	<i>18</i>	<i>20</i>	<i>21</i>	<i>22</i>
Heat Xer Temp	°C	< 30	<i>18</i>	<i>18</i>	<i>19</i>	<i>16</i>	<i>18</i>	<i>19</i>	<i>20</i>	<i>20</i>
Feed Pump Setting	%		<i>25%</i>	<i>25</i>	<i>20</i>	<i>0</i>	<i>20</i>	<i>25</i>	<i>25</i>	<i>23</i>
Feed Pump Control Mode	A or M		<i>A</i>	<i>A</i>	<i>A</i>	<i>M</i>	<i>A</i>	<i>A</i>	<i>A</i>	<i>A</i>
Feed Pressure	psi	< 1.0	<i>0.2</i>	<i>0.1</i>	<i>0.2</i>	<i>0.7</i>	<i>0.4</i>	<i>0.2</i>	<i>0.4</i>	<i>0</i>
Feed Tank Wt	Kg	decreasing	<i>17.2</i>	<i>15.2</i>	<i>10.9</i>	<i>adding feed</i>	<i>45.4</i>	<i>41.3</i>	<i>39.0</i>	<i>33.3</i>
Overflow Temp	°C	1000 - 1100	<i>1050</i>	<i>1050</i>	<i>1050</i>	<i>1050</i>	<i>1051</i>	<i>1050</i>	<i>1050</i>	<i>1050</i>
Discharge Can Temp	°C	750 - 850	<i>754</i>	<i>754</i>	<i>747</i>	<i>754</i>	<i>751</i>	<i>749</i>	<i>749</i>	<i>749</i>
Glass Scale	Kg	< 10	<i>0.07</i>	<i>0.28</i>	<i>0.70</i>	<i>0.23</i>	<i>1.49</i>	<i>2.00</i>	<i>2.80</i>	<i>3.09</i>
Alarm Condition	On/Off	off	<i>OFF</i>	<i>OFF</i>	<i>OFF</i>	<i>OFF</i>	<i>OFF</i>	<i>OFF</i>	<i>OFF</i>	<i>OFF</i>
time hh:mm			<i>08:52</i>		<i>10:28</i>	<i>11:09</i>	<i>12:21</i>	<i>13:22</i>	<i>14:07</i>	<i>15:12</i>

Reviewed and Approved:

*RW J. J.*Date: *4/23/01*

sheet

*9* of *18*



## Data Sheet #2: Routine Status Sheet

RSM-01-1

			By:	<i>AK</i>	<i>AK</i>	<i>AK</i>	<i>AK</i>	<i>AK</i>	<i>AK</i>	<i>AK</i>
			Date:	2/1/01	2/1/01	2/1/01	2/1/01	2/1/01	2/1/01	2/1/01
			Time:	1606	1710	1805	1856	1955	2101	2200
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)	6.25	6.4	6.3	6.2	6.2	6.2	6.2	6.0
Melter Vacuum-Magnehelic	in. H <sub>2</sub> O	0.1 - 2.0	1.2	1.2	1.9	1.8	1.9	1.5	1.9	1.5
EVS ΔP	in. H <sub>2</sub> O		0.7	0.6	.8	0.6	.55	.65	0.6	0.6
Film Cooler ΔP	in. H <sub>2</sub> O		0.9	0.7	1.1	1.3	1.1	2.0	2.1	0.2
HEME ΔP	in. H <sub>2</sub> O		1.1	1.0	.98	.99	1.0	1.0	1.0	1.1
System ΔP	in. H <sub>2</sub> O		3.2	2.7	3.3	3.3	3.3	4.0	4.2	2.3
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	3.7	3.6	3.6	3.6	3.6	3.6	3.6	3.6
EVS Scrub Tank Volume	gallon	35 - 50	43	43	43	43	44	44	44	44
EVS Nozzle Pressure	psi	50 - 55	55	55	55	55	55	55	55	55
EVS Scrub Solution pH	pH	> 9	11	11	11	10	10	10	10	10
OG Control Valve position	open% <del>down</del>		Manual	100%	100	100 open	100 open	100	100	100
OG Control Valve mode	A or M		M	Manual	M	M	M	M	M	M
Feed Pump (tubing) Condition			OK	OK	OK	OK	OK	OK	OK	OK
Agitator Setting	0-10 %		4.5	4.5	4.5	4.5	4.5	4.5	4.5	3
Blower Cooling Flow	gpm	1 - 1.5	1.2	1.2	1.3	1.2	1.2	1.2	1.2	1.2
Feed Nozzle Temp (FNT)	°C	< 40	18	18	20	21	20	19	18	18
Off-Gas Temp (OGT)	°C	< 250	91	86	88	97	86	95	91	121
Post EVS Off-Gas Temp	°C	< 50	34	23	30	32	31	33	32	33
Scrub Liquid Temp (SLT)	°C	< 40	22	21	20	20	20	21	21	21
Heat Xer Temp	°C	< 30	20	17	18	18	18	18	18	18
Feed Pump Setting	%		23	0.0	20.0	20	20	21	21	21
Feed Pump Control Mode	A or M		A	A	A	A	A	A	A	A
Feed Pressure	psi	< 1.0	0	0.6	0	0	0.1	0.2	0.1	0.1
Feed Tank Wt	Kg	decreasing	29.5	27.5	27.0	24.5	22.0	17.7	14.5	10.9
Overflow Temp	°C	1000 - 1100	1050	1050	1051	1051	1050	1051	1050	1050
Discharge Can Temp	°C	750 - 850	750	747	749	749	743	750	747	749
Glass Scale	Kg	< 10	3.96	4.65	4.61	4.73	5.26	6.07	6.81	7.40
Alarm Condition	On/Off	off	off	off	off	off	off	off	off	off
time	hh:mm			1744	1811	1903	2003	2107	2204	2304

Reviewed and Approved: *R. J. J.*Date: *4/23/01*sheet *10* of *13*

## Data Sheet #2: Routine Status Sheet

RSM-01-1

			By:	ASD	HS	ASD	HS	ASD	HS	ASD	HS
			Date:	2/2/01	2/2/1	2/2/01	2/2/1	2/2/01	2/2/1	2/2/01	2/2/1
			Time:	00:29	01:02	01:58	03:03	4:02	05:06	06:02	07:17
DESCRIPTION	units	range									
Feed Nozzle Cooling Flow	gpm	.5 - 1 (reg FNT)	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Film Cooler Air Supply	scfm	1-10 (reg OGT)	6.2	6.2	6.2	6.0	6.0	6.0	6.0	6.0	5.9
Melter Vacuum-Magnehelic	in. H <sub>2</sub> O	0.1 - 2.0	1.4	1.0	1.3	1.1	1.3	1.2	1.3	1.3	1.0
EVS ΔP	in. H <sub>2</sub> O		0.339	0.035	0.383	0.62	0.609	0.62	0.65	0.65	0.75
Film Cooler ΔP	in. H <sub>2</sub> O		0.039	0.35	0.047	0.161	0.156	0.21	0.219	0.25	0.25
HEME ΔP	in. H <sub>2</sub> O		0.949	0.95	0.992	1.05	1.06	1.03	1.04	1.05	1.05
System ΔP	in. H <sub>2</sub> O		1.710	1.7	1.86	2.25	2.26	2.222	2.80	2.46	2.46
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	3.6	3.6	3.6	3.5	3.4	3.6	3.6	3.6	3.6
EVS Scrub Tank Volume	gallon	35 - 50	45	45	45	45	45	46	46	47	47
EVS Nozzle Pressure	psi	50 - 55	55	55	55	55	55	55	55	55	55
EVS Scrub Solution pH	pH	> 9	9	9.5	9	9	9	9	9	9	9
OG Control Valve position	% closed		0	0	0	0	0	0	0	0	0
OG Control Valve mode	A or M		M	M	M	M	M	M	M	M	M
Feed Pump (tubing) Condition			OK	OK	OK	—	OK	OK	Adjusted	OK	OK
Agitator Setting	0-10		5	6	6	6	6	6	6	6	6
Blower Cooling Flow	gpm	1 - 1.5	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2
Feed Nozzle Temp (FNT)	°C	< 40	20	20	20	19	18	18	18	18	18
Off-Gas Temp (OGT)	°C	< 250	63	63	77	124	113	108	104	108	108
Post EVS Off-Gas Temp	°C	< 50	21	20	22	35	34	34	34	34	34
Scrub Liquid Temp (SLT)	°C	< 40	19	18	18	22	21	22	22	22	22
Heat Xer Temp	°C	< 30	15	15	16	19	20	20	20	20	20
Feed Pump Setting	%		0.0	0.0	0.0	25	25	25	25	25	25
Feed Pump Control Mode	A or M		A	A	A	A	A	A	A	A	A
Feed Pressure	psi	< 1.0	0.7	0.7	0.1	0.0	0.1	0.0	0.2	0.1	0.1
Feed Tank Wt	Kg	decreasing		43.3	42.2	38.3	33.6	29.3	25.0	20.0	20.0
Overflow Temp	°C	1000 - 1100	1050	1050	1051	1051	1048	1050	1051	1051	1051
Discharge Can Temp	°C	750 - 850	750	750	750	752	751	749	750	751	751
Glass Scale	Kg	< 10	7.73	7.75	7.79	8.31	8.83	9.13	0.97	2.06	2.06
Alarm Condition	On/Off	off	OFF	OFF	OFF	OFF	OFF	OFF	OFF	OFF	OFF
time hh:mm			0:36	01:17	02:04	03:11	4:07	05:12	06:14	07:25	

Reviewed and Approved: RW [Signature]Date: 4/23/01sheet 11 of 13

## Data Sheet #2: Routine Status Sheet

RSM-01-1

			By:	2/2/01	1/1	1/1	JAM	1/1	JAM	JAM
			Date:	2/2/01	2/2/01	2/2/01	2/2/01	2/2/01	2/2/01	2/2/01
			Time:	08:14	09:10	10:06	11:13	12:06	13:00	14:05
DESCRIPTION	units	range								
Feed Nozzle Cooling Flow	gpm	.5 - 1 (reg FNT)	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Film Cooler Air Supply	scfm	1-10 (reg OGT)	5.9	6.0	6.0	6.0	5.8	5.8	5.8	5.8
Melter Vacuum-Magnehelic	in. H <sub>2</sub> O	0.1 - 2.0	1.0	1.5	1.6	1.6	1.1	1.5	1.0	1.1
EVS ΔP	in. H <sub>2</sub> O		0.832	0.578	0.60	0.44.59	0.97	0.93	0.93	1.16
Film Cooler ΔP	in. H <sub>2</sub> O		0.238	0.175	0.20	0.63.91	0.31	0.30	0.31	0.402
HEME ΔP	in. H <sub>2</sub> O		1.05	1.0	0.98	0.765	1.03	1.04	1.05	1.02
System ΔP	in. H <sub>2</sub> O		2.53	2.12	2.17	2.15	2.67	2.62	2.67	2.97
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	3.6	3.6	3.6	3.6	3.6	3.5	3.5	3.5
EVS Scrub Tank Volume	gallon	35 - 50	45	47	47	47	47.5	48	48	49
EVS Nozzle Pressure	psi	50 - 55	55	55	55	55	55	55	55	55
EVS Scrub Solution pH	pH	> 9	9	9	9	9	9	8.5	9	9
OG Control Valve position	% closed		0	0	0	0	0	0	0	0
OG Control Valve mode	A or M		M	M	M	M	M	M	M	M
Feed Pump (tubing) Condition			OK	OK	OK	OK	OK	OK	OK	OK
Agitator Setting	0-10		6	6	6	6	5	5	5	5
Blower Cooling Flow	gpm	1 - 1.5	1.2	1.2	1.2	1.2	1.1	1.2	1.2	1.2
Feed Nozzle Temp (FNT)	°C	< 40	18	20	20	20	20	18	18	18
Off-Gas Temp (OGT)	°C	< 250	109	76	81	81	135	119	114	112
Post EVS Off-Gas Temp	°C	< 50	36	23	22	22	36	36	36	37
Scrub Liquid Temp (SLT)	°C	< 40	22	21	20	18	20	22	22	23
Heat Xer Temp	°C	< 30	20	17	16	16	20	20	20	21
Feed Pump Setting	%		25	0	0	0	30	25	22.5	27.5
Feed Pump Control Mode	A or M		A	A	A	A	A	A	A	A
Feed Pressure	psi	< 1.0	0.1	0.7	0.6	0.6	0.4	0.4	0.1	0.3
Feed Tank Wt	Kg	decreasing	15.9	—	47.9	55.1	45.8	41.0	37.0	27.9
Overflow Temp	°C	1000 - 1100	1051	1050	1051	1050	1050	1050	1051	1050
Discharge Can Temp	°C	750 - 850	756	747	749	750	751	749	749	750
Glass Scale	Kg	< 10	2.81	3.45	3.56	4.20.35	3.49	4.29	5.06	6.29
Alarm Condition	On/Off	off	OFF	OFF	OFF	OFF	OFF	OFF	OFF	OFF
time hh:mm			08:20	9:15	10:15	11:20	12:12	13:15	14:11	15:05

Reviewed and Approved: R. W. GelsDate: 4/23/01sheet 12 of 13

## Data Sheet #2: Routine Status Sheet

RSM-01-1

			By:	<i>AK</i>	<i>AK</i>	<i>AK</i>	<i>AK</i>				
			Date:	2/2/01	2/2/01	2/2/01	2/2/01				
			Time:	1903	1605	1901	2003				
DESCRIPTION	units	range									
Feed Nozzle Cooling Flow	gpm	.5 - 1 (reg FNT)	0.47	.5	0.5	0.5					
Film Cooler Air Supply	scfm	1-10 (reg OGT)	5.9	5.8	5.8	5.8					
Melter Vacuum-Magnehelic	in. H <sub>2</sub> O	0.1 - 2.0	1.5	1.75	1.75	1.6					
EVS ΔP	in. H <sub>2</sub> O		1.07	1.2	1.5	1.3					
Film Cooler ΔP	in. H <sub>2</sub> O		0.4	1.5	0.5	0.6					
HEME ΔP	in. H <sub>2</sub> O		1.04	2.9	1.0	1.0					
System ΔP	in. H <sub>2</sub> O		2.95	2.9	2.9	3.3					
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	3.5	3.5	3.6	3.6					
EVS Scrub Tank Volume	gallon	35 - 50	50	50	51	51					
EVS Nozzle Pressure	psi	50 - 55	55	55	55	55					
EVS Scrub Solution pH	pH	> 9	10	9	9	9					
OG Control Valve position	% closed		0	0	0	0					
OG Control Valve mode	A or M		M	M	M	M					
Feed Pump (tubing) Condition			OK	OK	OK	OK					
Agitator Setting	0-10		5	5	5	3					
Blower Cooling Flow	gpm	1 - 1.5	1.2	1.2	1.2	1.2					
Feed Nozzle Temp (FNT)	°C	< 40	17	18	18	18					
Off-Gas Temp (OGT)	°C	< 250	472	115	106	96					
Post EVS Off-Gas Temp	°C	< 50	37	35	36	35					
Scrub Liquid Temp (SLT)	°C	< 40	23	22	22	22					
Heat Xer Temp	°C	< 30	21	20	20	20					
Feed Pump Setting	%		23.5	25.0	25	25					
Feed Pump Control Mode	A or M		A	A	A	A					
Feed Pressure	psi	< 1.0	0.3	0.0	0.1	0.1					
Feed Tank Wt	Kg	decreasing	22.5	18.8	14.7	10.7					
Overflow Temp	°C	1000 - 1100	1051	1051	1051	1045					
Discharge Can Temp	°C	750 - 850	750	747	750	745					
Glass Scale	Kg	< 10	7.58	8.61	8.82	0.5					
Alarm Condition	On/Off	off	off	off	off	off					
time	hh:mm		17:11	18:13	19:06	20:07					

Reviewed and Approved: *RW J*

Date: 4/23/01

sheet 13 of 13

## **Off Gas Sampling Data Sheets**

RSM-01-1

## Off-Gas Sampling Data Sheet

By: <u>NS</u>									
Date: <u>1/30/01</u>									
Time: <u>1853</u>									
DESCRIPTION	units								
<b>Gas Chromatograph</b>									
He	ppm	<u>1054</u>	<u>1033</u>	<u>1073</u>	<u>1065</u>	<u>955</u>			
H <sub>2</sub>	ppm	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>			
O <sub>2</sub>	ppm	<u>44,700</u>	<u>45,000</u>	<u>44,800</u>	<u>51,390</u>	<u>44,600</u>			
N <sub>2</sub>	ppm	<u>168,000</u>	<u>169,000</u>	<u>168,000</u>	<u>193,100</u>	<u>168,500</u>			
NO	ppm	<u>-1</u>	<u>-1</u>	<u>-1</u>	<u>0</u>	<u>-1</u>			
CO	ppm	<u>190</u>	<u>207</u>	<u>288</u>	<u>0</u>	<u>721</u>			
CO <sub>2</sub>	ppm	<u>898</u>	<u>958</u>	<u>4,171</u>	<u>56,77</u>	<u>4,11</u>			
N <sub>2</sub> O	ppm	<u>63.7</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>12.4</u>			
NOx	ppm								
<b>NOx Analyzer Computer</b>									
Time		<u>1855</u>	<u>1925</u>	<u>2028</u>	<u>2139</u>	<u>2321</u>			
NOx	ppm	<u>105</u>	<u>96.3</u>	<u>81.6</u>	<u>4057</u>	<u>101</u>			
NO	ppm	<u>105</u>	<u>96.5</u>	<u>81.0</u>	<u>4085</u>	<u>99</u>			
NOx Voltage	Volts	<u>2.15</u>	<u>1.94</u>	<u>1.8839</u>	<u>0.4699</u>	<u>3.328</u>			
<b>NOx Instrument</b>									
Analog Reading	%FS	<u>100</u>	<u>100</u>						
Range selector	value	<u>A470</u>	<u>A470</u>	<u>A470</u>	<u>A470</u>	<u>A470</u>			
Mode selector	value	<u>NO → NOx</u>	<u>NOx</u>	<u>NOx</u>	<u>NOx</u>	<u>NOx</u>			
Sample Pressure	psig	<u>4.0</u>	<u>4.0</u>	<u>4.0</u>	<u>4.0</u>	<u>4.0</u>			
Sample bypass flow	scfm	<u>6.0</u>	<u>6.0</u>	<u>6.0</u>	<u>6.0</u>	<u>6.5</u>			
Ozone Pressure	psig	<u>30</u>	<u>30</u>	<u>30</u>	<u>30</u>	<u>30</u>			
Sierra Mass Flow: Flow#2	scfm	<u>106.4</u>	<u>106.4</u>	<u>106.4</u>	<u>106.4</u>	<u>102-112</u>			
<b>MKS Mass Flow</b>									
Ar Carrier, Chan-1	lpm	<u>1.98</u>	<u>1.98</u>	<u>1.98</u>	<u>1.98</u>	<u>1.98</u>			
He Tracer, Chan-2	lpm	<u>1.98</u>	<u>1.99</u>	<u>1.99</u>	<u>1.99</u>	<u>1.99</u>			
Total Sample, Chan-3	lpm	<u>2.28</u>	<u>2.28</u>	<u>2.28</u>	<u>2.28</u>	<u>2.28</u>			
N/A, Chan-4	N/A	<u>4.02</u>	<u>4.02</u>	<u>4.02</u>	<u>4.02</u>	<u>4.02</u>			
<b>Sample Pump*</b>									
Vac Gauge	"Hg								
Pres Gauge	psig								
<b>Gas Cylinders Tank Pres*</b>									
O <sub>2</sub>	psig								
Ar GC	psig								
Ar Sweep #1	psig								
Ar Sweep #2	psig								
He GC	psig								
He Tracer #1	psig								
He Tracer #2	psig								
<b>Fid Analyzer (in Hood)</b>									
Conc. (multi-digit display)	value	<u>8</u>	<u>16</u>	<u>6.1</u>	<u>-1.6</u>	<u>-</u>			
Range (single digit display)	value	<u>4</u>	<u>4</u>	<u>4</u>	<u>4</u>	<u>-</u>			
Pump Outlet Pres	psig	<u>4.3</u>	<u>4.3</u>	<u>4.3</u>	<u>4.3</u>	<u>-</u>			
Air Pres (reg next to pump)	psig	<u>8.0</u>	<u>8.0</u>	<u>8.0</u>	<u>8.0</u>	<u>-</u>			
Smple Pres. (Internal reg)	psig	<u>4.0</u>	<u>4.0</u>	<u>4.0</u>	<u>4.0</u>	<u>-</u>			
Fuel Pres. (Internal reg)	psig								
time	hh:mm								

