PNNL-14063

Test Summary Report Vitrification Demonstration of an Optimized Hanford C-106/AY-102 Waste-Glass Formulation

R. W. Goles
W. C. Buchmiller
C. R. Hymas
B. D. MacIsaac

November 2002

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

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor Battelle Memorial Institute, nor any of their employees, makes **any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights**. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof, or Battelle Memorial Institute. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

PACIFIC NORTHWEST NATIONAL LABORATORY
operated by
BATTELLE
for the
UNITED STATES DEPARTMENT OF ENERGY
under Contract DE-AC06-76RL01830

This document was printed on recycled paper.
(8/00)

PNNL-14063

Test Summary Report
Vitrification Demonstration of an Optimized Hanford C-106/AY-102 Waste-Glass Formulation

R. W. Goles
W. C. Buchmiller
C. R. Hymas
B. D. MacIsaac

November 2002

Prepared for the U.S. Department of Energy
under Contract DE-AC06-76RL0-1830

Pacific Northwest National Laboratory
Richland, Washington 99352

Abstract

In order to further the goal of optimizing Hanford's HLW borosilicate flowsheet, a glass-formulation effort was launched to develop an advanced high-capacity waste form exhibiting acceptable leach and crystal-formation characteristics. A simulated C-106/AY-102 waste envelope inclusive of LAW pretreatment products was chosen as the subject of these nonradioactive optimization efforts. To evaluate this optimized borosilicate waste formulation under continuous dynamic vitrification conditions, a research-scale Joule-heated ceramic melter was used to demonstrate the advanced waste form's flowsheet. The main objectives of this melter test was to evaluate 1) the processing characteristics of the newly formulated C-106/AY-102 surrogate melter-feed stream, 2) the effectiveness of sucrose as a glass-oxidation-state modifier, and 3) the impact of this reductant upon processing rates.

Summary

In Response to a U.S. Department of Energy (DOE) Headquarters directive to conduct a technical review of alternatives for solidification of high-level waste (HLW) that could achieve major cost reductions with reasonable long-term risks,[(a)] the Tanks Focus Area (TFA) chartered an independent Review Team to evaluate cost incentives associated with modifications to: waste-form product requirements, waste-stream processing constraints, product glass composition, and the reference waste-vitrification technology itself.

One of the conclusions drawn from the Review Team evaluation of alternative waste forms was that the borosilicate waste form has performed predictably and is firmly entrenched in the nuclear waste remediation program. However, it was felt that the vitrification process using this waste form had not been optimized (Perez et al. 2001). To further the goal of optimizing Hanford's HLW borosilicate flowsheet, a primary focus of the Review Team's effort, a TFA-sponsored glass-formulation effort was launched to develop an advanced high-capacity waste form exhibiting acceptable leach and crystal-formation characteristics (Peeler et al. 2002). Specifically, a simulated C-106/AY-102 waste envelope inclusive of low-activity waste (LAW) pretreatment products was chosen as the subject of these nonradioactive optimization efforts.

To evaluate the processing, off-gas emission, and product glass characteristics of this optimized borosilicate waste formulation under continuous dynamic vitrification conditions, a research-scale, Joule-heated, ceramic melter was used to demonstrate the advanced waste form's flowsheet. Specifically, 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, DWPF basis) joule-heated melter that is capable of processing melter feed on a continuous basis. This capability is key for:

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

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

The experimental scope of this initial, 5-d, 120-h, C-106/AY-102 vitrification test was to:

- determine the feasibility of vitrifying surrogate C-106/AY-102, at 60 wt% waste loading, to produce a regulatory-acceptable borosilicate glass waste form
- evaluate the glass product with regard to composition, crystal growth and concentration, phase separation, chemical durability, and oxidation state

(a) TTP: Advanced High-Level Waste (HLW) Melter and Waste Products Review, 25 August 2000.

- characterize the composition and concentration of condensed-phase and selected gaseous melter-generated off-gas emissions and the composition of secondary off-gas system waste streams
- establish maximum process rates at a glass temperature of 1150°C and a 95% cold-cap coverage
- access whether the melt rate or processing conditions can be optimized by changing reductant and/or melter-operating conditions
- measure the volume-reduction factor for vitrified surrogate C-106/AY-102 waste stream
- quantify specific glass-production rates and waste-vitrification energy requirements.