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

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

Reviewed and Approved: RWBDate: 4/23/01sheet 1 of 10

## Off-Gas Sampling Data Sheet

RSM-01-1

By:	WOC	WOC	WOC	WOC	WOC	WOC	WOC
Date:	1-31-01	1/31/01	1/31/01	1/31/01	1/31/01	1/31/01	1/31/01
Time:	0027	0117	0217	0300	03:55	0637	0742
units	(not feeding)	(just started feeding)					
<b>Gas Chromatograph</b>							
He	ppm	1168	999	984	969	957	1385
H <sub>2</sub>	ppm	0	0	0	0	0	0
O <sub>2</sub>	ppm	51,293	42933	42,457	41791	42,572	66505
N <sub>2</sub>	ppm	1,92,108	162,246	160,392	158000	160,661	249,558
NO	ppm	0	-1	-1	-1	-1	0
CO	ppm	0	356	328	368	181	0
CO <sub>2</sub>	ppm	116.7	137.8	145.3	154.4	126.2	378
N <sub>2</sub> O	ppm	0	195	224	239	240	106
NOx	ppm						
<b>NOx Analyzer Computer</b>							
Time		00:33			0301	03:56	0637
NOx	ppm	0.99361	125	123	123	207	168
NO	ppm	0.92414	120	117	118	205	166
NOx Voltage	Volts	0.43716	9.99	2.5	0.5	2.37	3.0
<b>NOx Instrument</b>							
Analog Reading	%FS	0	46	49	48	35	0
Range selector	value	Auto	Auto	Auto	Auto	Auto	Auto
Mode selector	value	NOx	NOx	NOx	NOx	NOx	NOx
Sample Pressure	psig	4	4	4	4	4	4
Sample bypass flow	scfm	5	6	6	6	4	5
Ozone Pressure	psig	30	30	30	30	30	30
Sierra Mass Flow: Flow#2	scfm	100	100.4 steady	100.4	106.4	106	200 Flowing
<b>MKS Mass Flow</b>							
Ar Carrier, Chan-1	lpm	1.98	1.98	1.98	1.98	1.98	1.98
He Tracer, Chan-2	lpm	1.99	1.99	1.99	1.99	1.99	1.99
Total Sample, Chan-3	lpm	2.28	2.28	2.28	2.28	2.28	2.28
N/A, Chan-4	N/A						
<b>Sample Pump*</b>							
Vac Gauge	cm Hg	1.5	1.5	1.5	1.5	1.5	8
Pres Gauge	psig	10	10.2	10.2	10.2	7	4
<b>Gas Cylinders Tank Pres*</b>							
O <sub>2</sub>	psig	650	650	650	640	600	575
Ar GC	psig	175	2500	2500	2500	175	2500
Ar Sweep #1	psig	500	500	500	500	500	500
Ar Sweep #2	psig	1300	1300	1280	1200	1200	1000
He GC	psig	1630	1650	1650	1640	1640	1630
He Tracer #1	psig	2260	2260	2260	2280	2270	2260
He Tracer #2	psig	1530	1530	1500	1500	1450	1300
<b>Fid Analyzer (in Hood)</b>							
Conc. (multi-digit display)	value	-1.7	varies 8-12	varies 9-13	13.24	-1.3	6.710
Range (single digit display)	value	4	4	4	4	4	4
Pump Outlet Pres	psig	4.3	4.3	4.3	4.2	4.2	4.2
Air Pres (reg next to pump)	psig	8	8	8	8	8	8
Smple Pres. (Internal reg)	psig	4	4	4	4	4	4
Fuel Pres. (internal reg)	psig	24	24	24	24	24	24
time hh:mm		00:44	01:27	02:23	0309	04:01	0639

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

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

GC Screen

Reviewed and Approved: *[Signature]*(2200 on GC screen) GC Screen  
Date: 4/23/01

sheet 2 of 10

## Off-Gas Sampling Data Sheet

RSM-01-1

	By:	RWZ	NRS	Jam	RWZ	RWZ	JMR	NRS	NRS
	Date:	1/31/01	Jan 31 2001	1/31/01	1/31/01	1/31/01	1/31/01	1/31/01	1/31/01
	Time:	0935	1300	1406	1500	1600	1708	1800	1926
DESCRIPTION	units	Filter							
Gas Chromatograph		plugged							
He	ppm	348.8	1308	1298	1235.5	1254	1232	1225	1215
H <sub>2</sub>	ppm	0	0	0	0	0	0	0	0
O <sub>2</sub>	ppm	41304	48000	48534	46460	47920	46324	46112	47600
N <sub>2</sub>	ppm	156240	183000	183200	179118	178091	175032	174183	180000
NO	ppm	-1	-1	-1	-1	-1	-1	-1	-1
CO	ppm	39.92	286	368	349	327	299	250	250
CO <sub>2</sub>	ppm	414.6	1320	1318	1294.7	1467	1316	1308	1435
N <sub>2</sub> O	ppm	119.97	148	150	26.6	165	195	195	237
NOx	ppm								
NOx Analyzer Computer									
Time	0	0939	1300	1407	1500	1602	1709	1800	1928
NOx	ppm	25	111	111	119	122	123	110	121
NO	ppm	24	109	109	115	119	121	109	119
NOx Voltage	Volts	1.28	10.0	2.24	2.40	2.47	2.21	2.32	2.48
NOx Instrument									
Analog Reading	%FS	100	110	112	50	49	43	110	119
Range selector	value	NOx	Auto	Auto	Auto	Auto	Auto	Auto	Auto
Mode selector	value	Auto	NOx	NOx	NOx	NOx	NOx	NOx	NOx
Sample Pressure	psig	4	4.0	4.0	4	4	4	4.0	4.0
Sample bypass flow	scm	5	5	4.8	5	5	5	5	5
Ozone Pressure	psig	30	30	30	30	30	30	30	30
Sierra Mass Flow: Flow#2	scm	100	107	107.2	107.2	106.4	106.4	106	106
MKS Mass Flow									
Ar Carrier, Chan-1	lpm	1.98	1.98	1.98	1.98	1.98	1.98	1.98	1.98
He Tracer, Chan-2	lpm	2.99	1.99	1.99	1.99	1.99	1.99	1.99	1.99
Total Sample, Chan-3	lpm	2.28	2.31	2.32	2.32	2.32	2.32	2.32	2.32
N/A, Chan-4	N/A								
Sample Pump*									
Vac Gauge	"Hg	12	1	1	12.1	1	1	4	4
Pres Gauge	psig	5	11	11	12	12	10	10	10
Gas Cylinders Tank Pres*									
O <sub>2</sub>	psig	400	1800	1800	1800	1790	1800	1800	1800
Ar GC	psig	2500	2500	2500	2500	2500	2500	2500	2500
Ar Sweep #1	psig	500	2700	2700	2700	2700	2700	2650	2650
Ar Sweep #2	psig	900	800	790	750	700	700	500	500
He GC	psig	1600	1600	1650	1650	1600	1600	1600	1650
He Tracer #1	psig	2300	2300	2260	2300	2300	2300	2300	2250
He Tracer #2	psig	8000	1050	1020	1000	900	800	700	700
Fid Analyzer (in Hood)									
Conc. (multi-digit display)	ppm	2.1	9	11	1.4	8.8	8.5	1.6	1.8
Range (single digit display)	value	4	4	4	4	4	4	4	4
Pump Outlet Pres	psig	4.2	4.4	4.4	4.2	4.2	4.2	4.2	4.4
Air Pres (reg next to pump)	psig	3	8.0	7.8	7.9	7.9	7.9	8.0	8.0
Smple Pres. (Internal reg)	psig	4	4	4	4	4	4	4	4
Fuel Pres. (internal reg)	psig	2.1	2.4	2.4	2.1	2.1	2.4	2.4	2.4
time	hh:mm	09:44	1319	1419	1501	1610	1715	1810	1932

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

# Multiple tanks (Ar Sweep #s 182 and He Tracer #s 1,2) can be switched by manual valving if need be.

Reviewed and Approved: RWZDate: 4/23/01sheet 3 of 10



RSM-01-1

## Off-Gas Sampling Data Sheet

By:	MKS	MKS	MKS	MKS	WCB
Date:	1-31-01	1/31	1/31/01	1/31/01	2/10/01
Time:	2008	2108	2215	2317	0209
DESCRIPTION	units				
Gas Chromatograph					
He	ppm	1475	1435	1207	1317
H <sub>2</sub>	ppm	0	0	0	0
O <sub>2</sub>	ppm	55502	51576	48579	56901
N <sub>2</sub>	ppm	209218	194705	183700	214013
NO	ppm	-1.0	-1.0	-1.0	0
CO	ppm	344	313	363	0
CO <sub>2</sub>	ppm	1536	1371	1619	96.1
N <sub>2</sub> O	ppm	220	200	224	0
NO <sub>x</sub>	ppm				
NOx Analyzer Computer					
Time		2015	2109	2217	2319
NOx	ppm	115	115.20	116	2.03
NO	ppm	114	110	114	1.95
NOx Voltage	Volts	2.3	2.43	2.3	0.9
NOx Instrument					
Analog Reading	%FS	47	43.1/1102	43	15
Range selector	value	Auto	Auto	Auto	Auto
Mode selector	value	NOx	NOx	NOx	NOx
Sample Pressure	psig	4	4.0	4.0	4.0
Sample bypass flow	scfm	5	5	5	5.5
Ozone Pressure	psig	30	30	30	30
Sierra Mass Flow: Flow#2	scfm	106	106.4	106.4	106.4
MKS Mass Flow					
Ar Carrier, Chan-1	lpm	1.98	1.98	1.98	1.98
He Tracer, Chan-2	lpm	1.99	1.99	1.99	1.99
Total Sample, Chan-3	lpm	2.31	2.31	2.31	2.31
N/A, Chan-4	N/A				
Sample Pump*					
Vac Gauge	"Hg	1	3	3	5
Pres Gauge	psig	10	9.5	9	9.5
Gas Cylinders Tank Pres*					
O <sub>2</sub>	psig	1700	1700	1700	1686
Ar GC	psig	2500	2502	2500	2500
Ar Sweep #1	psig	2700	2700	2700	2700
Ar Sweep #2	psig	400	400	350	350
He GC	psig	1600	1600	1600	1600
He Tracer #1	psig	2300	2300	2300	2260
He Tracer #2	psig	700	650	650	600
Fid Analyzer (in Hood)					
Conc. (multi-digit display)	value	15	13.8	12	-0.9
Range (single digit display)	value	4/0-100	4	4	4
Pump Outlet Pres	psig	4.4	4.4	4.3	4.3
Air Pres (reg next to pump)	psig	8	8	8	8
Sample Pres. (Internal reg)	psig	4	4	4	4
Fuel Pres. (internal reg)	psig	24	24	24	24
time hh:mm		2027	2117	2225	2325

\*Located on floor 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 need be.

GC Clock

Reviewed and Approved: RW EadsDate: 4/23/01sheet 4 of 10

## Off-Gas Sampling Data Sheet

RSM-01-1

By:	490	2005	111	078	050	110	150	RW2
Date:	2/1/01	2/1/01	2/1/01	2/1/01	2/1/01	2/1/01	02/01/01	02/01/01
Time:	1:00	2:01	03:04	04:17	05:07	06:08	7:13	9:09
DESCRIPTION	units							
Gas Chromatograph								
He	ppm	1389	1087	941	806	1421	582	798
H <sub>2</sub>	ppm	0	0	0	0	0	0	0
O <sub>2</sub>	ppm	56893	44984	44631	45857	54488	44745	36,870
N <sub>2</sub>	ppm	213972	2170299	168933	173367	205,191	169,292	139,760
NO	ppm	0	0	0	0	0	0	0
CO	ppm	0	332	237	26997	0	149.9	62.96
CO <sub>2</sub>	ppm	94.4	1448	1155	1123	103	855	715.8
N <sub>2</sub> O	ppm	0	309	0	0	0	0	0
NOx	ppm							247
NOx Analyzer Computer								
Time		0:39	02:03	02:04	04:18	5:08	06:11	7:14
NOx	ppm	1.19	114.38	93.0	82.3	3.12	63	44.86
NO	ppm	1.22	111.06	91.7	80.45	2.98	60	43.06
NOx Voltage	Volts	0.528	2.36	1.88	4.22	1.39	3.2	2.37
NOx Instrument								
Analog Reading	%FS	12%	46	58	24	2	25	20
Range selector	value	Auto	Auto	Auto	Auto	Auto	Auto	Auto
Mode selector	value	NOx	NOx	NOx	NOx	NOx	NOx	NOx
Sample Pressure	psig	4	4	4	4	4	4	4
Sample bypass flow	scfm	4.5	5.5	5.5	5	5	5.5	5
Ozone Pressure	psig	30	30	30	30	30	30	30
Sierra Mass Flow: Flow#2	scfm	106.4	106.4	106.4	106	106	106.4	106.4
MKS Mass Flow								
Ar Carrier, Chan-1	lpm	1.98	1.98	1.98	1.98	1.98	1.98	1.98
He Tracer, Chan-2	lpm	1.99	1.99	1.99	1.99	1.99	1.99	1.99
Total Sample, Chan-3	lpm	2.32	2.31	2.32	2.32	2.32	2.32	2.31
N/A, Chan-4	N/A							
Sample Pump*								
Vac Gauge	"Hg	3	3	6	7.5	2.5	16.2	10.5
Pres Gauge	psig	9.5	9	6.5	4.2	7.5	1.5	2
Gas Cylinders Tank Pres*								
O <sub>2</sub>	psig	1700	1680	1620	1600	1600	1600	1560
Ar GC	psig	2470-2480	2480	2500	2500	2500	2500	2480
Ar Sweep #1	psig	2640	2580	2550	2500	2500	2420	2370
Ar Sweep #2	psig	300	380	320	300	300	320	240
He GC	psig	1640	1630	1630	1630	1630	1630	1630
He Tracer #1	psig	2280	2280	2280	2280	2280	2280	2280
He Tracer #2	psig	510	580	410	400	350	310	170
Fid Analyzer (in Hood)								
Conc. (multi-digit display)	value	7.4	10.713	10-11	10	8	8	2.3
Range (single digit display)	value	4	4	4	4	4	4	4
Pump Outlet Pres	psig	4.2	4.2	4.2	4.2	4.2	4.2	4.2
Air Pres (reg next to pump)	psig	7	7.8	7.9	7.9	7.9	7.9	7.9
Smple Pres, (Internal reg)	psig	4	4	4	4	4	4	4
Fuel Pres, (Internal reg)	psig	24	24	24	24	24	24	24
time	hh:mm	1:04	02:08	03:09	4:23	5:13	06:13	07:18

\* Located on floor 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 need be.

Reviewed and Approved: RW2Date: 4/23/01sheet 5 of 10

C.47

RSM-01-1

Off-Gas Sampling Data Sheet

still no feed  
just stopped feeding  
10:28

RW2

By:								
Date:								
Time:								
DESCRIPTION	units							
Gas Chromatograph								
He	ppm							
H <sub>2</sub>	ppm							
O <sub>2</sub>	ppm							
N <sub>2</sub>	ppm							
NO	ppm							
CO	ppm							
CO <sub>2</sub>	ppm							
N <sub>2</sub> O	ppm							
NOx	ppm							
NOx Analyzer Computer								
Time								
NOx	ppm							
NO	ppm							
NOx Voltage	Volts							
NOx Instrument								
Analog Reading	%FS							
Range selector	value							
Mode selector	value							
Sample Pressure	psig							
Sample bypass flow	scfm							
Ozone Pressure	psig							
Sierra Mass Flow: Flow#2	scfm							
MKS Mass Flow								
Ar Carrier, Chan-1	lpm							
He Tracer, Chan-2	lpm							
Total Sample, Chan-3	lpm							
N/A, Chan-4	N/A							
Sample Pump*								
Vac Gauge	"Hg							
Pres Gauge	psig							
Gas Cylinders Tank Pres*								
O <sub>2</sub>	psig							
Ar GC	psig							
Ar Sweep #1	psig							
Ar Sweep #2	psig							
He GC	psig							
He Tracer #1	psig							
He Tracer #2	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							

\*Located on floor 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 need be.

Reviewed and Approved:

RW2

Date: 4/22/01

sheet 6 of 10

no lead 1759 started reading  
Off-Gas Sampling Data Sheet

RSM-01-1

DESCRIPTION	units	2/1/01	2/1/01	2/1/01	2/1/01	2/1/01	2/1/01	2/1/01	2/1/01
By:									
Date:		2/1/01	2/1/01	2/1/01	2/1/01	2/1/01	2/1/01	2/1/01	2/1/01
Time:		15:20	16:16	17:17	18:01	19:03	20:01	20:17	20:59
Gas Chromatograph									
He	ppm	1072	1211	1291	1403	1097	1161	1100	1105
H <sub>2</sub>	ppm	0	0	0	0	0	0	0	0
O <sub>2</sub>	ppm	46040	51154	58000	50700	47650	48000	48000	47000
N <sub>2</sub>	ppm	174830	193908	220000	192000	141000	183000	183000	178000
NO	ppm	-1	-1	0	0	-1	-1	-1	-1
CO	ppm	252	0	0	0	242	116	516	576
CO <sub>2</sub>	ppm	1200	1196	72	81	1188	955	1370	1400
N <sub>2</sub> O	ppm	204	194	0	0	177	0	150	157
NOx	ppm								
NOx Analyzer Computer									
Time			16:17	17:16	18:02	19:01		20:17	20:59
NOx	ppm	97	94	1.8	2.7	86.0	85	63	66
NO	ppm	95	98	1.8	2.6	83.7	82	62	64
NOx Voltage	Volts	5.13	1.99	0.3	2.7	4.4	4.3	3.25	3.40
NOx Instrument									
Analog Reading	%FS	99	99	18	40	90		27/65ppm	28/70ppm
Range selector	value	Auto	Auto	A	A	A		A	A
Mode selector	value	NOx	NOx	NOx	NOx	NOx		NOx	NOx
Sample Pressure	psig	4	4	4.0	4.0	4.0		4.0	4.0
Sample bypass flow	scfm	4.6	4.4	5.0	4.5	5		5.0	5
Ozone Pressure	psig	30	30	30	30	30		30	30
Sierra Mass Flow: Flow#2	scfm	97.6	96.0	96.8	94.4	94.4		30	30
MKS Mass Flow									
Ar Carrier, Chan-1	lpm	1.98	1.99	1.98	1.98	1.98		1.98	1.98
He Tracer, Chan-2	lpm	1.99	1.99	1.99	1.99	1.99		1.99	1.99
Total Sample, Chan-3	lpm	2.31	2.31	2.32	2.32	2.31		2.32	2.32
N/A, Chan-4	N/A								
Sample Pump*									
Vac Gauge	"Hg	1	1	1	2	2		2	3
Pres Gauge	psig	10	10	10	10	10		10	10
Gas Cylinders Tank Pres*									
O <sub>2</sub>	psig	1420	1400	1400	1400	1400		1350	1350
Ar GC	psig	2450	2450	2450	2450	2450		2450	2450
Ar Sweep #1	psig	2050	1950	1950	1950	1950		1800	1800
Ar Sweep #2	psig	1970	1950	2000	1950	1950		2000	2000
He GC	psig	1620	1600	1600	1600	1600		1650	1600
He Tracer #1	psig	2030	1900-2000	1900	1900	1850		1800	1800
He Tracer #2	psig	2450	2450	2400	2450	2450		2500	2500
Fid Analyzer (in Hood)									
Conc. (multi-digit display)	value	14	17.2	-0.5	3.5	5.1		17	23
Range (single digit display)	value	4	4	4	4	4		4	4
Pump Outlet Pres	psig	4.3	4.3	4.4	4.2	4.2		4.4	4.4
Air Pres (reg next to pump)	psig	7.8	7.9	8	8	8		8	8
Smple Pres. (Internal reg)	psig	4	4	4.0	4	4		4.0	4.0
Fuel Pres. (Internal reg)	psig	24	24	24	24	24		24	24
time hh:mm		15:33	16:21	17:19	18:05	19:07		20:21	21:03

\*Located on floor 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 need be.

Reviewed and Approved: *Ric Rodriguez*

Date: 4/23/01

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C.48

## Off-Gas Sampling Data Sheet

RSM-01-1

By:	2/1/01	2/1/01	2/1/01	2/1/01	2/2/01	2/2/01	2/2/01	2/2/01	2/2/01
Date:	2201	2303	6036	1103	02:06	3:12	0404	05:09	
Time:									
DESCRIPTION	units								
Gas Chromatograph									
He	ppm	1093	1071	1247	1309	1287	984	1193	1052
H <sub>2</sub>	ppm	0	0	0	0	0	0	0	0
O <sub>2</sub>	ppm	47138	48107	55916	56356	57091	43002	30515	42645
N <sub>2</sub>	ppm	179190	182701	210521	214171	215069	159676	116408	162107
NO	ppm	-1	-1	0	0	0	-1	-1	-1
CO	ppm	519	628	0	0	0	357	234	344
CO <sub>2</sub>	ppm	1303	1394	93.1	88.0	89.4	1382	1756	1432
N <sub>2</sub> O	ppm	147	0	0	0	0	204	229	152
NOx	ppm								
NOx Analyzer Computer									
Time		2203	2305	01:37	01:04	02:06	3:12	0405	05:09
NOx	ppm	65	62	1.13	1.18	1.4	92.8	768	1025
NO	ppm	64	60	1.08	1.20	1.37	91.1	7879	1055
NOx Voltage	Volts	3.4	3.2	0.503	0.525	0.622	1.87	3.9	2.17
NOx Instrument									
Analog Reading	%FS	70	26	1	1	0-100	38	42	42
Range selector	value	A	A	A	A	A	A	A	A
Mode selector	value	NOx	NOx	NOx	NOx	NOx	NOx	NOx	NOx
Sample Pressure	psig	4	4	4	4	4	4	4	4
Sample bypass flow	scfm	5	5	5	5	5	5	5.5	5
Ozone Pressure	psig	30	30	30	30	30	30	30	30
Sierra Mass Flow: Flow#2	scfm				87	102.4	102.4	101.6	101.8
MKS Mass Flow									
Ar Carrier, Chan-1	lpm	198	198	1.98	1.98	1.98	1.98	198	1.98
He Tracer, Chan-2	lpm	199	199	1.99	1.99	1.99	1.99	1.99	1.99
Total Sample, Chan-3	lpm	232	232	2.32	2.32	2.31	2.31	2.32	2.32
N/A, Chan-4	N/A								
Sample Pump*									
Vac Gauge	"Hg	2	2	1.5	1.5	1.5	1.5	1.5	
Pres Gauge	psig	10	10	10	10	10.3	10.5	10.2	
Gas Cylinders Tank Pres*									
O <sub>2</sub>	psig	1350	1300	1300	1300	1300	1260	1260	1240
Ar GC	psig	2500	2500	2470	2480	2500	2470	2470	2470
Ar Sweep #1	psig	1750	1700	1660	1650	1600	1520	1510	1470
Ar Sweep #2	psig	2000	2000	2000	2000	2000	2000	2000	2000
He GC	psig	1600	1600	1620	1620	1620	1620	1620	1620
He Tracer #1	psig	1700	1700	1680	1660	1630	1580	1520 mwy	1500
He Tracer #2	psig	2500	2500	2500	2520	2520	2520	2500	2500
Fid Analyzer (in Hood)									
Conc. (multi-digit display)	value	29.3	25	-1.3	-1.4	6	25	19-22	2
Range (single digit display)	value	4	4	4	4	4	4	4	4
Pump Outlet Pres	psig	4.2	4.2	4.2	4.2	4.2	4.2	4.4	4.2
Air Pres (reg next to pump)	psig	8	8	8	8	8	8	7.9	8
Smple Pres, (Internal reg)	psig	4	4	4	4	4	4	4	4
Fuel Pres, (Internal reg)	psig	24	24	24	24	24	24	24	24
time hh:mm		2207	2310	00:41	24	02:10	3:15	0414	05:13

\* Located on floor 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 need be.

Reviewed and Approved: R. W. ZelleDate: 4/23/01sheet 8 of 10

## Off-Gas Sampling Data Sheet

RSM-01-1

By:	MLJ	RWJ		WKS	RUR	DIR	WJR	NRS
Date:	2/2/01	2/2/01	9:00, 10:00	2/2/01	2/2/01	2/2/01	2/2/01	2/2/01
Time:	0726	0811		1200	1259	2:00pm	1500	1530
DESCRIPTION	units							
Gas Chromatograph								
He	ppm	987	1043	1024	1031	818	0	1107
H <sub>2</sub>	ppm	0	0	0	0	0	0	0
O <sub>2</sub>	ppm	44485	43097	43000	41618	42712	43000	43000
N <sub>2</sub>	ppm	1631866	163794	161000	158460	169874	163000	167500
NO	ppm	-1	-1	-1	-1	-1	-1	-1
CO	ppm	390	483	646	431	401	544	681
CO <sub>2</sub>	ppm	1318	1515	1626	1548	1312	1690	1866
N <sub>2</sub> O	ppm	0	180	144	100	202	246	0
NOx	ppm							
NOx Analyzer Computer								
Time		7:21	08:13	1200	100	2:01	-	1530
NOx	ppm	101.6	103.29	113	111.99	87.4	113	109
NO	ppm	99.3	102.38	11	110.85	85.7	112	107
NOx Voltage	Volts	2.05	2.0987	2.39	2.30	4.36		2.2
NOx Instrument								
Analog Reading	%FS	40	42	47/118pm	46	35		44/110
Range selector	value	A	A	A	A	A		A
Mode selector	value	NOx	NOx	NOx	NOx	NOx		NOx
Sample Pressure	psig	4	4	40	4	4		40
Sample bypass flow	scm	5	5	5	5	5		5
Ozone Pressure	psig	30	30	30	30	30		30
Sierra Mass Flow: Flow#2	scm	100	100	30	106.4	106.4		106
MKS Mass Flow								
Ar Carrier, Chan-1	lpm	1.88	1.98	1.98	1.98	1.97		1.99
He Tracer, Chan-2	lpm	1.99	1.99	1.99	1.99	1.99		1.99
Total Sample, Chan-3	lpm	2.32	2.32	2.32	2.32	2.32		2.32
N/A, Chan-4	N/A							
Sample Pump*								
Vac Gauge	"Hg	3	3	3	3	4.5	12	10
Pres Gauge	psig	8.5	8.5	10	10	12	2	2
Gas Cylinders Tank Pres*								
O <sub>2</sub>	psig	1200	1200	1100	1100	1100		1100
Ar GC	psig	2490	2500	2500	2500	2500		2500
Ar Sweep #1	psig	1320	1300	1150	1100	1050		1000
Ar Sweep #2	psig	2000	2000	2000	2000	2000		2000
He GC	psig	1630	1630	1600	1600	1600		1600
He Tracer #1	psig	1400	1390	1200	1200	1150		1100
He Tracer #2	psig	2510	2550	2500	2500	2500		2500
Fid Analyzer (in Hood)								
Conc. (multi-digit display)	value	21	30	26	35	24.6	40	46
Range (single digit display)	value	4	4	4	4	4		4
Pump Outlet Pres	psig	4.2	4.2	4.3	4.2	4.2		4.3
Air Pres (reg next to pump)	psig	8	7.9	7.9	7.9	7.9		7.9
Smple Pres. (Internal reg)	psig	4	4	4	4	4		4
Fuel Pres. (Internal reg)	psig	24	24	24	24	24		24
time	hh:mm	07:31	08:16	12:04	13:06	14:05		15:32

\*Located on floor 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 need be.

1311 added  
 10 g Sugar / l Feed

Reviewed and Approved: *RWJ*

Date: 4/23/01

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## Off-Gas Sampling Data Sheet

RSM-01-1

	By:	JAM	JMR	JAM	JAM	JAM			
	Date:	2/2/1	2/2/1	2/2/1	2/2/1	2/2/1			
	Time:	16:10	17:29	18:09	19:11	20:04			
DESCRIPTION	units								
Gas Chromatograph									
He	ppm	1139	1059	1127	1098	1076			
H <sub>2</sub>	ppm	0	0	0	0	0			
O <sub>2</sub>	ppm	44736	41764	43290	42006	40900			
N <sub>2</sub>	ppm	170515	159285	167950	160200	156020			
NO	ppm	-1	-1	-1	-1	-1			
CO	ppm	630	632	853	535	552			
CO <sub>2</sub>	ppm	1876	1740	1734	1664	1787			
N <sub>2</sub> O	ppm	0	0	0	0	178			
NOx	ppm	-	-NA						
NOx Analyzer Computer									
Time		4:12	17:28	18:09	19:12	20:05			
NOx	ppm	112	100-3	93	83	83			
NO	ppm	109	99	90	80	83			
NOx Voltage	Volts	2.26	2.02	1.87	4.25	4.27			
NOx Instrument									
Analog Reading	%FS	111	39	94	85	91			
Range selector	value	Auto	Auto	Auto	Auto	Auto			
Mode selector	value	NOx	NOx	NOx	NOx	NOx			
Sample Pressure	psig	4	4	4	4	4			
Sample bypass flow	scfm	5	6	6	6	6			
Ozone Pressure	psig	30	30	30	30	30			
Sierra Mass Flow: Flow#2	scfm	106.4	106.4	106.4	106.4	106.4			
MKS Mass Flow									
Ar Carrier, Chan-1	lpm	1.98	1.98	1.98	1.98	1.98			
He Tracer, Chan-2	lpm	1.99	1.99	1.99	1.99	1.99			
Total Sample, Chan-3	lpm	2.32	2.32	2.32	2.32	2.32			
N/A, Chan-4	N/A								
Sample Pump*									
Vac Gauge	"Hg"	1	8.5	1	1	1			
Pres Gauge	psig	10.5	3.5	10.5	10.5	10.5			
Gas Cylinders Tank Pres*									
O <sub>2</sub>	psig	1100	1080	1070	1050	1020			
Ar GC	psig	2600	2500	2500	2500	2500			
Ar Sweep #1	psig	930	910	900	860	810			
Ar Sweep #2	psig	2000	2000	2000	2000	2000			
He GC	psig	1620	1620	1620	1620	1620			
He Tracer #1	psig	1060	1020	990	950	910			
He Tracer #2	psig	2530	2530	2520	2520	2570			
Fid Analyzer (in Hood)									
Conc. (multi-digit display)	value	44	45	40	35	45			
Range (single digit display)	value	4	4	4	4	4			
Pump Outlet Pres	psig	4.3	4.3	4.3	4.3	4.3			
Air Pres (reg next to pump)	psig	7.8	7.9	7.9	7.9	7.9			
Smple Pres, (Internal reg)	psig	4	4	4	4	4			
Fuel Pres, (Internal reg)	psig	24	24	24	24	24			
time	hh:mm	4:19	5:39	6:14	7:16	8:08			

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

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

Reviewed and Approved:

Date: 4/23/01

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## **Appendix D**

### **Colorimetric Procedure for Determining Fe(II) to Total Iron Ratio**



## Iron II and Total Iron Ratio

### 1.0 APPLICABILITY

This procedure is applicable for the determination of Iron II by completing the ferrous ion with 1, 10-phenanthroline. The concentration of the orange-red complex is 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 (Iron II).
- 2.2 Hydroquinone - An organic reducing agent that will reduce ferric ion (Iron III) to ferrous ion Iron II in an aqueous solution at room temperature.
- 2.3 Standard blank - The Total Iron (Iron II + Iron III) concentration in all the reagents and the water used in this method.
- 2.4 Reagent blank - The Iron II 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 100 ppm certified iron solution is used to prepare the calibration curve.
- 4.2.2 0.25% Phenanthroline Solution – Weigh  $0.25 \pm 0.05$  gram of phenanthroline and dissolve in 100 ml of iron free water.
- 4.2.3 4% Boric, Acid Solution - Weigh  $40 \pm 4$  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  $100 \pm 10$  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 gloves 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 100 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 IRON II 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 dropwise 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  $20 \pm 10$  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 IRON II 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 y 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 IRON II 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  $\pm 5\%$  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 Iron II analyses. A copy of the signed and approved IRON II 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 an 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 Iron II Analyses

- 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 glass stir-bar,
  - 25 ml of boric acid solution,
  - 7 ml of KHP solution,
  - 6 ml of 4% phenanthroline solution and in a fume hood, pipet,
  - 2 ml of concentrated ammonium hydroxide.
- 4.8.2 To a second set 100 ml disposable plastic beakers, weight  $0.025 \pm 0.010$  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 IRON II AND TOTAL IRON DATA SHEET (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 Iron II 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 II 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 Total Iron 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 II absorbance reading onto the Xerox copy of the IRON II AND TOTAL IRON DATA SHEET in the column marked Fe II 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 IRON II AND TOTAL IRON DATA SHEET. Repeat reading and recording of the absorbance until all the samples in step 4.8.11 are completed.

#### 4.9 Total Iron Analyses

Soluble iron in an acidic aqueous solution is present either as ferrous or ferric ion. Hydroquinone will reduce all ferric ion (Iron III) in the solution to a ferrous ion (Iron II). With the reduction of all the Iron III to Iron II, the analyses of Iron II in the solution with the phenanthroline are the Total Iron 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  $20 \pm 10$  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 Total Iron absorbance of the solution. Record the Total Iron absorbance on the Xerox copy of the IRON II AND TOTAL IRON DATA SHEET in the column labeled tot Fe Abs. Also record the volume of solution pipetted in step 4.9.1 (divided by 100) onto the IRON II AND TOTAL IRON DATA SHEET.

#### 4.10 Calculation

The Iron II and Total Iron are calculated by entering the values from the IRON II AND TOTAL IRON DATA SHEET onto the first page of the Excel program labeled “FERATIO4.XLS” and titled IRON II AND TOTAL IRON 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 IRON II AND TOTAL IRON 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” 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  $0.20 \pm 0.01$  mg of Total Iron /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  $0.005 \pm 0.002$  mg of Fe/100 ml of solution, notify the Responsible Scientist.

#### 4.12 Archiving of Data

- 4.12.1 A copy of all the IRON II CALIBRATION CURVE analysis performed on the Milton Roy, “Spectronic”, model 601 spectrophotometer are posted in the notebook for this instrument. The calibration curve used for the Iron II analyses is identified by the slope (m), the intercept (y), and correlation coefficient (r) recorded on the IRON II AND TOTAL IRON DATA SHEET, the IRON II ANAD TOTAL IRON DATA ENTRY SHEET, and the ANALYSIS OF IRON AND TOTAL IRON forms.

- 4.12.2 The completed IRON II AND TOTAL IRON 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 IRON II AND TOTAL IRON form sent to the customer shall be stored in the sample data file stored under the Laboratory Number.



**Figure 1**

**IRON II 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**

**IRON II AND TOTAL IRON 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 (FeII) Absorbance = \_\_\_\_\_

Sample blank (Fe Tot) Absorbance = \_\_\_\_\_

Row	Laboratory Number	Customer's Sample ID	Sample Wt	Fe II Dil	Fe II Abs	Tot Fe Dil	Tot Fe 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

#### IRON II AND TOTAL IRON 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 (FeII) Absorbance = \_\_\_\_\_

Sample blank (Fe Tot) Absorbance = \_\_\_\_\_

Row	Laboratory Number	Customer's Sample ID	Sample Wt	Fe II Dil	Fe II Abs	Tot Fe Dil	Tot Fe 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 IRON II AND TOTAL IRON**

Analytical and Process Support Laboratory

To:

The Iron II (Fe II) 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 colorimetric 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 II	Wt % Fe Tot	Fe II/Fe Tot	Precipitate		If yes, comment
					Yes	No	

Comments: 1. Dark non-magnetic precipitate.