During the 120 h of experimental testing, a validated C-106/AY-102, Envelope-D surrogate[(a)] was prepared, combined, at 60 wt% waste loading, with an optimized suite of glass formers, composed of B, Li and Si, and successfully vitrified in a Joule-heated melter. Beyond the processing characteristics of the optimized, baseline glass formulation, the impacts of a sugar-reductant additive at varying concentrations (2.5, 5, 7.5, and 9 g/L-waste) upon processing rates, gaseous emission characteristics, and glass oxidation state [Fe(II):Fe(III)] were also evaluated. The effects of a ~10% increase in melter-feed-water concentration upon melter performance was also assessed during melter testing.

The melting kinetics of the optimized CY-106/AY-102 feed formulation was found to be influenced by sugar-additive concentration. Although initial short-term processing of the baseline feed formulation, without the use of sugar additive, was quite successful, the glass formed was highly oxidized, and because of its relatively high Mn content, was quite susceptible to foaming. Without physical methods to mitigate the impact of foam-layer development, long-term processing of feeds without a supplemental reductant does not appear to be a viable option.

Less dynamic foaming occurred when a waste sugar concentration of 2.5 g/L was employed, but unstable processing conditions nevertheless resulted from a glass that remained too highly oxidized. Stable long-term melter processing conditions were achieved at all other sugar concentrations evaluated. At a sugar waste loading of 5 g/L, the product glass' Fe(II):Fe(total) ratio was <5% while at 9 g/L, a value slightly less than the limiting condition of 30% was measured

Although stable melting conditions resulted for waste sugar loadings ≥5 g/L, cold-cap characteristics appeared to be deleteriously affected by the sugar additive. The addition of sugar at all levels seemed to exacerbate feed coning beneath melter's feed nozzle. The collapse of dried feed matter that accumulated within the cone produced temporary processing instabilities by creating 100% cold-cap coverage conditions at all sugar loadings used. Water dilution of the incoming feed stream was used somewhat successfully to spread the incoming feed material over the entire glass pool; however, reduced glass production rates resulted from the power losses required to dry the diluted feed.

Melter-glass production rates varied from 8.3 to 4.3 lbs/h/ft^2 for the various feed batches processed. These values comfortably exceed the reference (cold-lid) Liquid Fed Ceramic Melter (LFCM) design production rate of 4 lbs/h/ft^2 that is often quoted and used for flowsheet and equipment sizing estimates

---

(a) WK Kot, H Gan, and IL Pegg. 2000. *Physical and Rheological Properties of Waste Simulants and Melter Feeds for RPP-WTP HLW Vitrification*, Vitreous State Laboratory, The Catholic State University, Washington, D.C.

(Perez et al. 2001). Indeed, this reference-normalized production rate is exceeded even when projections are based upon the overall average rate data (5.5 lbs/h/ft^2) 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 the surrogate C-106/AY-102 feeds. The average value derived for all batches processed, 3.9 kW•h/kg, agrees well with typical energy requirements for slurry-fed, Joule-heated ceramic melters: 2 to 4 kW•h/kg of glass produced (Perez et al. 2001).

Representative glass samples generated throughout the duration of RSM testing were subjected to standard durability tests. Specifically, glass samples were subjected to both product consistency test (PCT) (ASTM 1997) and toxicity characteristic leach procedure (TCLP 1992) leach-testing protocols. The PCT results suggest that all C-106/AY-102 glasses were more durable than the standard environmental assessment glass to which they were compared. Although marginal Ag results were obtained, corresponding TCLP tests indicate that all glasses produced, even the more highly reduced ones, conform to all existing Resource Conservation and Recovery Act (RCRA) land-disposal limits (40 CFR 268).