2. Dark magnetic precipitate.

3. Light colored (whitish) precipitate.

4. Other

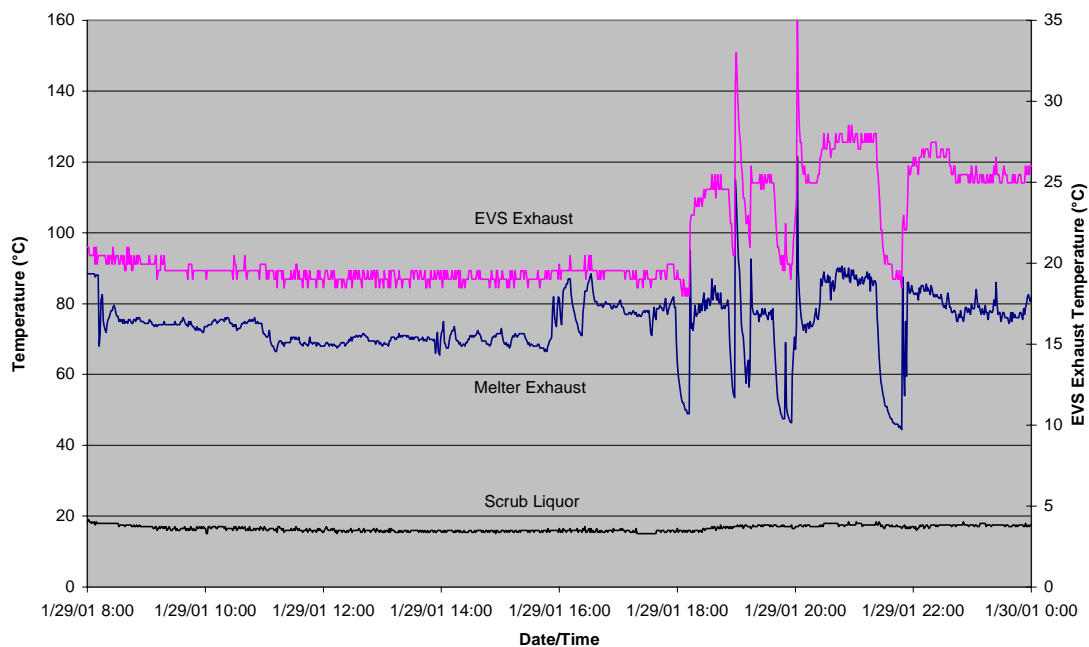
Analyst signature and date

Approved by and date

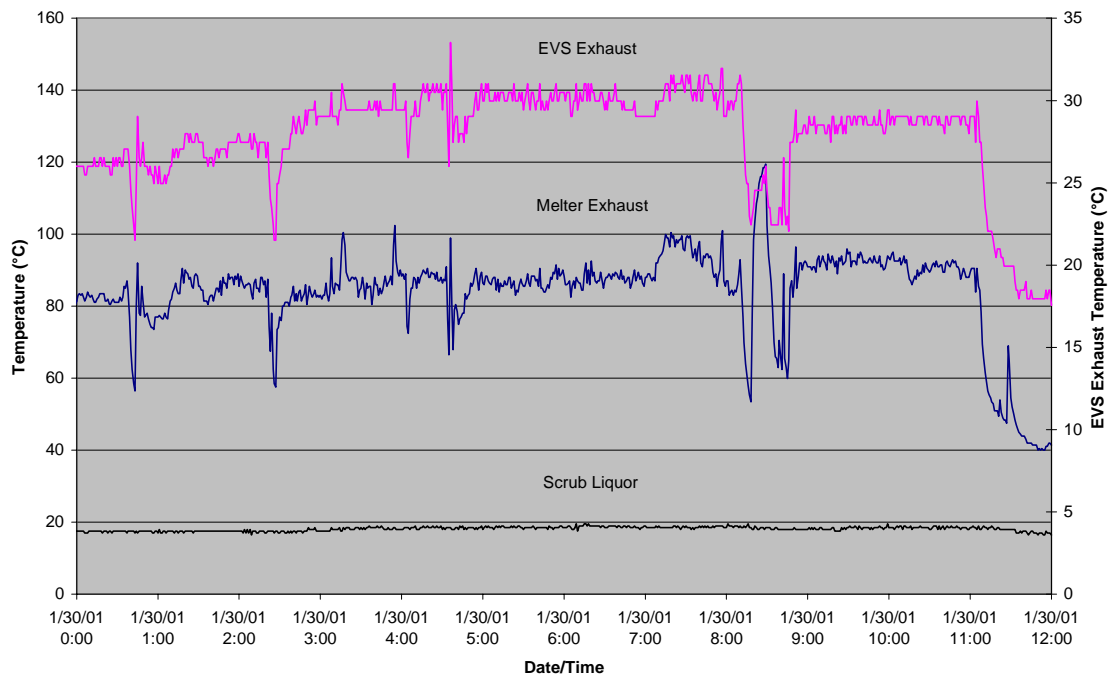
## **Appendix E**

### **Variable Process-Temperature Data Collected During the SBW Flowsheet Evaluations**

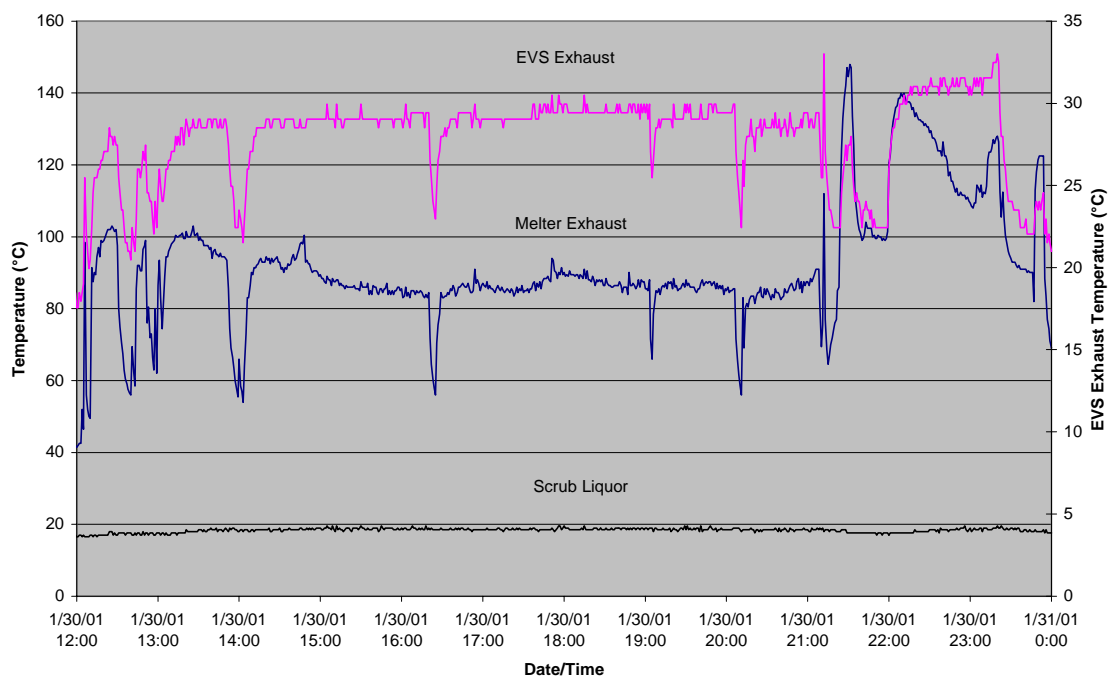
Off-Gas Processing System Temperatures



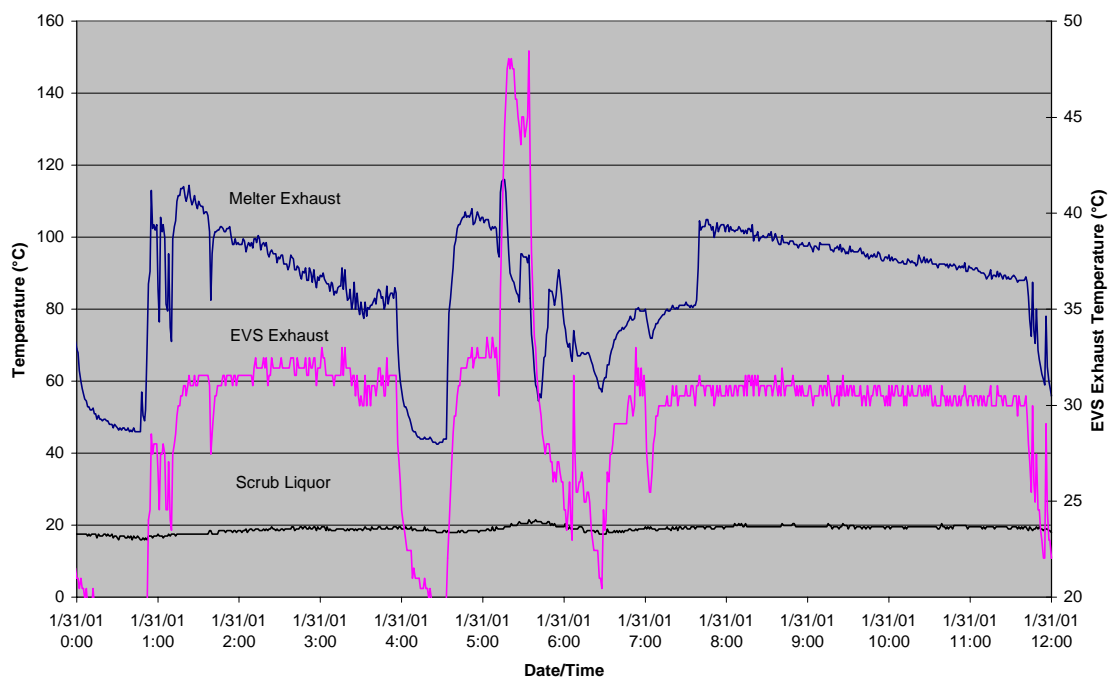
Off-Gas Processing System Temperatures

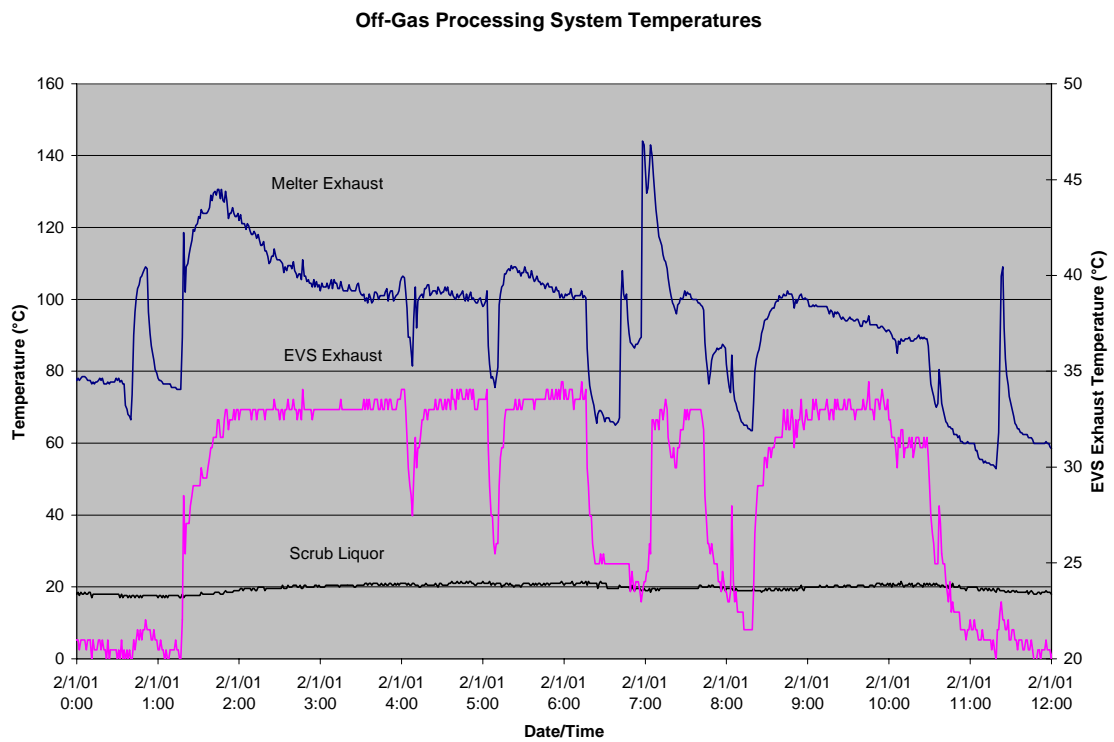
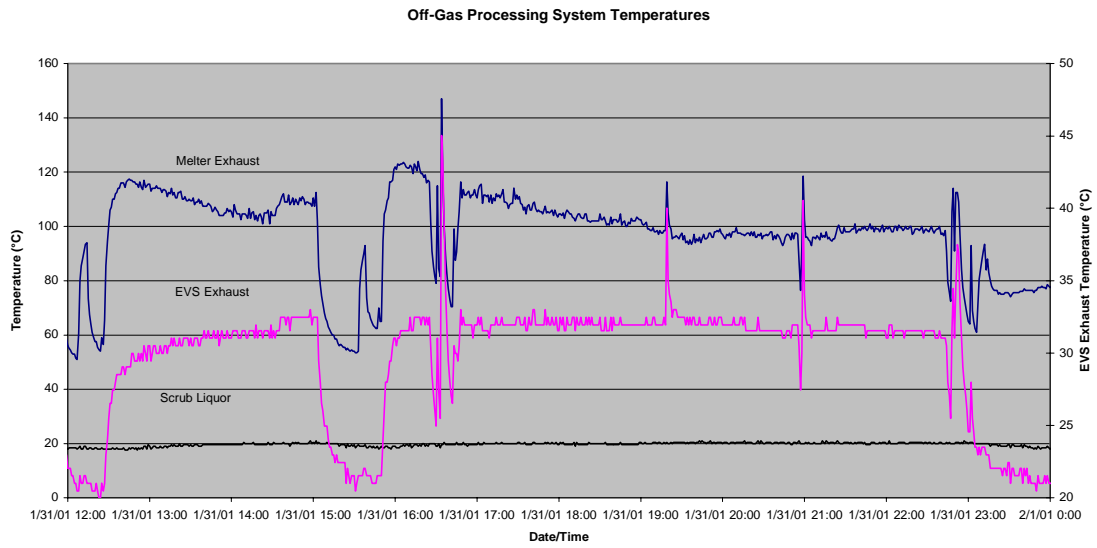


Off-Gas Processing System Temperatures



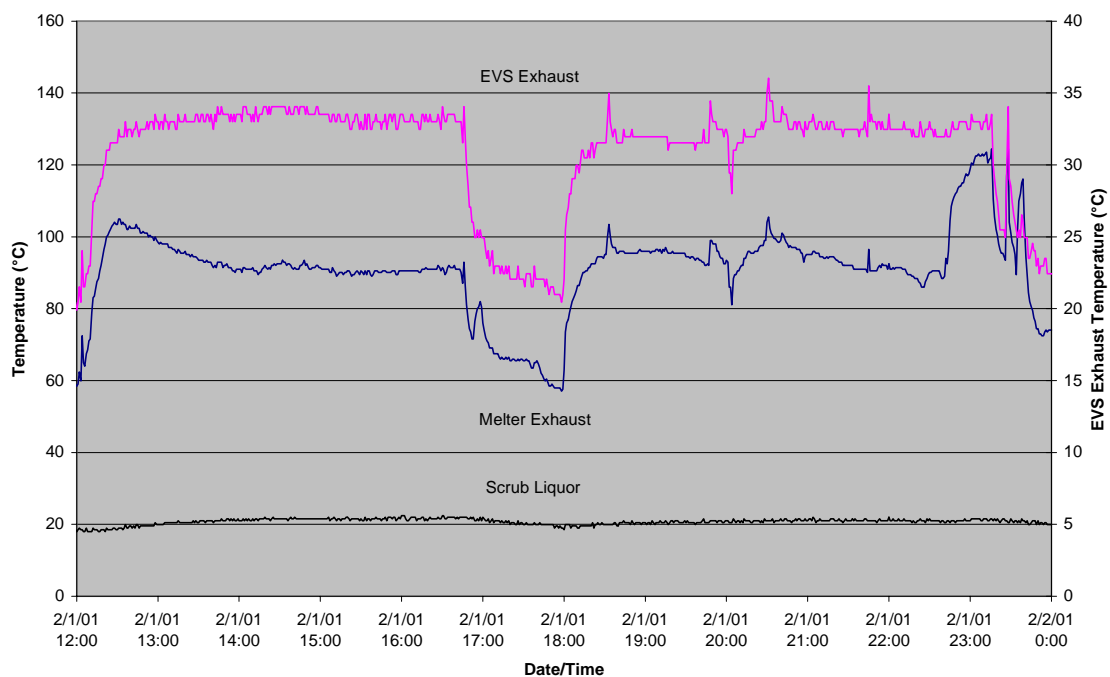
Off-Gas Processing System Temperatures



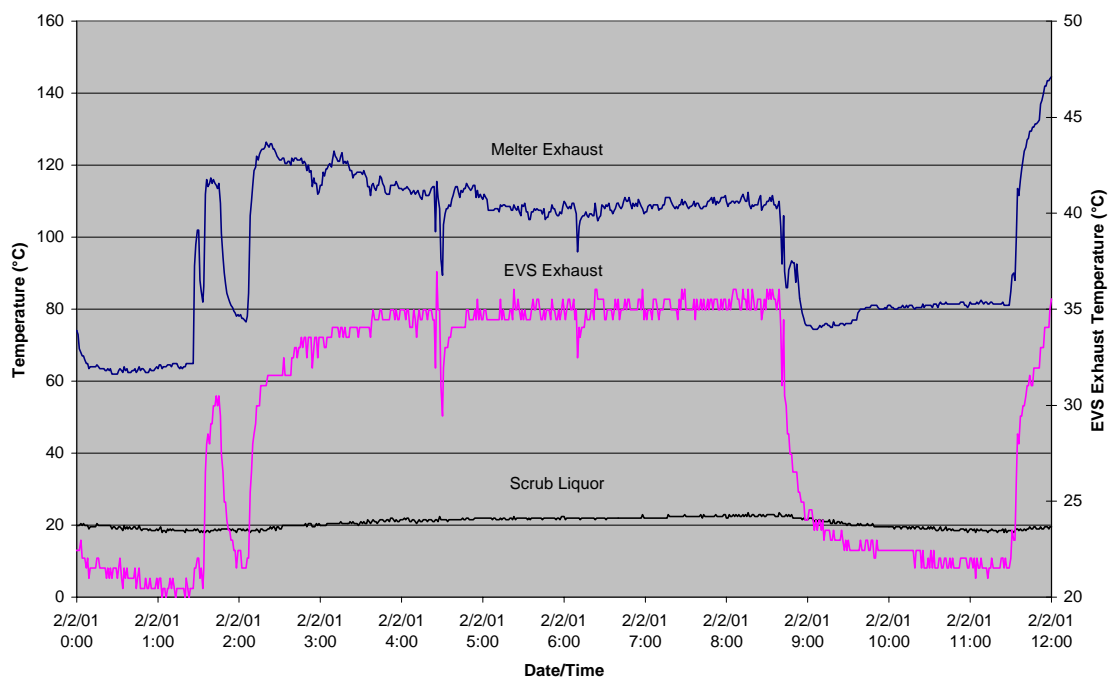




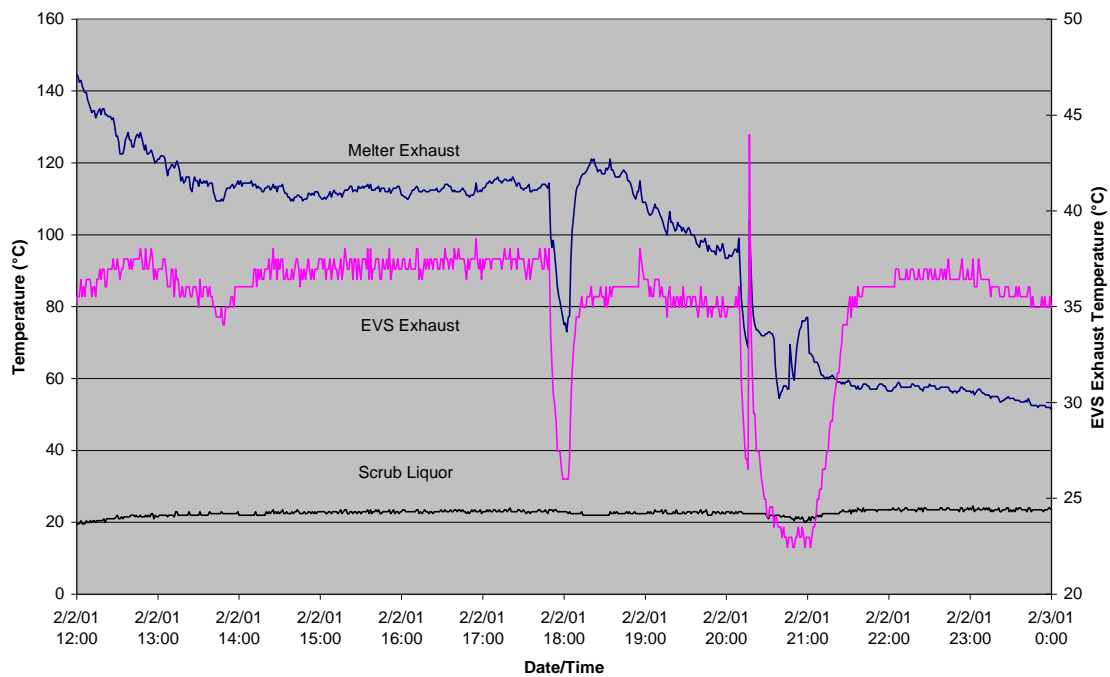
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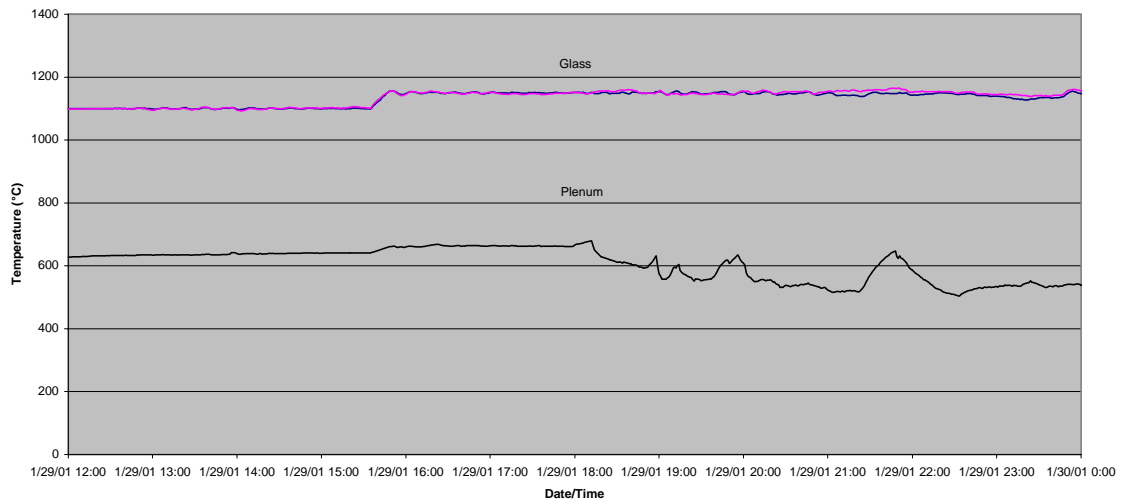
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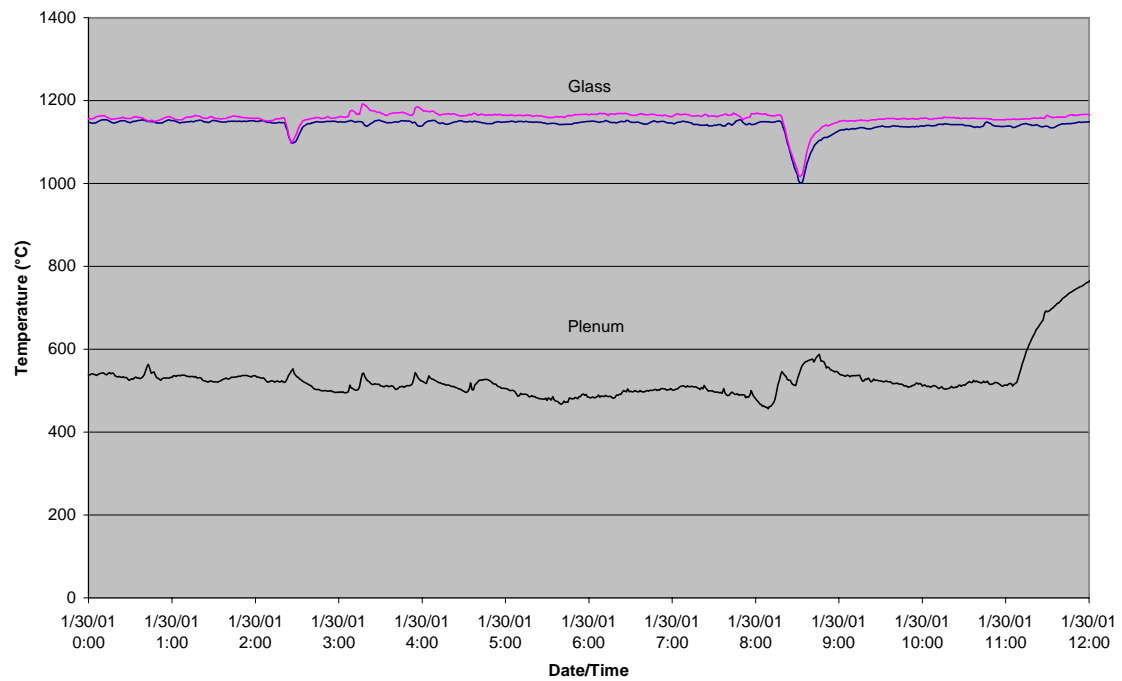
Off-Gas Processing System Temperatures



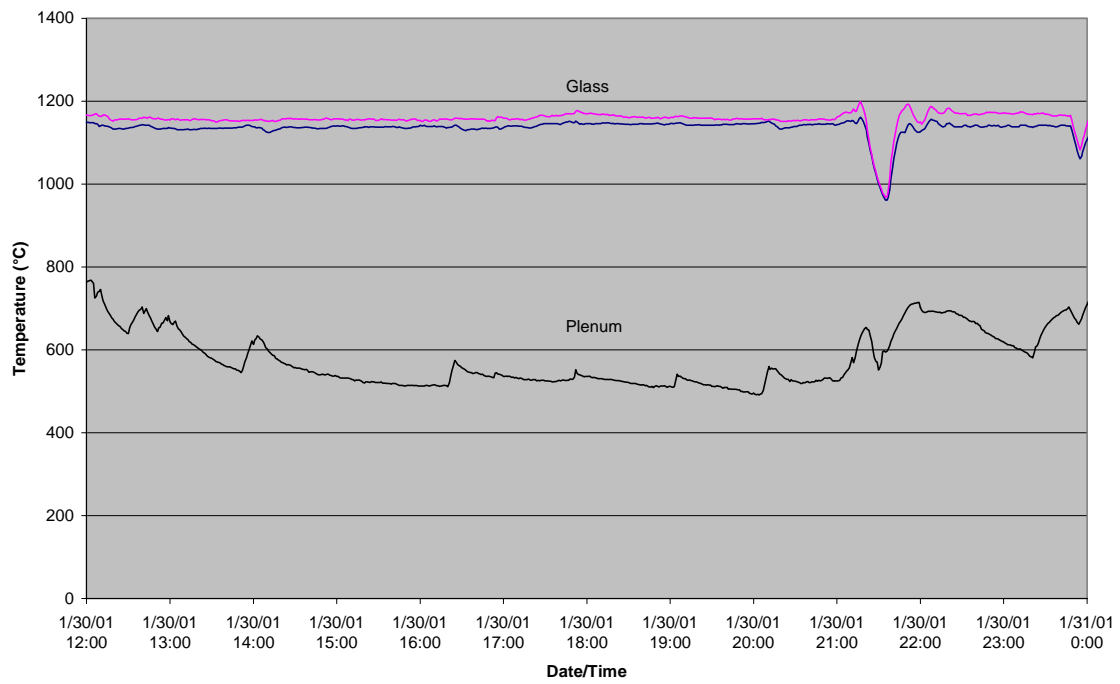
Melter Operating Temperature Characteristics



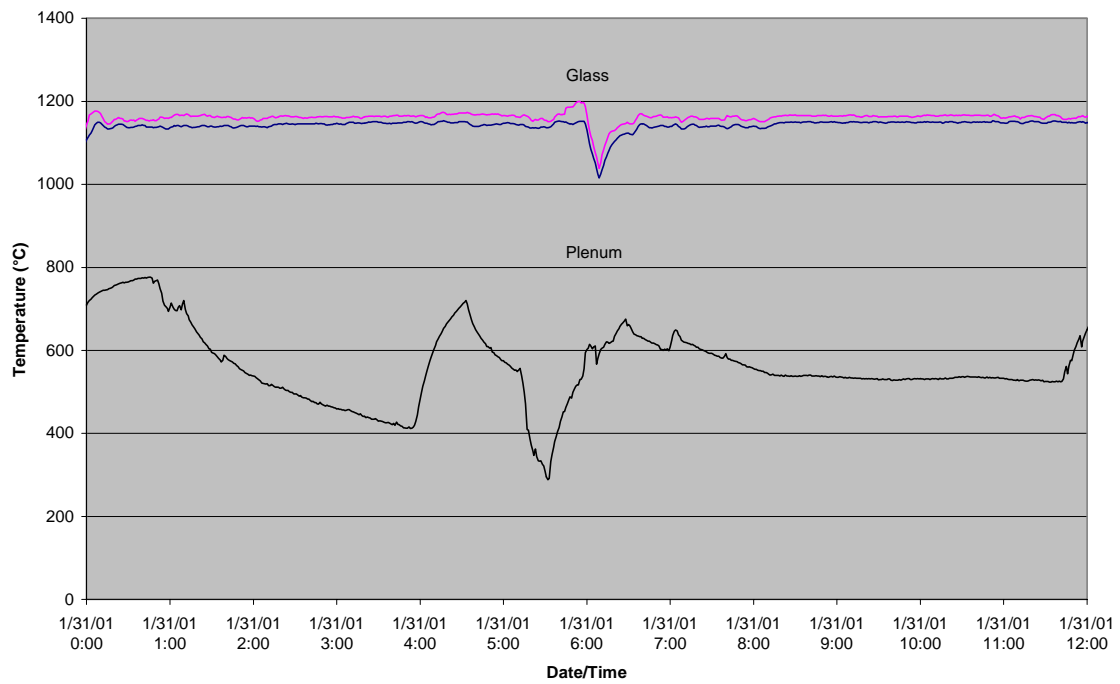
Melter Operating Temperature Characteristics