Pour-stream glass samples taken throughout the melter testing campaign were examined for the presence of secondary phases. The results obtained indicated that spinel crystals were present at an average concentration of 0.5 wt% (~0.25 vol%), which was significantly lower than a comparable, pre-test, qualifying crucible test result (1.8 wt%) obtained at 1150°C. In addition, post-test core drilling of the solidified melter glass pool allowed spinel crystal uniformity throughout the melt volume to be accessed. No significant stratification of spinel could be discerned from the somewhat noisy and scattered data obtained.

For the surrogate C-106/AY-102 melter feed used during RSM testing, CO_2 and NO_x (specifically NO) were the major non-condensable (~ 25°C) gases produced by the vitrification process. Feed nitrates were found to be efficiently reduced to N_2 under all waste sugar loadings as evidenced by significantly reduced NO emission rates. Of the process-generated combustible gases of concern, CO was undetectable (<250 ppm) at all reductant concentrations, and the maximum concentration of H_2 recorded was 0.02%. These concentrations are well below the lower flammability limits of these combustible gases, 4.65% for H_2 and 15.5% for CO and, consequently pose no reasonable off-gas system hazard under any conceivable processing conditions..

Condensed-phase effluents were also monitored during C-106/AY-102 melter testing. The melter's aerosol mass decontamination factors (DFs), as measured by isokinetic filter catches, were determined while processing feed containing a waste-sugar concentration of 7.5 g/L. These melter aerosol mass DFs ranged from 500 to 1200. Cold-cap accumulation (coning) during off-gas sampling is likely responsible for the range of the observed values. The overall mass DF as determined from secondary waste stream compositions is 240.

Melter partitioning for individual feed components was also derived from the off-gas sampling and secondary waste-stream data. With the exception of boron, sulfur, and the halogens, essentially all feed constituents (excluding C, N, H_2O, etc.) were found to be primarily in a condensed state downstream of the film cooler. Overall, the element-specific DFs recorded during the current test are reasonably close to

general expectations and are generally consistent with previous melter-testing results. Furthermore, both off-gas sampling and secondary waste-stream-derived DF values exhibited the same relative trends for related groups of elements and were of comparable magnitudes for corresponding waste constituents. Indeed, the very reasonable mass closure demonstrated for most of the feed constituents for which complete analytical data exist suggests that the current melter test has successfully characterized the vitrification flowsheet of the optimized, 60 wt% C-106/AY-102 glass formulation.

During the July 2002 melter-flowsheet evaluation studies, 100 L (26 gal) of 60 wt% C-106/AY-102 simulated melter feed (~90 L-waste), having a total mass of 142 kg, was processed by the RSM, producing 20 L (5.3 gal) of glass having a total mass of 51 kg. These results suggest that an overall waste-volume-reduction factor of 4.6 was achieved during the RSM processing of the optimized C-106/AY-102 melter-feed formulation.

References

40 CFR 268. 2000. U.S. Environmental Protection Agency, "Land Disposal Restrictions." *U.S. Code of Federal Regulations*, Washington D.C.

American Society for Testing and Materials (ASTM). 1997. *Standard Test Methods for Determining Chemical Durability of Nuclear, Hazardous, and Mixed Waste Glasses: The Product Consistency Test (PCT),* ASTM C1285-97, West Conshohoken, PA.

Peeler DK, TB Edwards, CC Herman, RJ Workman, IA Reamer, JD Vienna, JV Crum, DE Smith, and DS Kim. 2002. *Development of High Waste Loading Glasses for Advanced Melter Technologies*, WSRC-TR-2002-00426, Westinghouse Savannah River Company, Aiken, SC.

Perez JM, DF Bickford, DE Day, DS Kim, SL Lambert, SL Marra, DK Peeler, DM Strachan, MB Triplett, JD Vienna, and RS Wittman. 2001. High-Level Waste Melter Study Report, PNNL-13582, Pacific Northwest National Laboratory, Richland, WA.

Toxicity Characteristic Leaching Procedure (TCLP). 1992. *Test Methods for Evaluating Solid Waste, Volume 1C: Laboratory Manual Physical/Chemical Methods,* SW-846, Method 1311, Rev. 2, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington D.C.