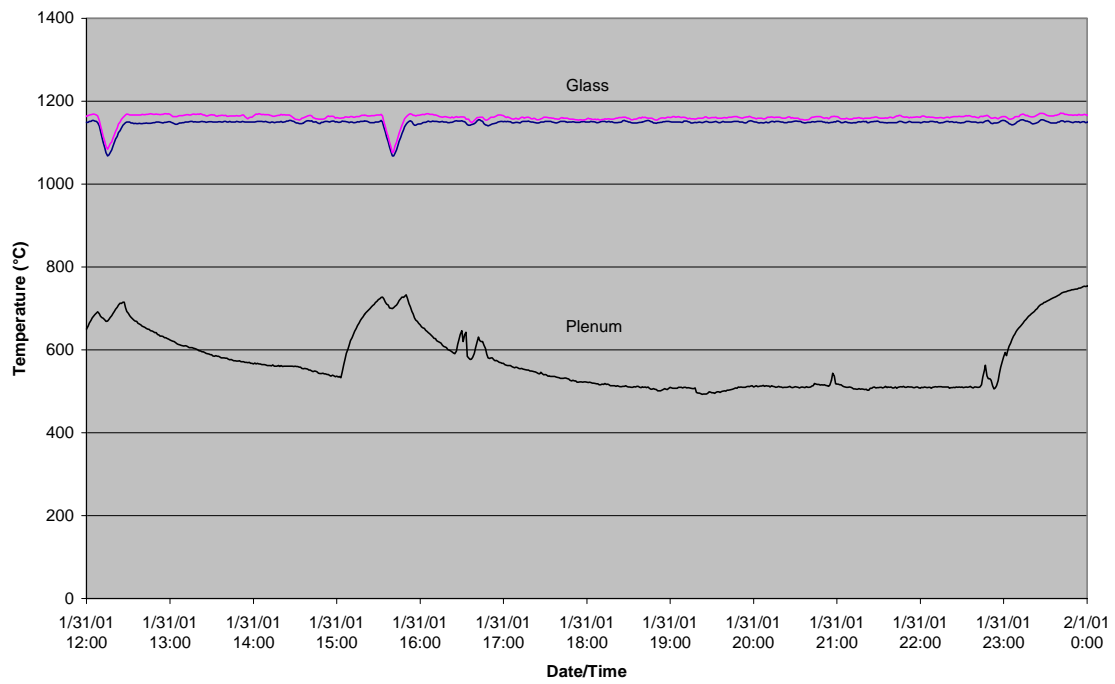
**Melter Operating Temperature Characteristics**



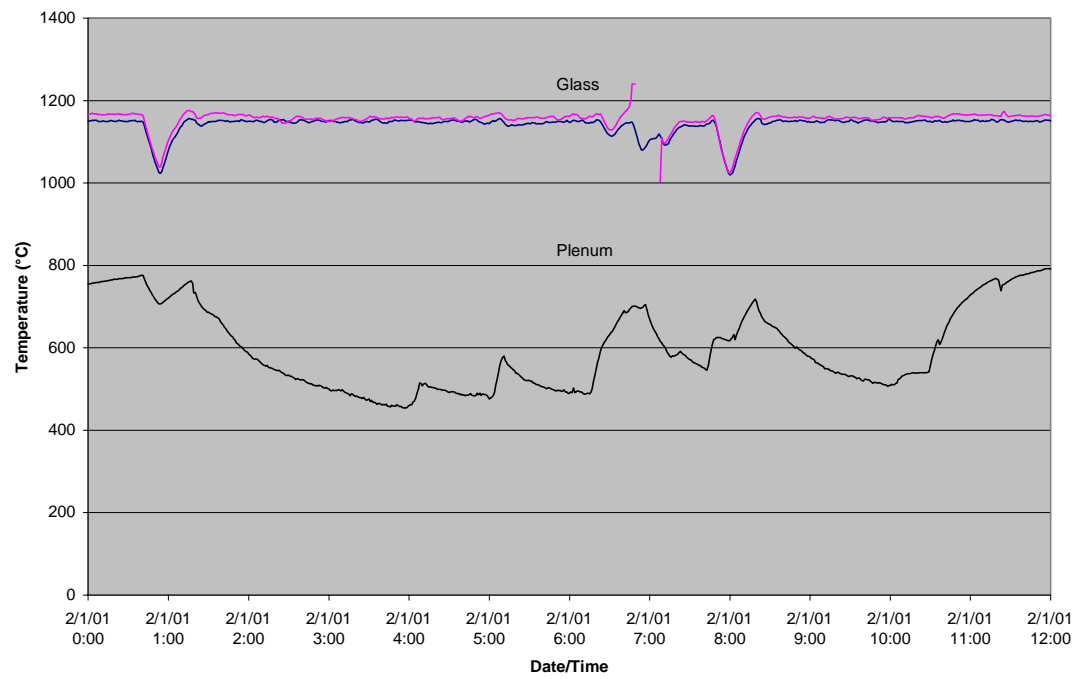
**Melter Operating Temperature Characteristics**



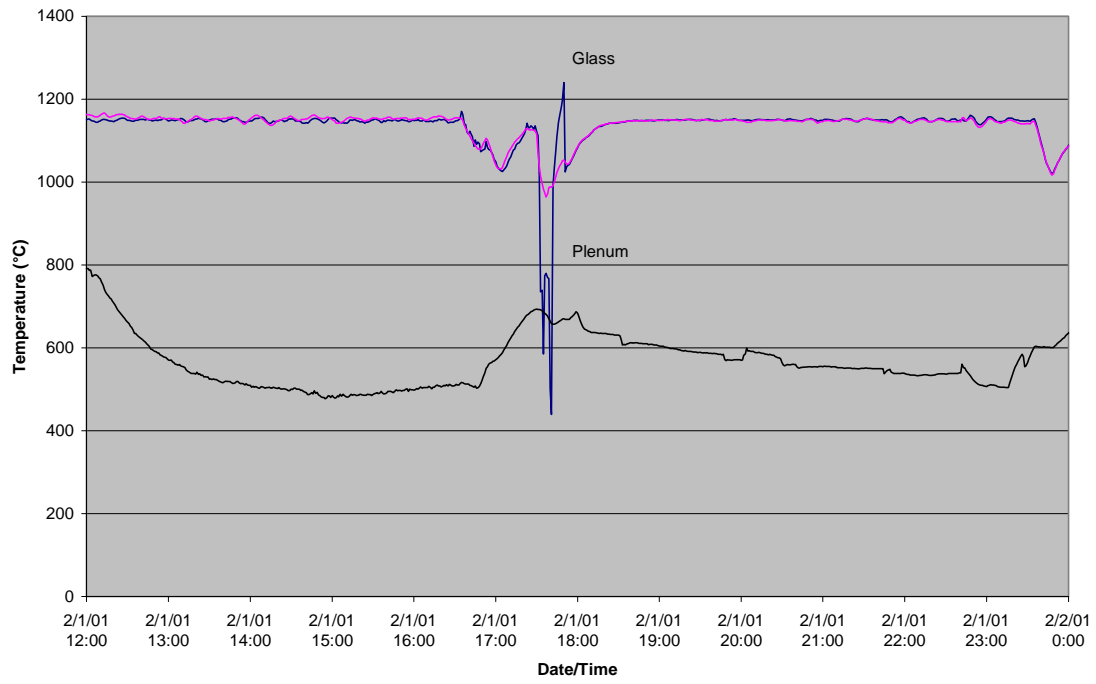
**Melter Operating Temperature Characteristics**



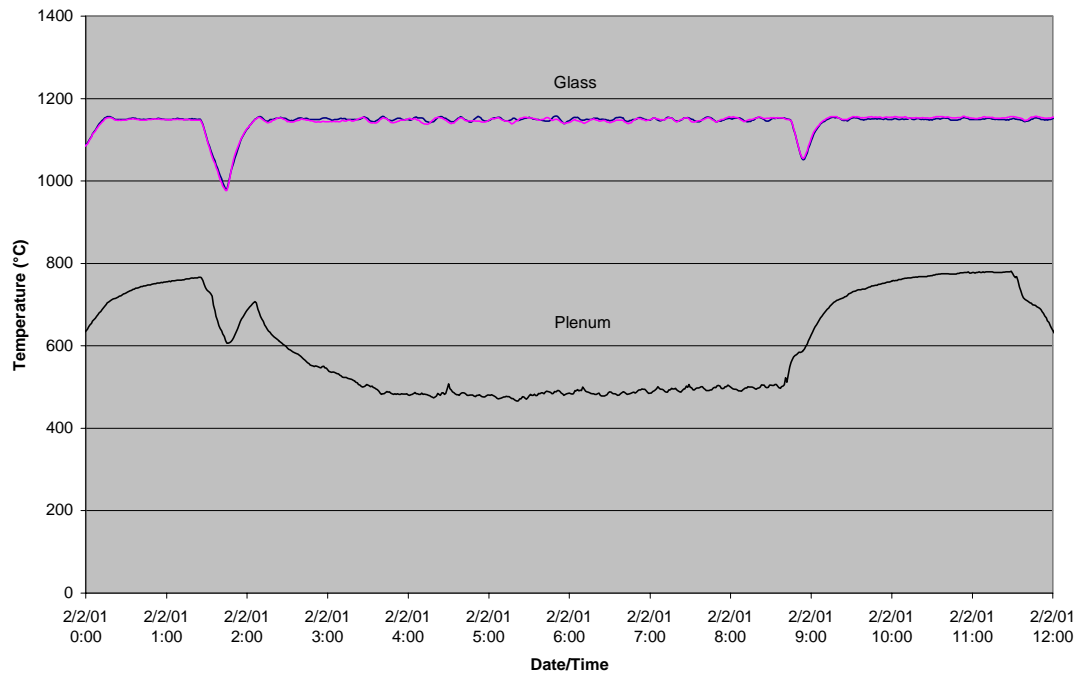
**Melter Operating Temperature Characteristics**



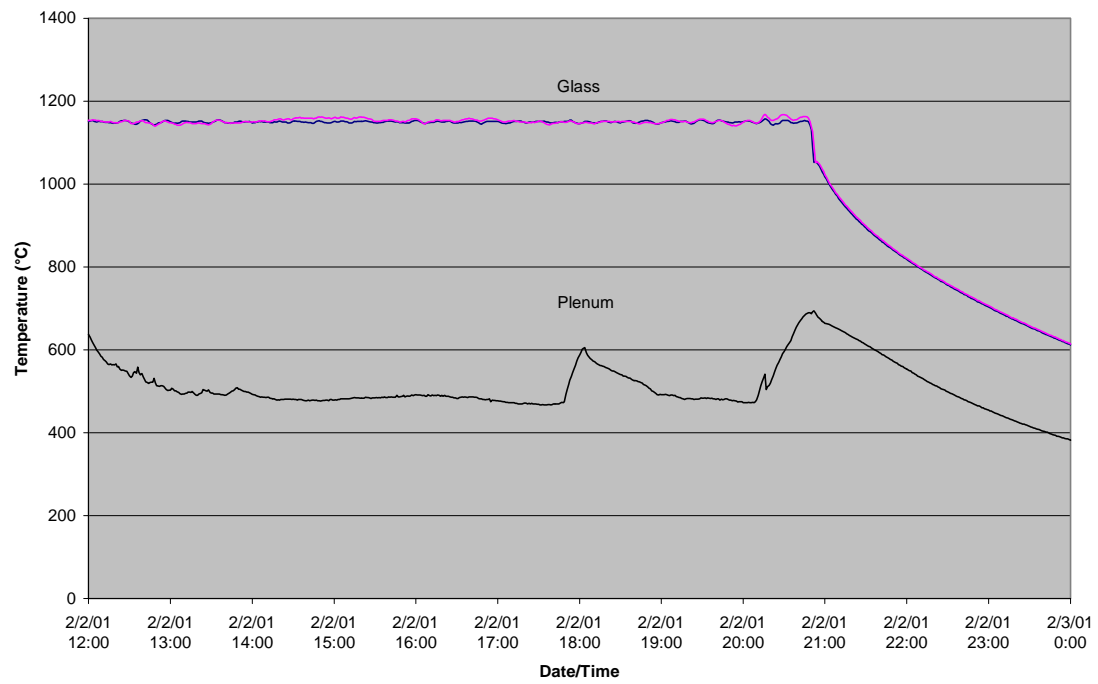
Melter Operating Temperature Characteristics



Melter Operating Temperature Characteristics



### Melter Operating Temperature Characteristics

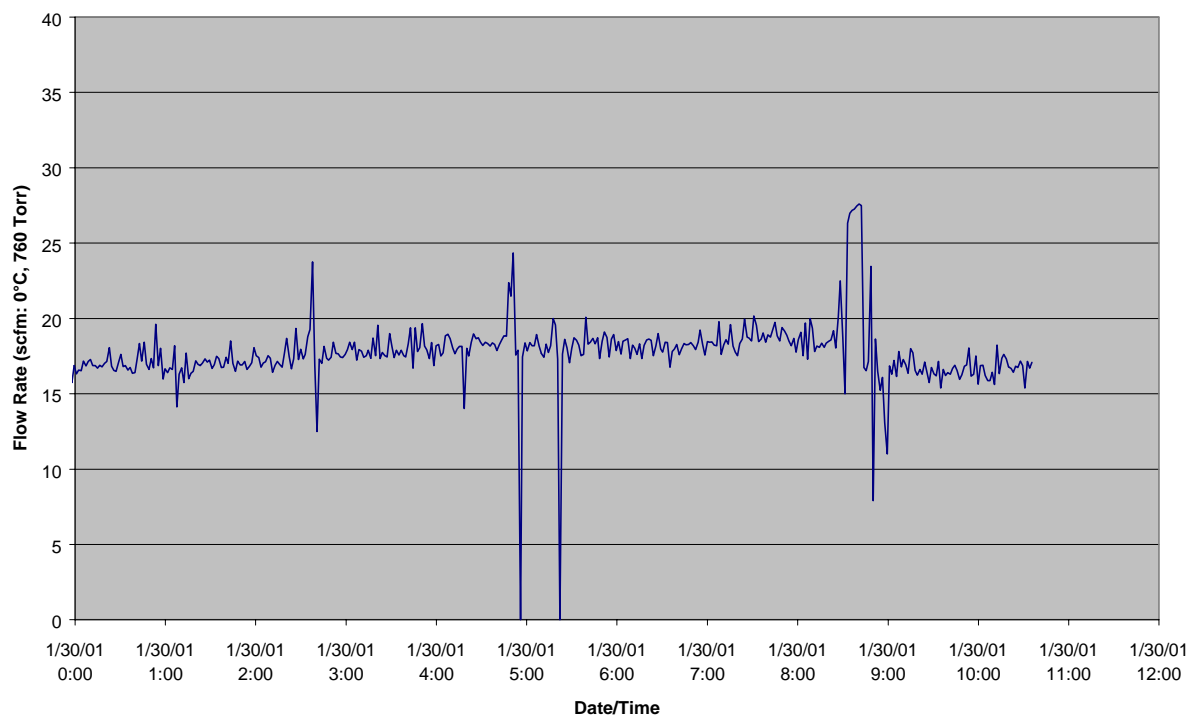


## **Appendix F**

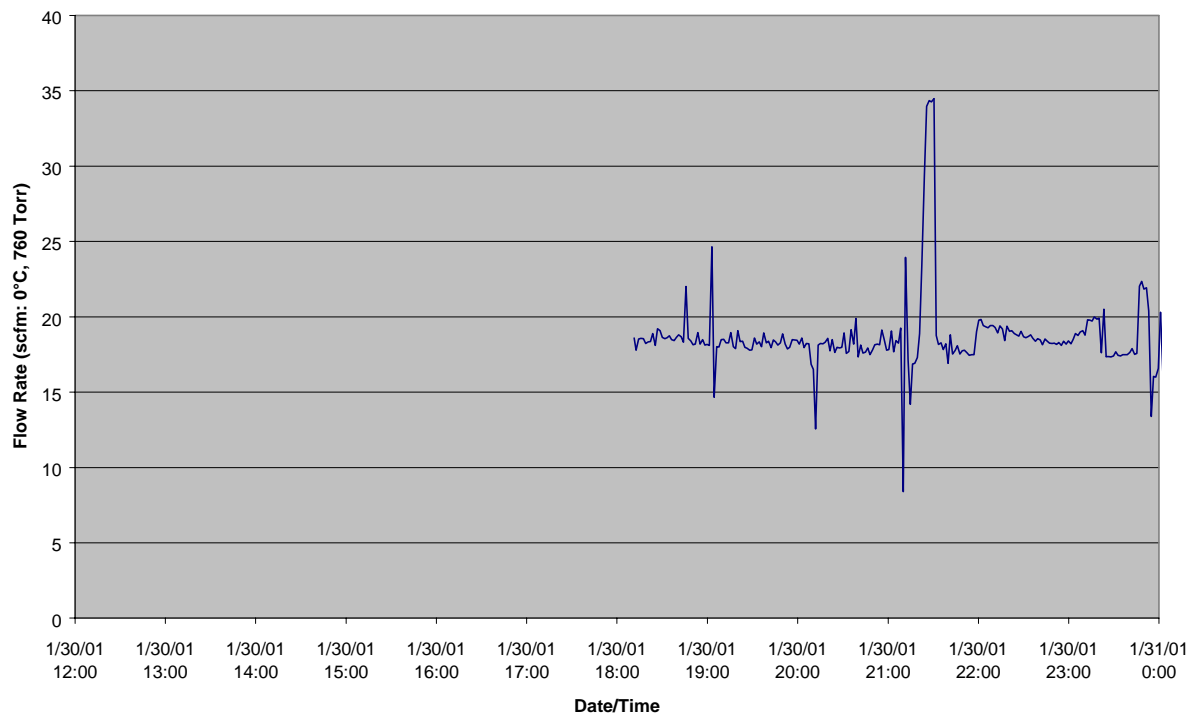
### **Total Unquenched Melter Flow Rates**



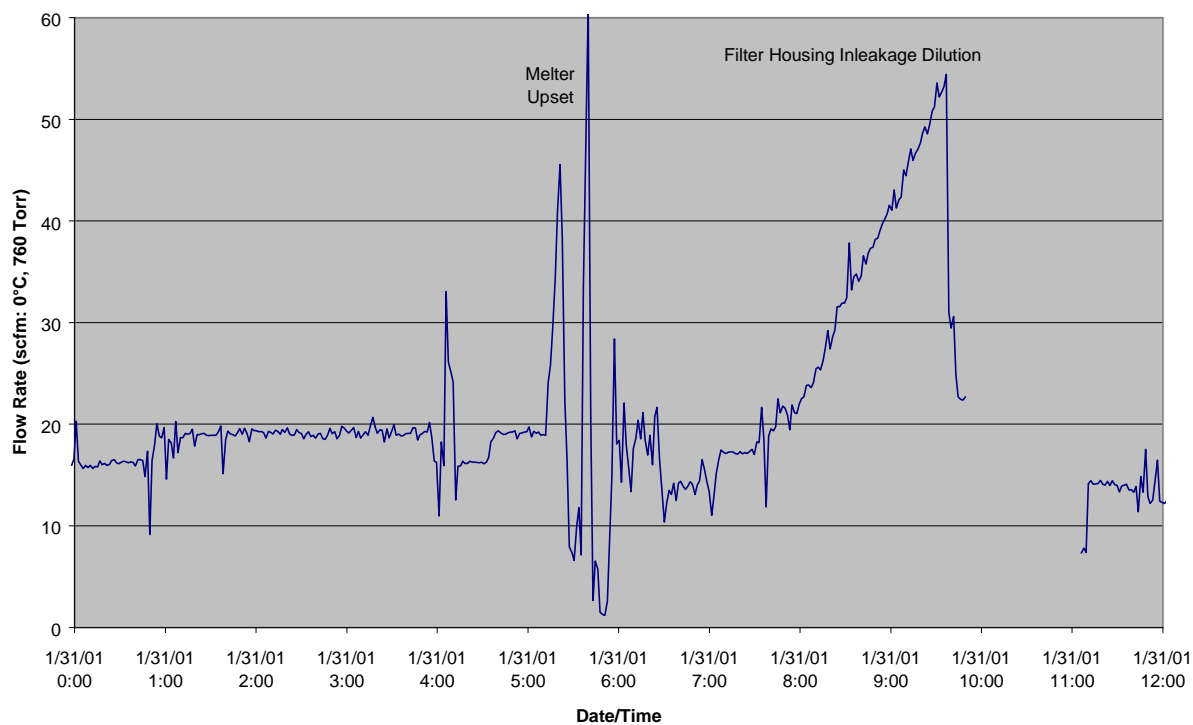
**Total, Unquenched Off-Gas Flow Rate**



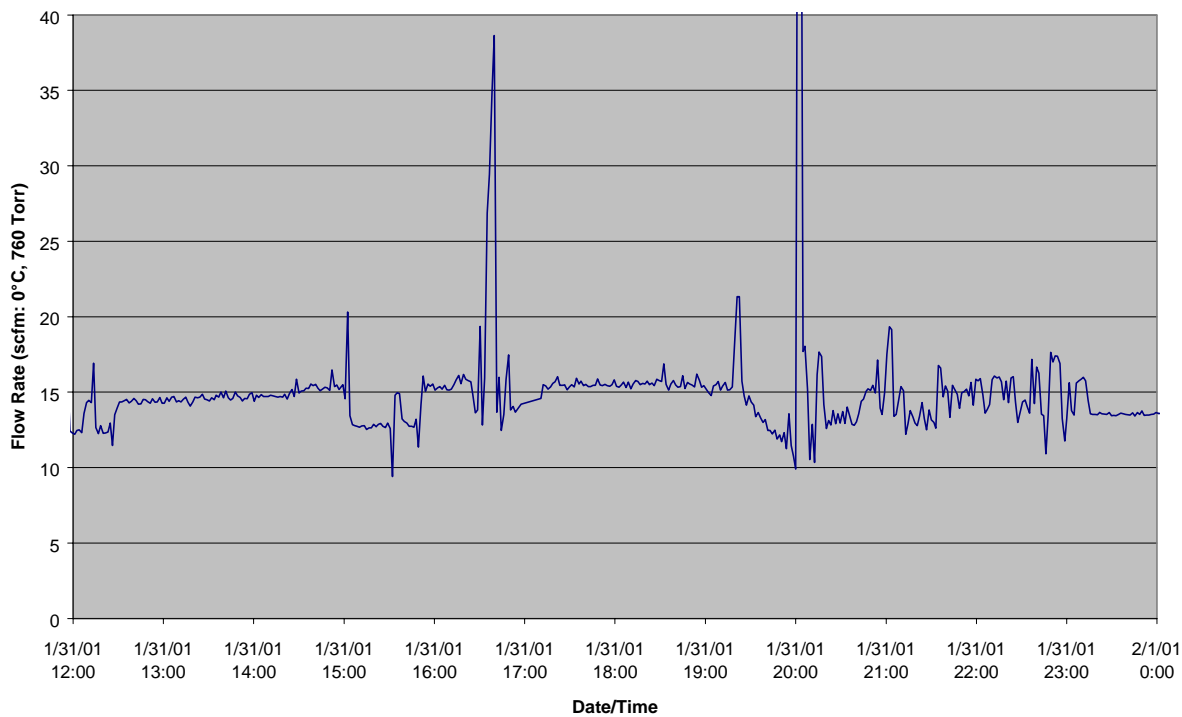
**Total, Unquenched Off-Gas Flow Rate**



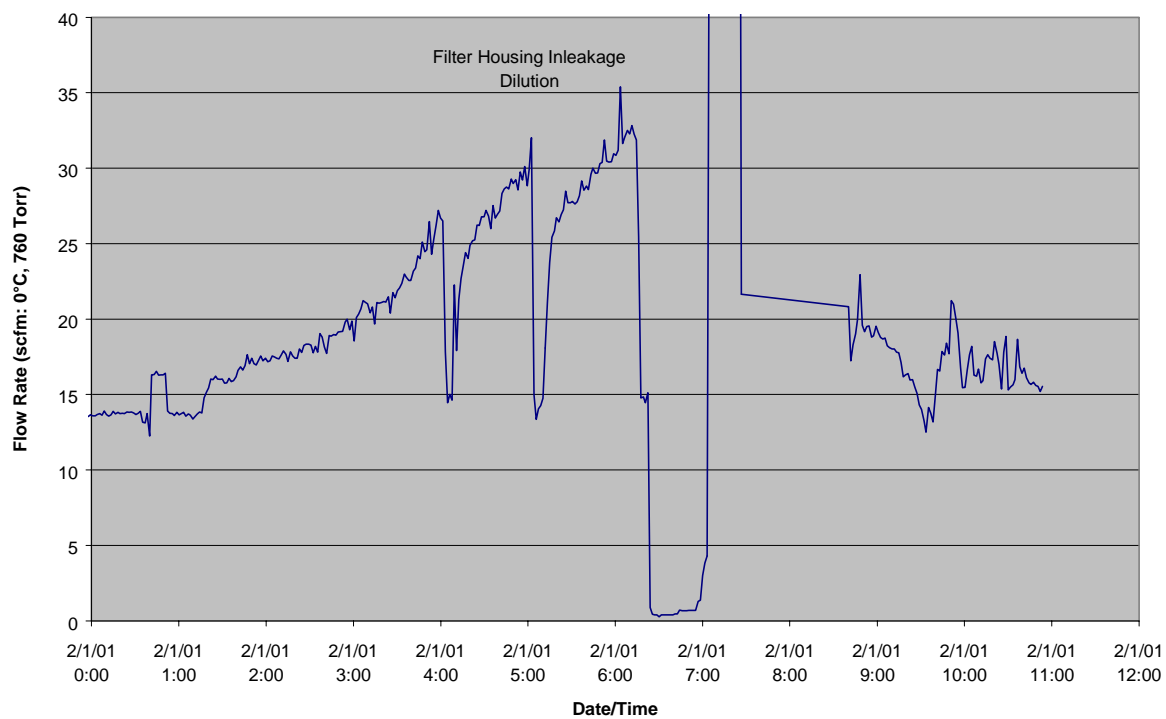
**Total, Unquenched Off-Gas Flow Rate**



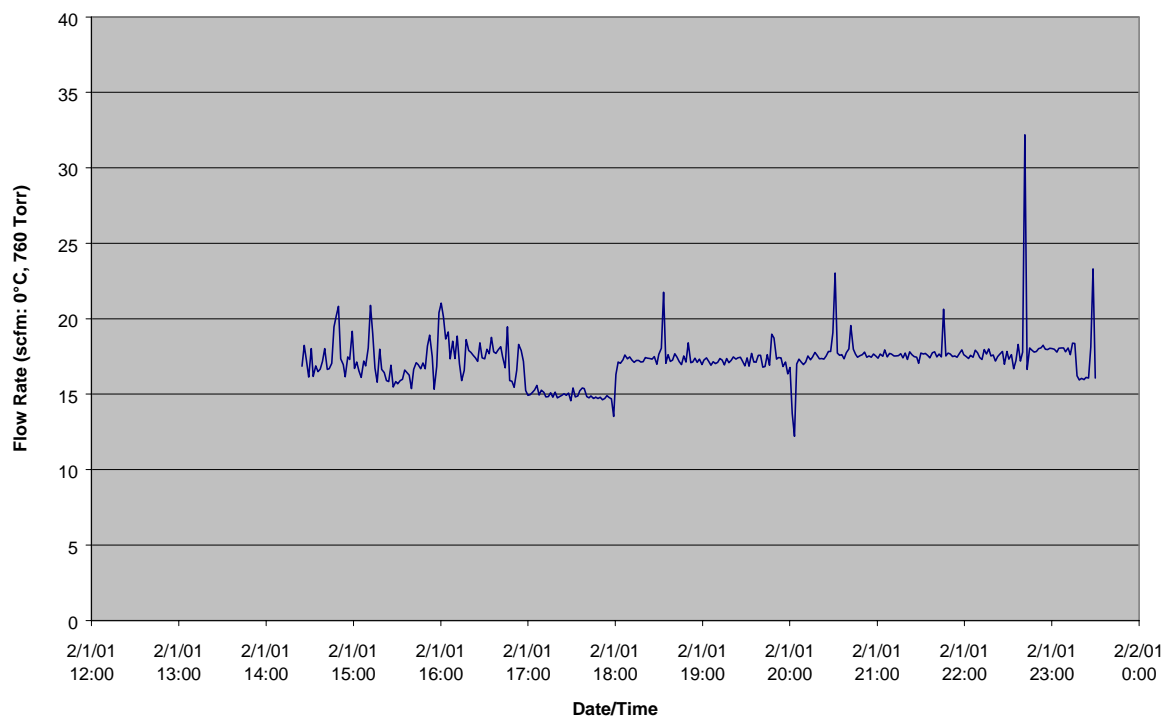
**Total, Unquenched Off-Gas Flow Rate**



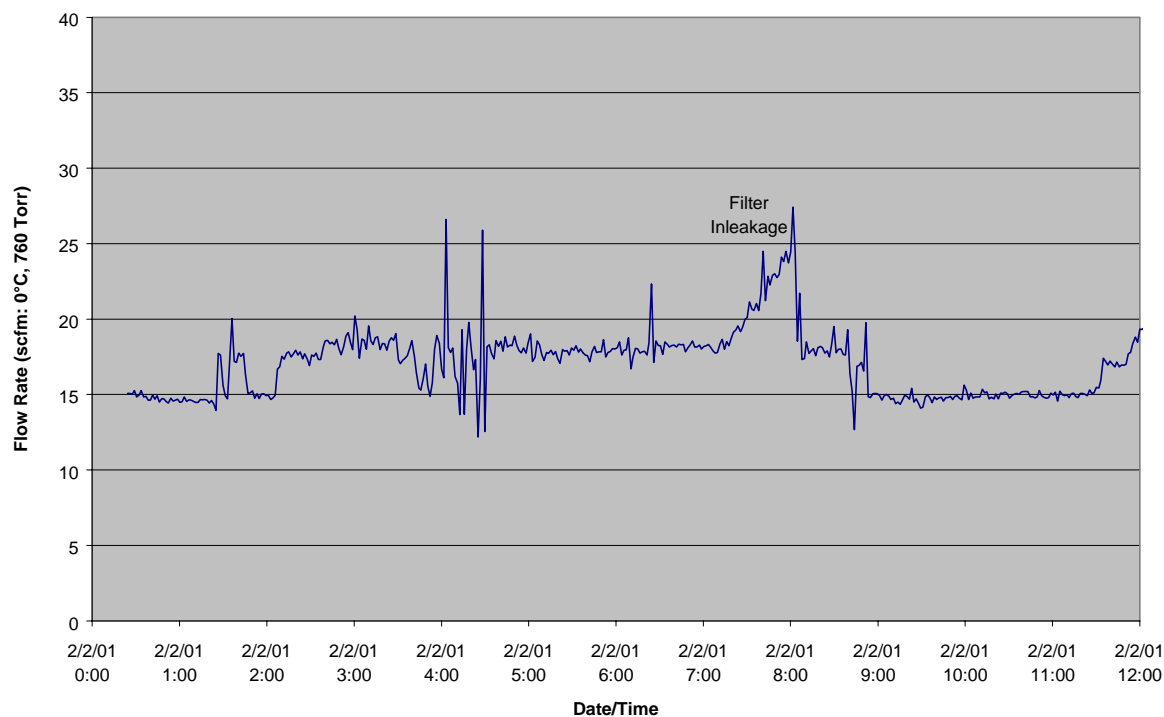
**Total, Unquenched Off-Gas Flow Rate**



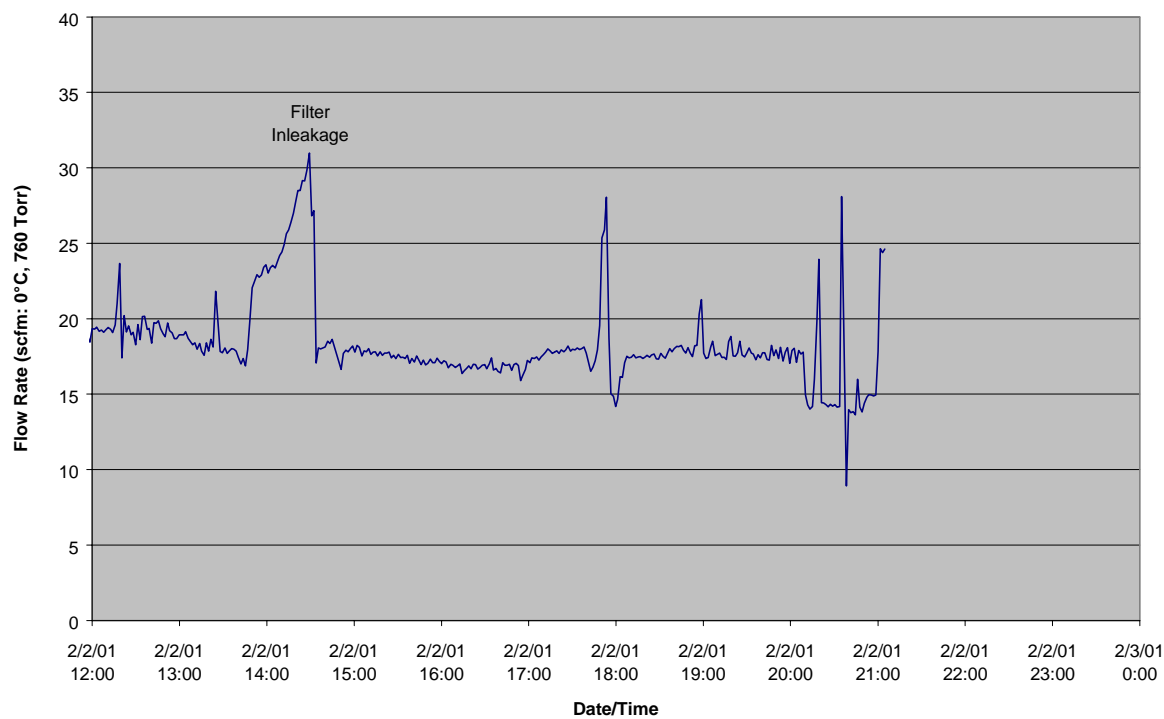
**Total, Unquenched Off-Gas Flow Rate**



Total, Unquenched Off-Gas Flow Rate



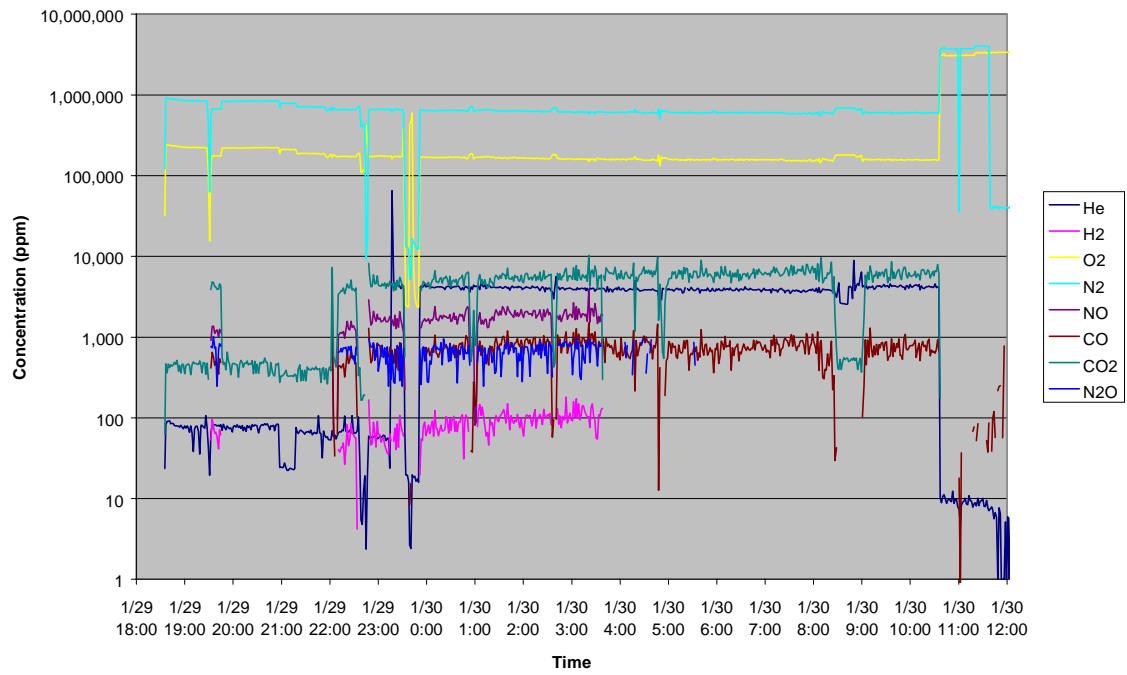
Total, Unquenched Off-Gas Flow Rate



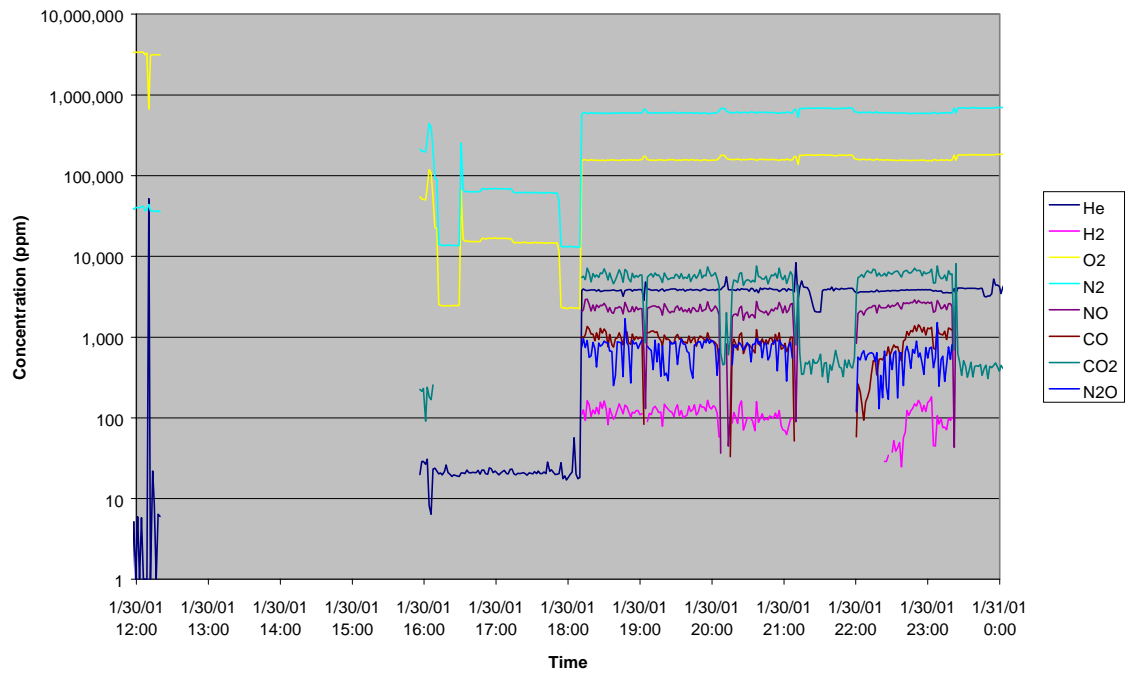
## **Appendix G**

### **Temporal Behavior of Melter Off-Gas Effluents**

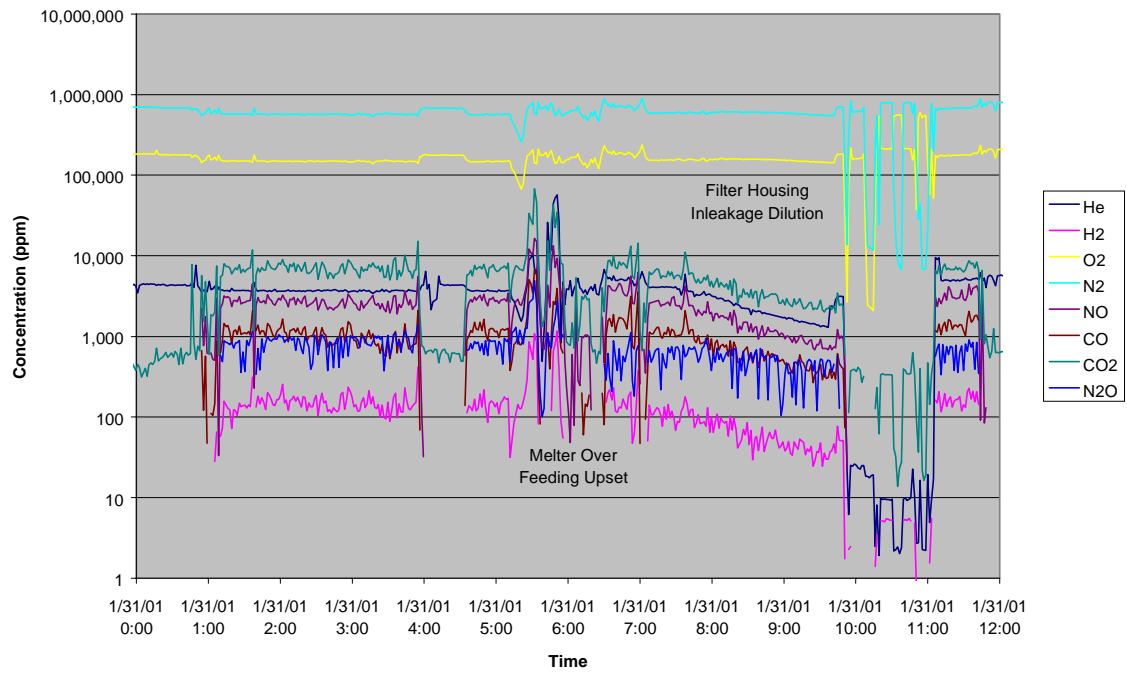
### Gas Chromatographic Off-Gas Analysis



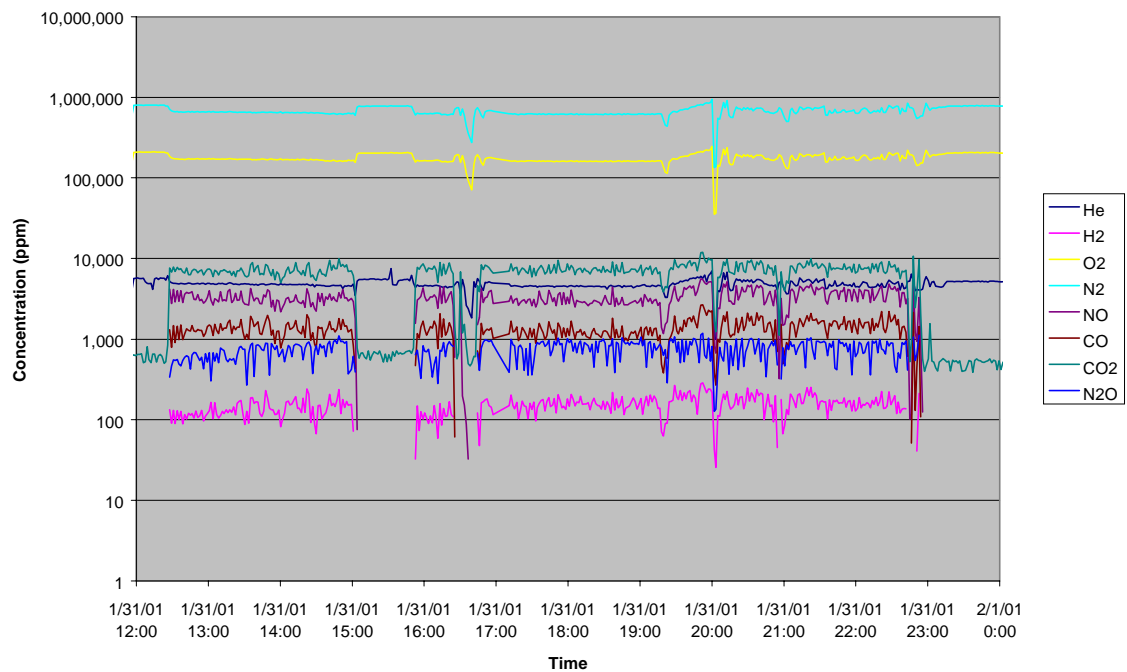
### Gas Chromatographic Off-Gas Analysis