Acronyms / Symbols

APEL	Applied Process Engineering Laboratory
DF	decontamination factor
DOE	U.S. Department of Energy
EA	environmental assessment
EPA	U.S. Environmental Protection Agency
EVS	ejector venturi scrubber
HEME	high-efficiency mist eliminator
HEPA	high-efficiency particulate air (filter)
HLW	high-level waste
LAW	low-activity waste
LFCM	Liquid Fed Ceramic Melter
LOD	loss on drying
LOI	loss on ignition
MOG	melter off-gas
PCT	Product Consistency Test
PLC	Programmable Logic Controller
PNNL	Pacific Northwest National Laboratory
POG	process off-gas
RCRA	Resource Conservation and Recovery Act
RSM	Research-Scale Melter
SCR	silicon-controlled rectifier
SRS	Savannah River Site
TCLP	toxicity characteristic leach procedure
TFA	Tanks Focus Area
UDS	undissolved solids
UTS	Universal Test Standard
----	not measured / not detected / not available / not applicable

Acknowledgments

The authors would like to acknowledge the following people:

- PNNL staff who performed laboratory work and RSM shift activities
- Wayne Cosby for editorial assistance
- S. K. Sundaram for Technical Review
- E. William Holtzscheiter for management and guidance.

This study was funded by the U.S. Department of Energy's (DOE's) Office of Science and Technology, through the Tanks Focus Area, and was performed and documented by Pacific Northwest National Laboratory (operated for DOE by Battelle under Contract DE-AC06-76RL01830).

Contents

Abstract iii

Summary v

Acronyms ix

Acknowledgments xi

1.0 Introduction 1.1

2.0 Test Objectives 2.1

3.0 RSM System Description 3.1

3.1 Melter 3.1

3.2 Feed System 3.5

3.3 Off-Gas Processing System 3.6

3.4 Off-Gas Sampling System 3.6

3.5 Data Acquisition and Process Control System 3.7

4.0 CY106/AY102 Simulant, Melter Feed, and Product Glass 4.1

4.1 CY106/AY102 Surrogate 4.1

4.2 Target Glass Composition 4.6

4.3 Glass Former and Chemical Additives 4.6

4.4 Melter Feed Characteristics 4.7

4.5 Product Glass Characteristics 4.8
4.5.1 Oxidation State Results 4.9
4.5.2 Compositional Data 4.10
4.5.3 TCLP Results 4.14
4.5.4 PCT Results 4.14
4.5.5 Spinel Secondary Phase 4.15

5.0 Discussion of Results 5.1

5.1 Processing Observations and Parameters 5.1

5.2 Operating Parameters 5.5
5.2.1 Process Temperatures 5.5

5.2.2 Process Pressures 5.5
5.2.3 Melter Electrical Data 5.8
5.2.4 EVS Condensate Tank, Film Cooler Injection Air 5.10

6.0 Melter Off-Gas Emission Characterization 6.1

6.1 Gaseous Effluent 6.1

6.2 Condensed Phase Effluents 6.3
6.2.1 Aerosol Mass DFs 6.4
6.2.2 Aerosol Elemental DFs 6.5
6.2.3 Volatile Partitioning and Total Elemental DFs 6.7
6.2.4 Process-Waste-Stream Composition 6.9

7.0 Byproducts, Residuals, Mass Balance, and Volume Reduction 7.1

7.1 Off-Gas Line Deposits 7.1

7.2 Process Mass Balance 7.2

7.3 Process Statistics 7.4

7.4 Process Summary 7.4

8.0 References 8.1

Appendix A: Test Plan and Test Instructions for Vitrification Demonstration Tests of C-106/AY-102 Waste Simulant A.1

Appendix B: Test Instructions for Preparing C-106/AY-102 Waste Simulant B.1

Appendix C: RSM Feed Batching and Manual Data Sheets C.1

Appendix D: Colorimetric Procedure for Determining Fe(II) to Total Iron Ratio D.1

Appendix E: Variable Process Data Collected During the C-106/AY-102 Flowsheet Evaluations E.1

Figures

3.1. Photograph of RSM Demonstration Unit 3.2

3.2. Research-Scale Melter Test Apparatus 3.3

3.3. Schematic View of the Research-Scale Melter 3.4

3.4. Schematic Arrangement of Off-Gas Sampling System Components 3.7

4.1. Historical Sugar Concentration and Glass-Oxidation-State Values 4.11

4.2. Spinel Concentrations Within the RSM's Frozen Glass Volume 4.16

5.1. Process History of RSM Feed Batches and Average Overall Processing Rate 5.2

5.2. C-106/AY-102 Glass Accumulation Data 5.4

5.3. Melter Vacuum and Off-Gas Pressure Drop Data 5.7

5.4. RSM Electrode Operating Characteristics 5.8

5.5. EVS Condensate Tank, Film-Cooler Operating History 5.12

6.1. Temporal Behavior of Major Process Effluent Gases 6.4

7.1. Melter Off-gas Jumper Configuration and Sample Site Locations 7.2

Tables

3.1. RSM Dimensions and Operational Features 3.5

3.2. RSM's Effluent Gas Analyzers 3.6

4.1. Composition Summary for C-106/AY-102 Waste, LAW Pretreatment Products and C-106/AY-102 Simulant 4.2

4.2. Composition and Preparation Recipe for C-106/AY-102 Simulant 4.3

4.3. RSM C-106/AY-102 Waste Surrogate Composition 4.4

4.4. Comparison of Analytical and Manufacturer Assay Reports 4.5

4.5. Physical Characteristics of C-106/AY-102 Surrogate Waste 4.5

4.6. Target Glass Composition 4.6

4.7. Comparative CY-106/AY-102 Glass-Former Additives 4.7

4.8. Melter Feed Glass-Former Additives and Equivalent Frit Composition 4.7

4.9. Physical Properties of Surrogate C-106/AY-102 Melter Feeds 4.8

4.10. Melter Feed Batch Composition 4.9

4.11. C-106/AY-102 Sugar Loadings and Glass-Oxidation States 4.10

4.12. Oxide Composition of Melter C-106/AY-102 Glass Samples 4.12

4.13. Independent (SRS) Oxide Composition Data for Melter C-106/AY-102 Glass Samples 4.13

4.14. TCLP Leachate Concentrations from C-106/AY-102 Glasses 4.14

4.15. PCT Leachate Results from C-106/AY-102 Glasses 4.15

4.16. Spinel Content of Laboratory and Melter Glasses 4.16

5.1. C-106/AY-102 Feed Processing Rates and Derived Glass Production Rates 5.3

5.2. Experimental C-106/AY-102 Glass Production Rate Data 5.4

5.3. RSM's Operating Temperature Characteristics 5.6

5.4. Melter Kiln, Pour Spout, and Canister Oven Temperatures 5.6

5.5. RSM Electrode Circuit Operating Characteristics 5.9

5.6. Specific Process Energy Requirements For C-106/AY-102 Feeds 5.9

5.7. Operational Characteristics of Melter Kiln and Overflow Heaters 5.11

6.1. Unquenched Melter Off-Gas Composition 6.2

6.2. Gross Melter Aerosol Emission Characteristics 6.5

6.3. Oxide Composition of Melter Generated Aerosols and Melter Feed 6.6

6.4. Elemental Melter DFs Associated With Aerosol Emissions 6.7

6.5. Off-Gas Sampler Condensate Solution Composition (ppm) 6.8

6.6. Volatile Melter DFs for Boron, Halogen, and Sulfur Feed Constituents 6.8

6.7. Total Individual Elemental Melter DF Values 6.9

6.8. EVS Condensate Soluble Effluent Composition 6.11

6.9. HEME Waste Stream Composition For Soluble Effluent 6.12

6.10. Composition of EVS Undissolved Solids and Off-Gas Pipe Deposits 6.13

6.11. Off-Gas Sampler and Waste-Stream Total Melter DF Values 6.13

6.12. Secondary Waste Stream Physical Properties 6.14

7.1. Distribution of Melter Off-Gas Line Deposits 7.1

7.2. Stream-Dependent %-Partitioning of C-106/AY-102 Melter Feed Constituents 7.3

1.0 Introduction

In Response to a U.S. Department of Energy (DOE) Headquarters directive to conduct a technical review of alternatives for solidification of high-level waste (HLW) that could achieve major cost reductions with reasonable long-term risks,[(a)] the Tanks Focus Area (TFA) chartered an independent Review Team that collected and analyzed data, performed specific analyses, and recommended a research and development program, as warranted, for future waste form and melter advancements.