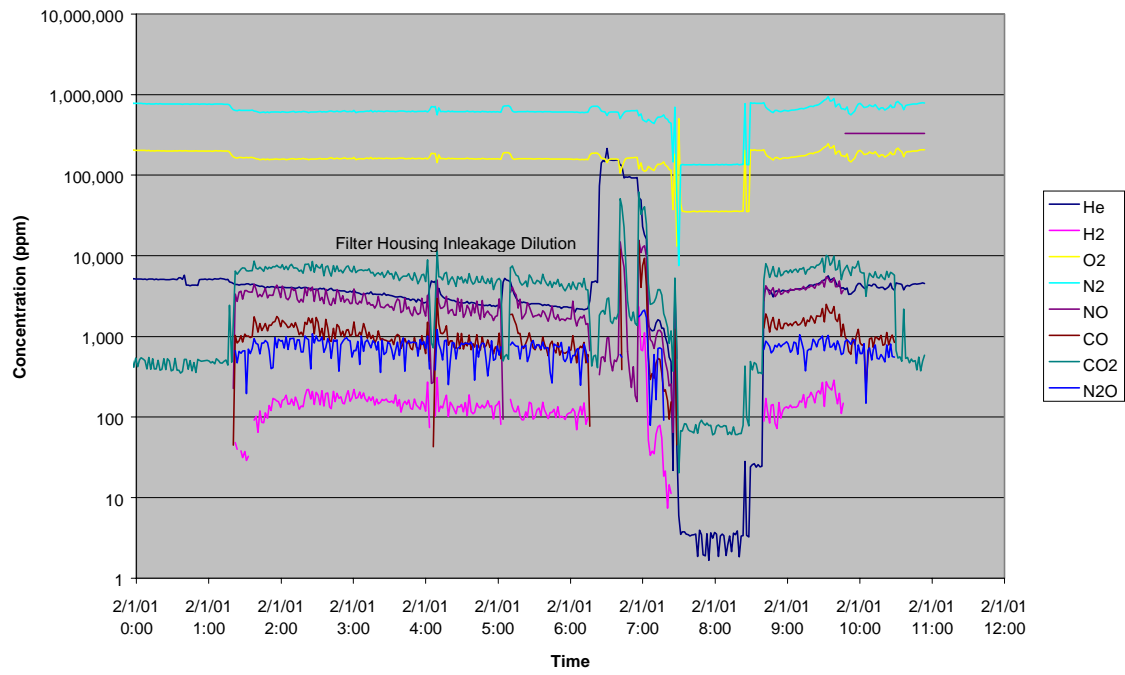
### Gas Chromatographic Off-Gas Analysis



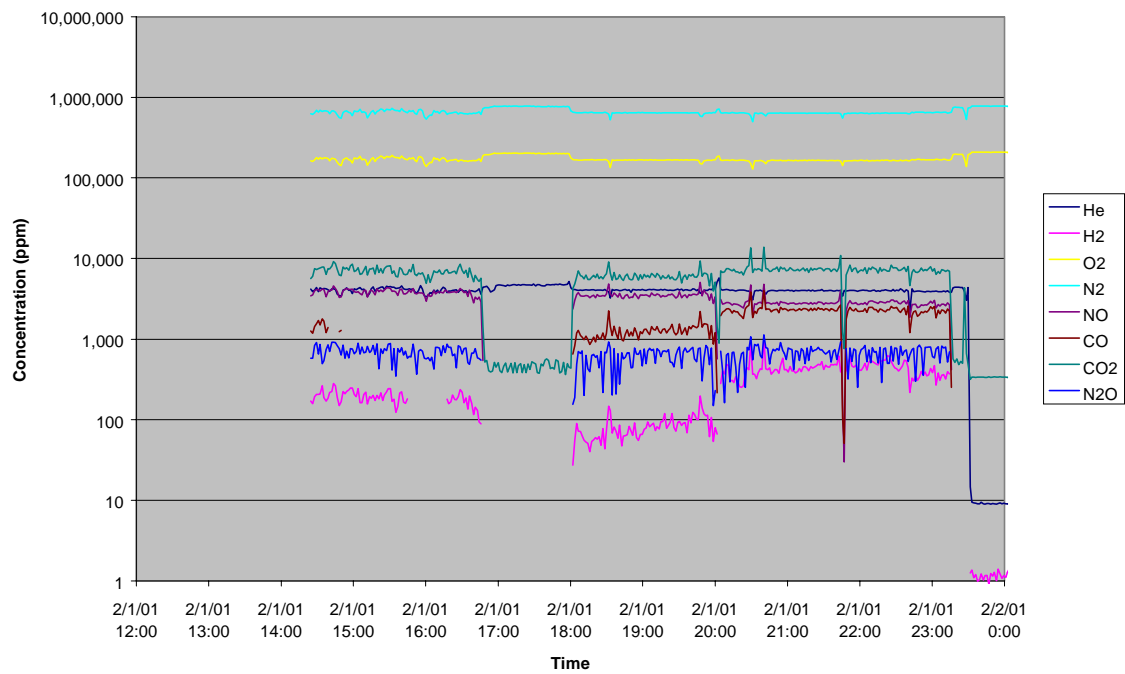
### Gas Chromatographic Off-Gas Analysis



### Gas Chromatographic Off-Gas Analysis

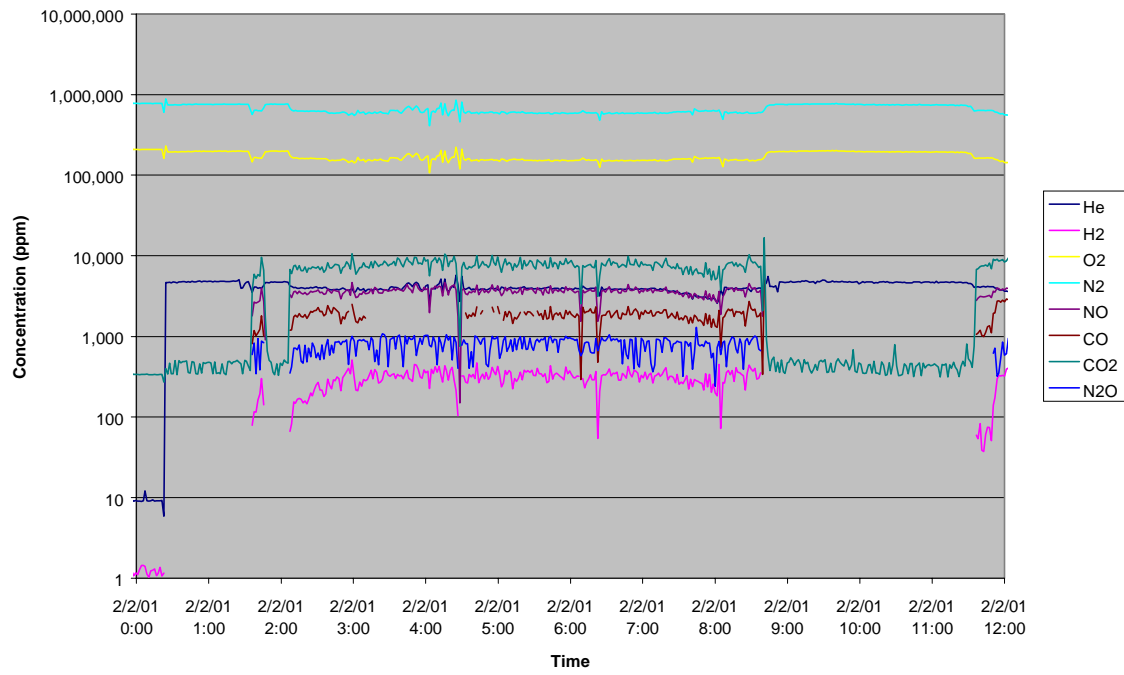


### Gas Chromatographic Off-Gas Analysis

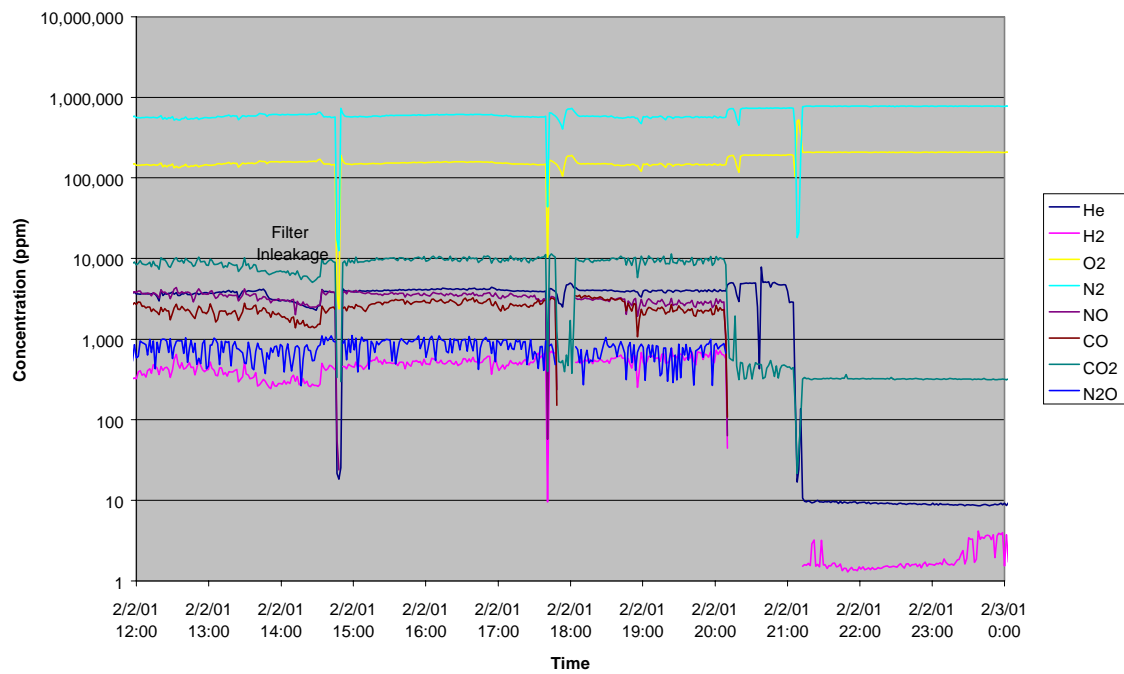


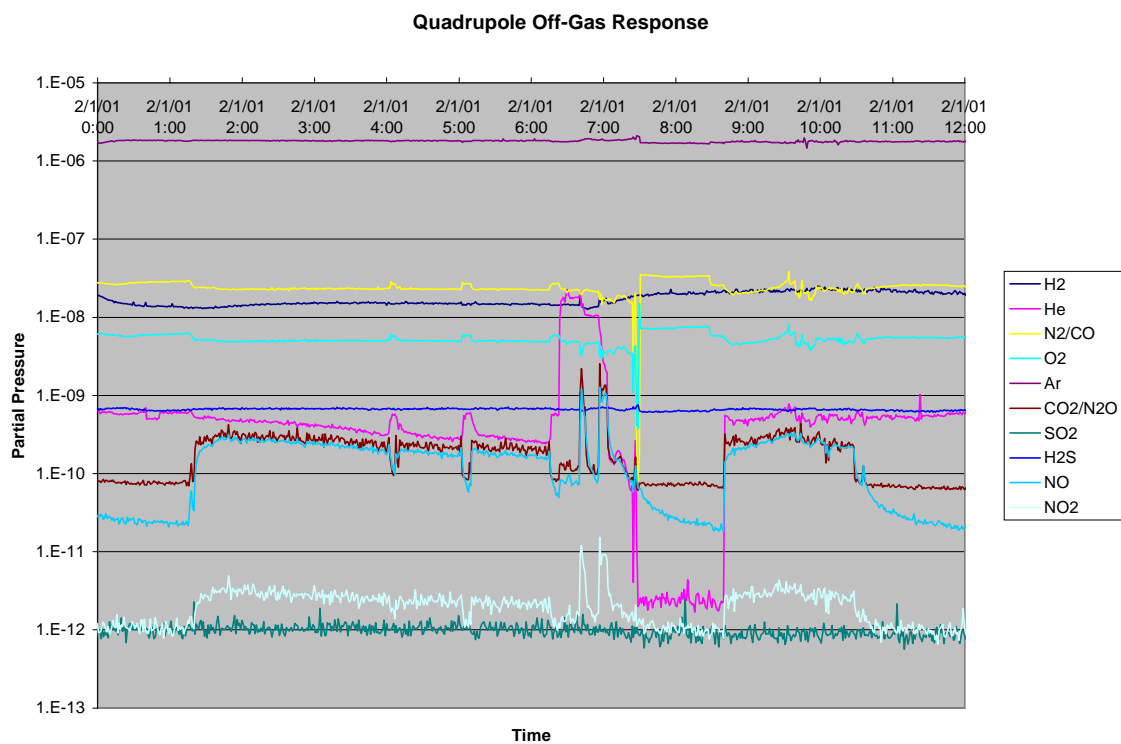
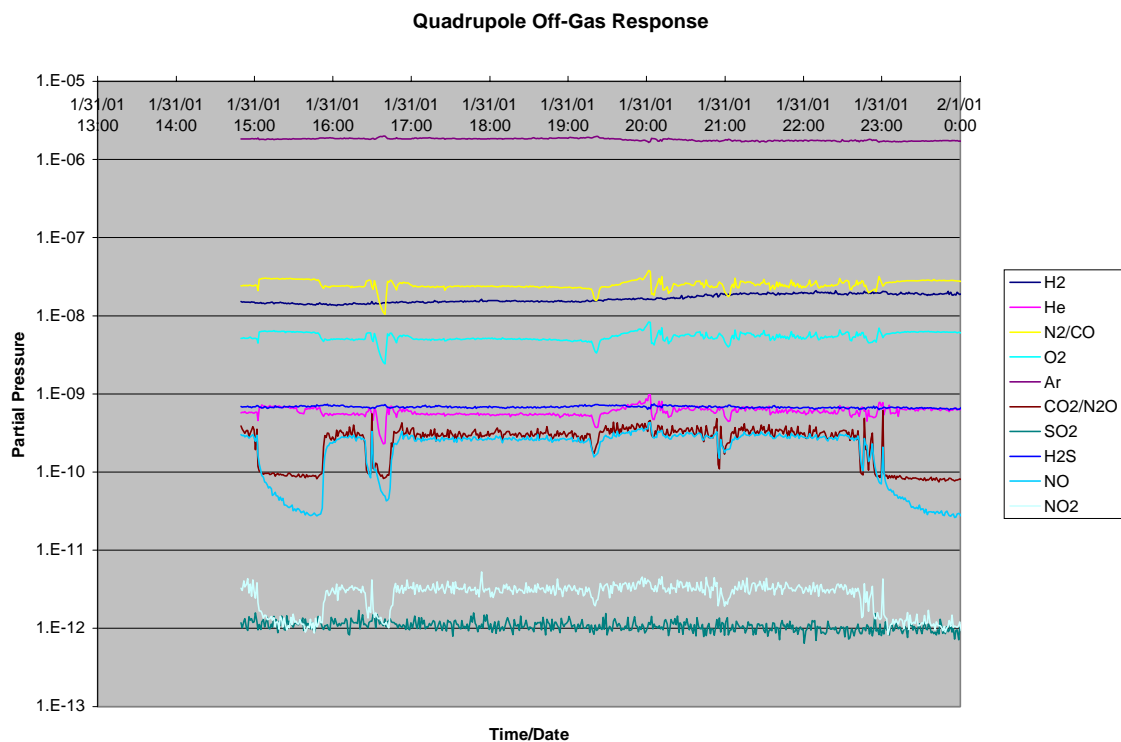


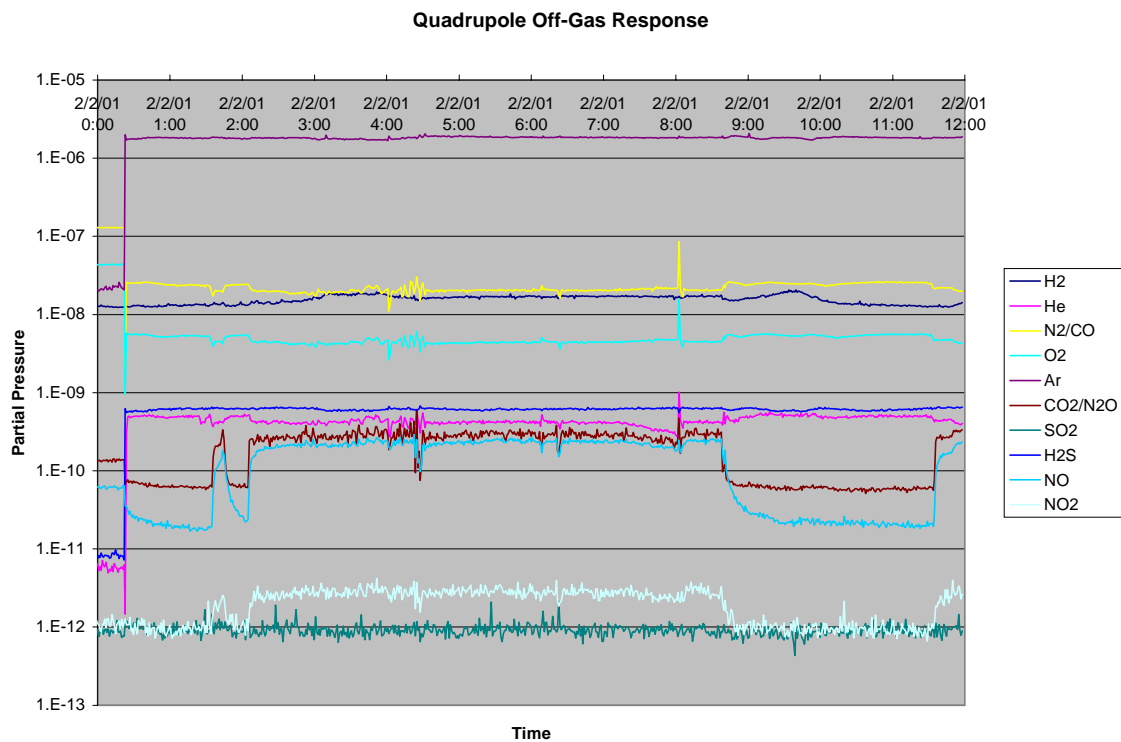
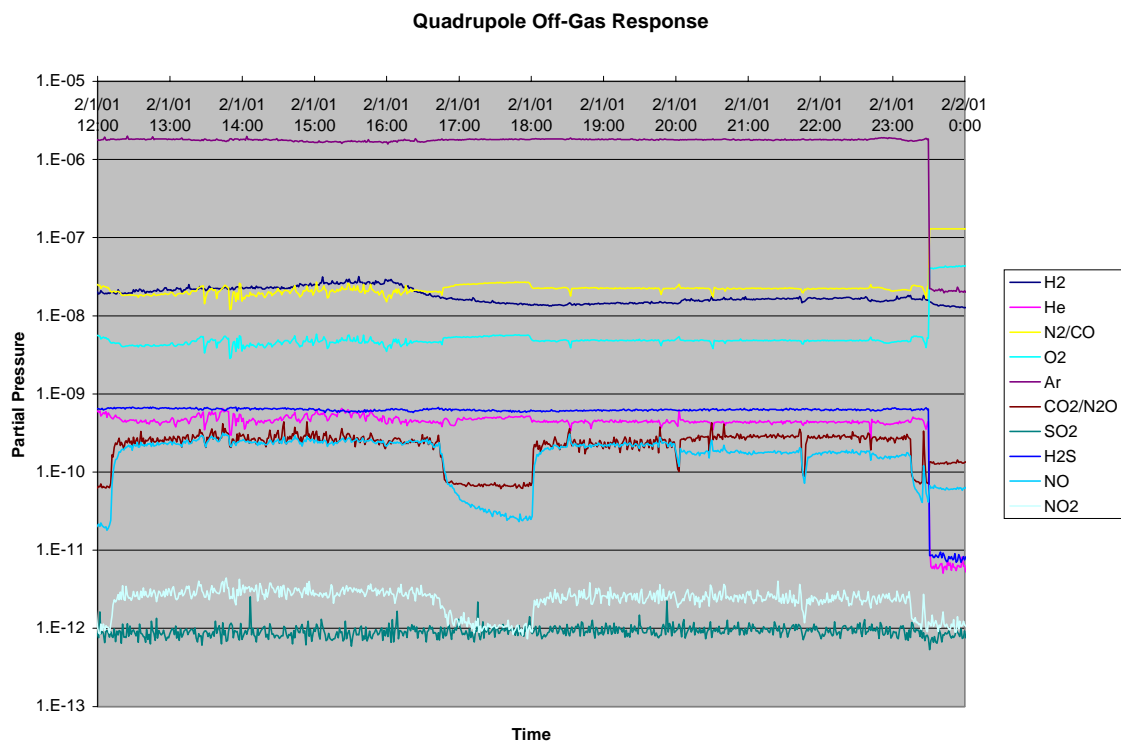
### Gas Chromatographic Off-Gas Analysis



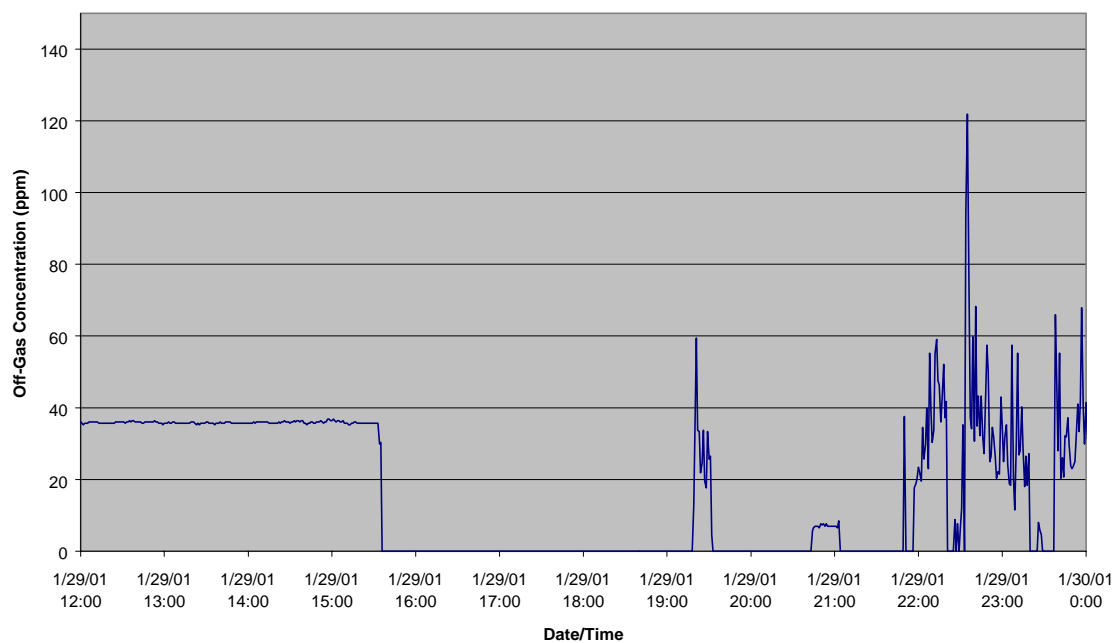
### Gas Chromatographic Off-Gas Analysis



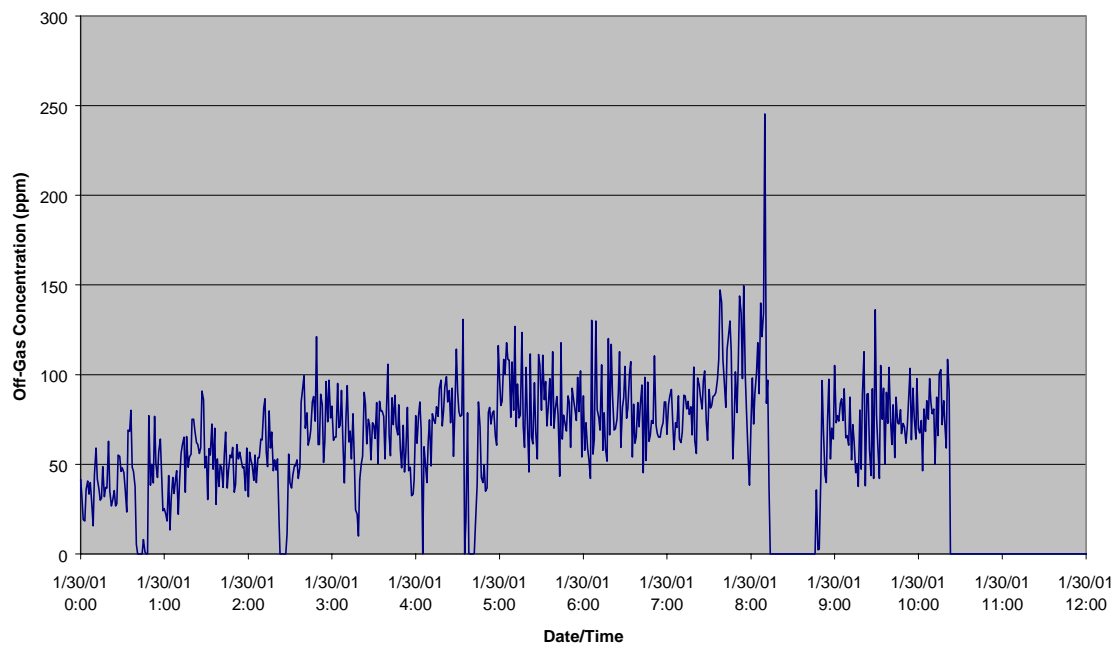




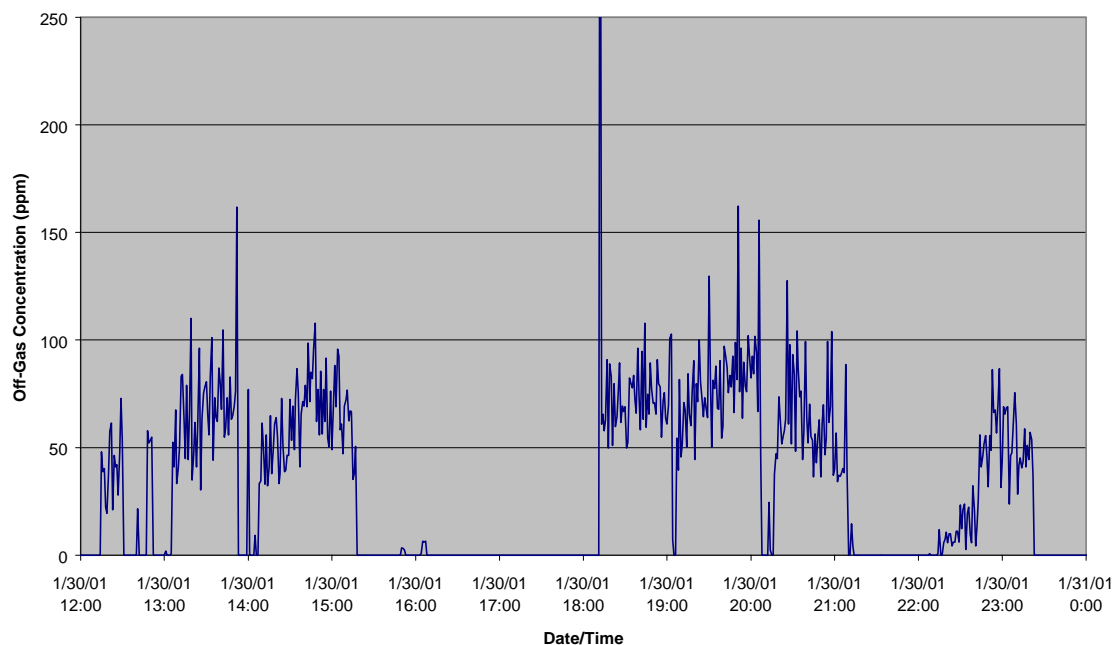
**Total Hydrocarbon Analysis**  
**Waste Sugar: 135 g/l**



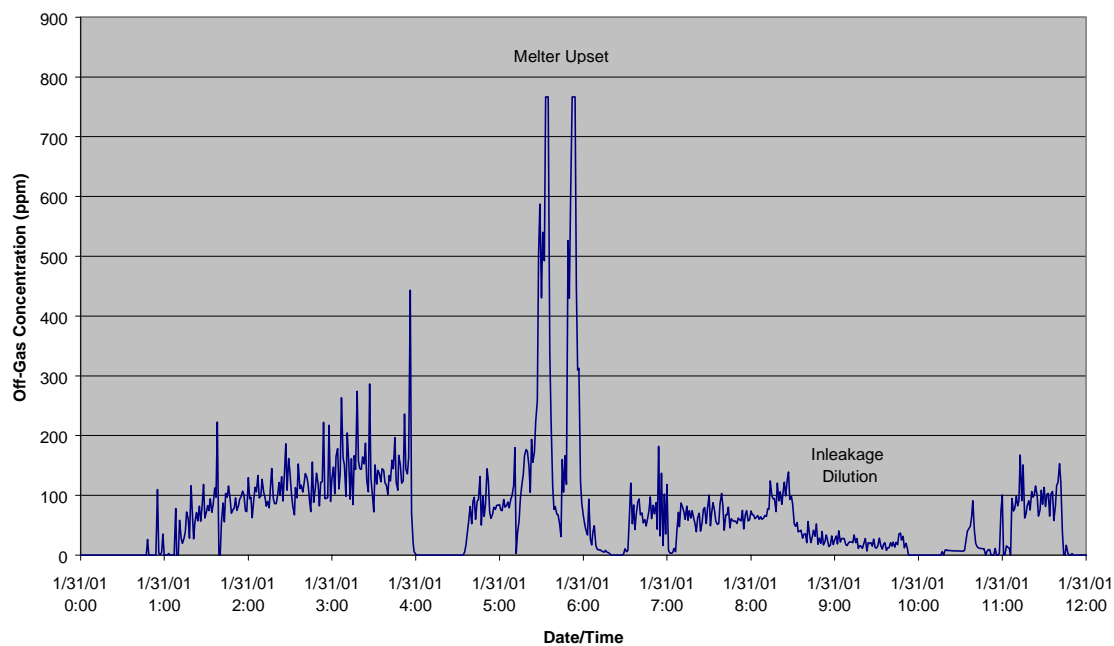
**Total Hydrocarbon Analysis**  
**Waste Sugar: 135 g/l**



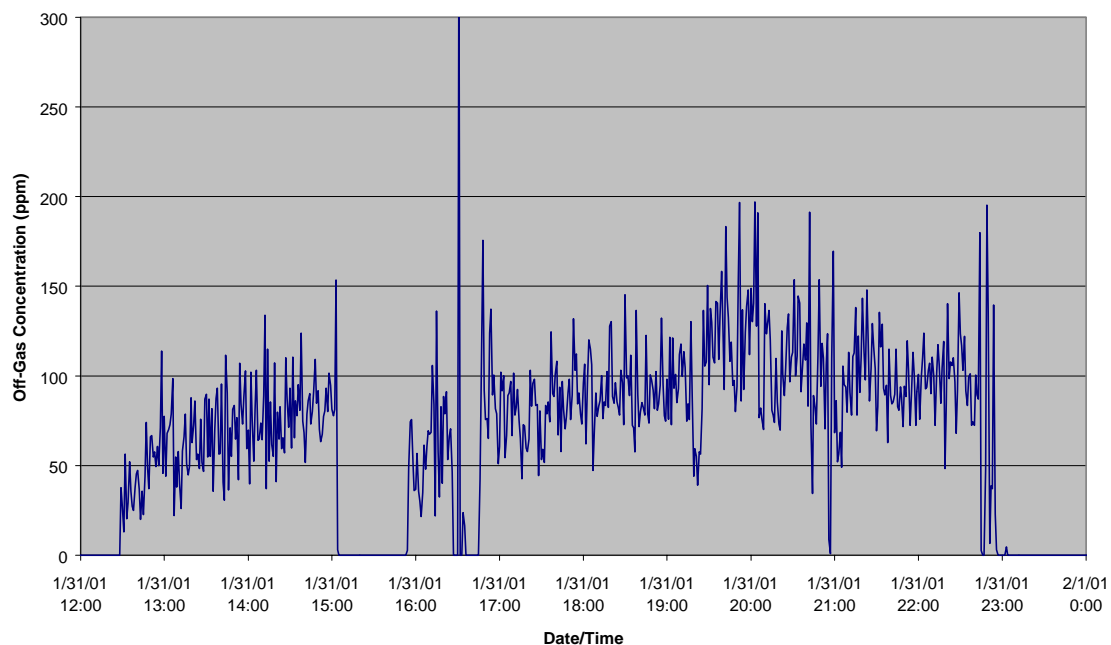
**Total Hydrocarbon Analysis**  
**Waste Sugar: 135 g/l**



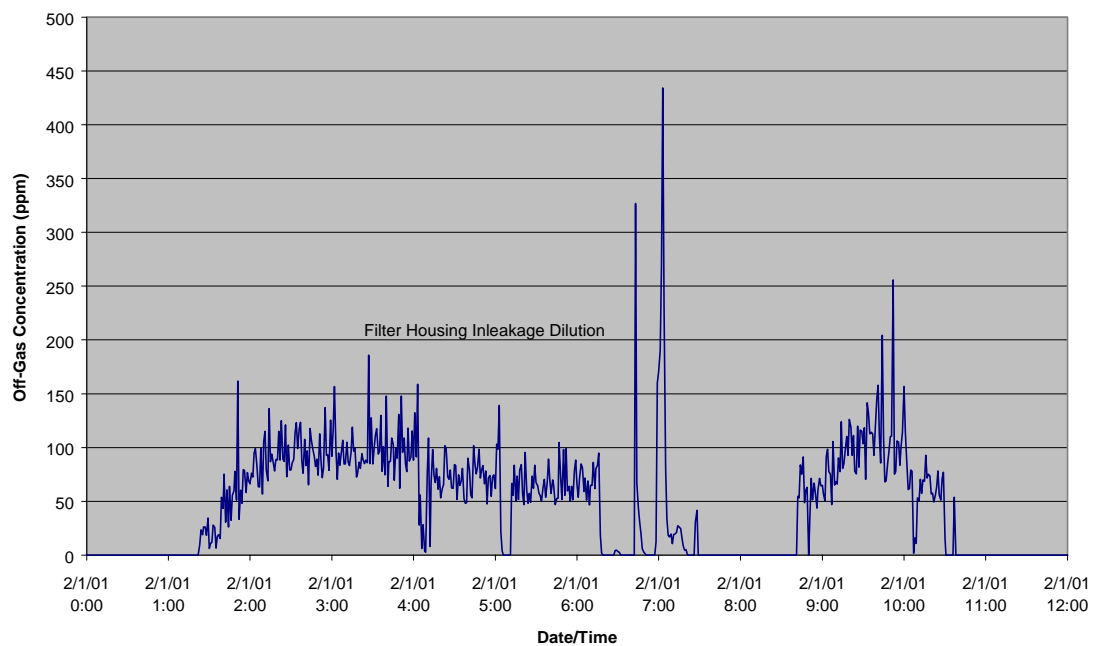
**Total Hydrocarbon Analysis**  
**Waste Sugar: 135 g/l**



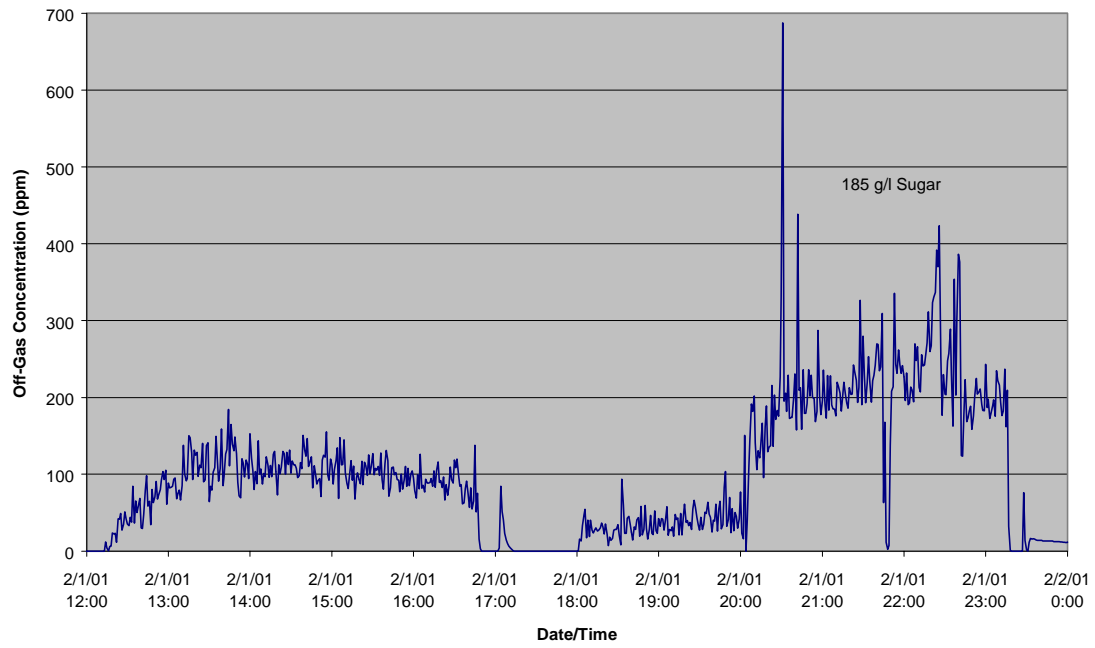
**Total Hydrocarbon Analysis**  
**Waste Sugar: 135 g/l**



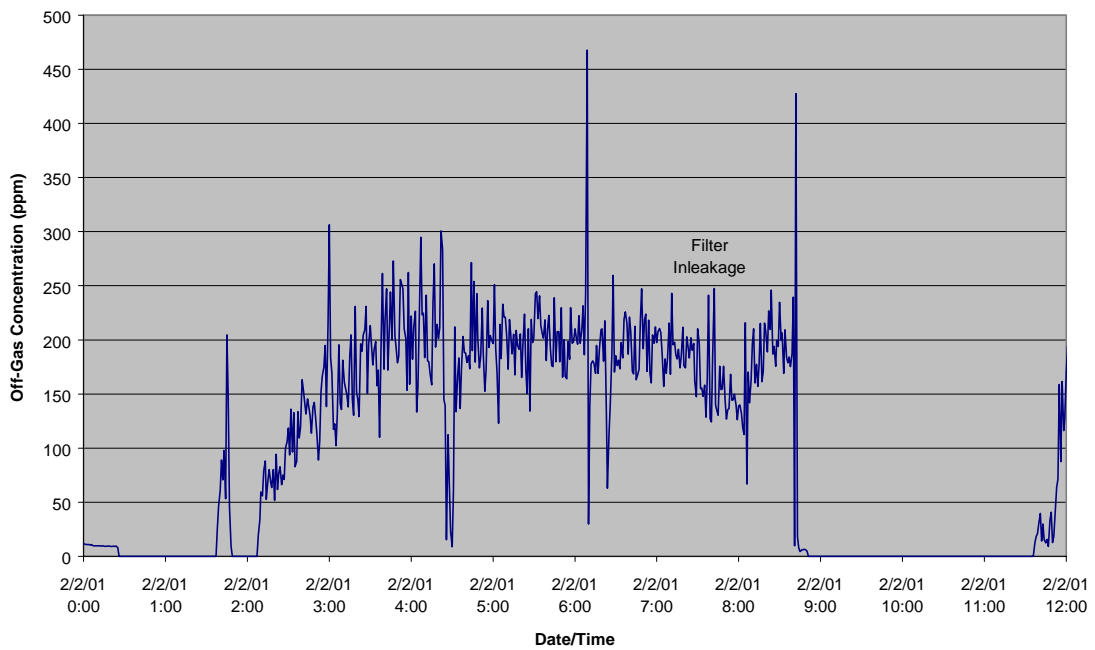
**Total Hydrocarbon Analysis**  
**Waste Sugar: 135g/l**



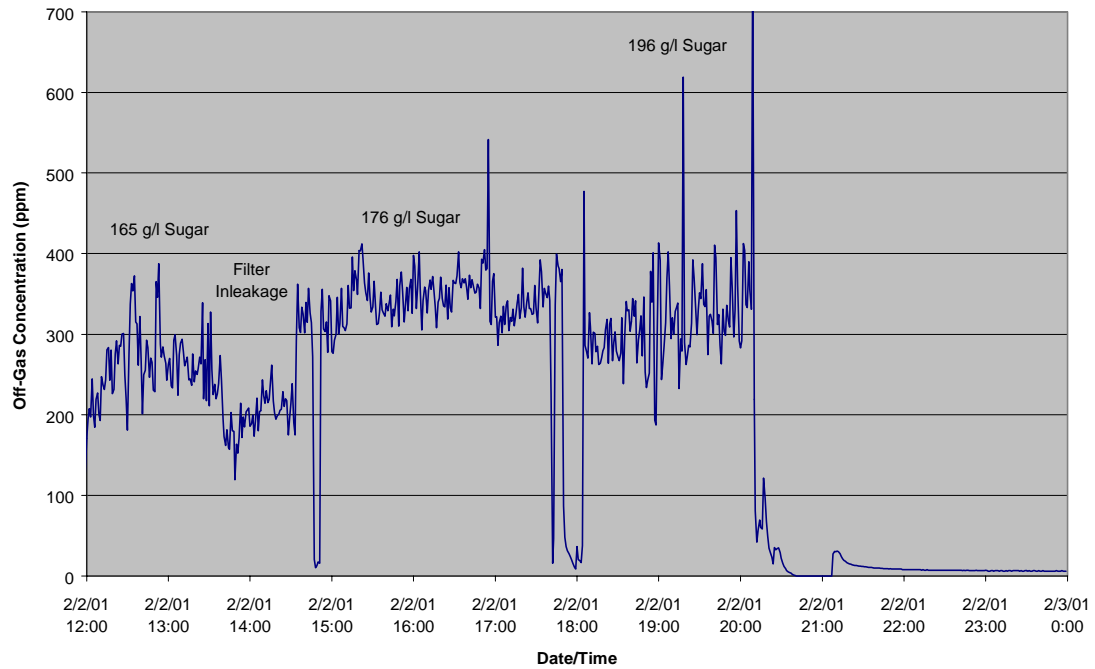
**Total Hydrocarbon Analysis**  
**Waste Sugar 135 g/l**



**Total Hydrocarbon Analysis**  
**Waste Sugar: 168 g/l**



### Total Hydrocarbon Analysis





## **Appendix H**

### **Photographs of the Off-Gas Line Deposits**

## Appendix H: Photographs of the Film Cooler, the Off-Gas Jumper, and the EVS Inlet



Operational and Spare Film Coolers (left to right). Inlet in the Foreground



Inlet View Of RSM 1.5” IPS Film Cooler



2" IPS Film Cooler Outlet Adapter



Horizontal 2" IPS Off-Gas Pipe Run Between The Film Cooler and EVS



Horizontal 2" IPS to 3"IPS EVS Inlet adapter



3" IPS EVS Inlet Port



Encrusted EVS Spray Nozzle



EVS Outlet Port