The focus of the Review Team effort was on HLW at the Hanford site in Richland, Washington, and on cost incentives associated with modifications to: waste form product requirements, waste stream processing constraints, product glass composition and the reference waste vitrification technology itself.

Based upon the results of the study, the review team addressed whether alternate glasses or glass-ceramic compositions, including borosilicate glass, could accommodate DOE HLW with greater efficiency, cost savings, and/or lower risks. One of the conclusions drawn from the Review Team evaluation of this subject area was that the borosilicate waste form has performed predictably and is firmly entrenched in the nuclear-waste remediation program. However, it was felt that the vitrification process using this waste form has not been optimized (Perez et al. 2001).

To further the goal of optimizing Hanford's HLW borosilicate flowsheet, a glass-formulation effort was launched to develop an advanced high-capacity waste form exhibiting acceptable leach and crystal-formation characteristics. A simulated C-106/AY-102 waste envelope inclusive of low-activity waste (LAW) pretreatment products was chosen as the subject of these nonradioactive optimization efforts (Peeler et al. 2002).

To evaluate the processing, off-gas emission, and product glass characteristics of this advanced borosilicate waste formulation under continuous dynamic vitrification conditions, a research-scale Joule-heated ceramic melter was used to characterize the advanced waste form's flowsheet.

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 C-106/AY-102 surrogate melter-feed stream, 2) the effectiveness of sucrose as a glass-oxidation-state modifier, and 3) the impact of this reductant upon processing rates. This summary report documents the results obtained from melter flowsheet tests conducted to evaluate the efficacy of this new C-106/AY-102 melter-feed formulation.

(a) TTP: Advanced High-Level Waste (HLW) Melter and Waste Products Review, 25 August 2000.

2.0 Test Objectives

The primary objective of the current RSM test is to characterize the melter-process flowsheet based upon the Hanford's C-106/AY-102 waste Envelope D and an optimized target glass composition. The targeted vitreous-product composition was established from a series of laboratory crucible tests involving C-106/AY-102 surrogate material and suitable glass-forming additives that were chosen to maximize product glass waste loading (Peeler et al. 2002). Although these crucible tests suggested that the optimized glass formulation with waste loading as high as 60 wt% could be achieved while maintaining a durable vitreous waste product, it was recognized that continuous feeding and dynamic processing conditions under Joule-heating conditions were necessary to validate this finding. 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 flowsheet assessments of processing conditions and glass-product quality, the experimental objectives of the current RSM C-106/AY-102 flowsheet test was to:

- determine the feasibility of vitrifying surrogate C-106/AY-102 at 60 wt% waste loading using an optimized glass-former composition to produce a regulatory acceptable borosilicate-glass waste form
- evaluate the glass product with regard to composition, crystal growth and concentration, phase separation, chemical durability, and oxidation state
- characterize the composition and concentration of condensed-phase and selected gaseous melter-generated off-gas emissions and the composition of secondary off-gas system waste streams
- establish a maximum process rate at a glass temperature of 1150°C and a 95% cold-cap coverage
- access whether the melt rate or processing conditions can be optimized by changing reductant and/or melter-operating conditions
- measure the volume-reduction factor for vitrified surrogate C-106/AY-102 waste stream
- quantify specific glass-production rates and waste vitrification energy requirements.

Testing objectives, operating conditions, system configuration, and sampling and data-recording requirements are documented in the RSM Test Plan that was prepared and approved before the start of testing. However, due to a last-minute glass formulation change that was necessitated by durability concerns associated with centerline-canister-cooling conditions, a single glass formulation at a single fixed waste loading (60 wt%) was evaluated rather than the two conditions (55 wt% and 60 wt%) indicated in the earlier documented *Test Plan and Test Instructions*. The *Test Plan and Test Instructions* developed for this melter flowsheet evaluation study are presented in Appendix A of this report.

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 current C-106/AY-102 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 × 0.64 cm thick [3 in. square × ¼ in. thick]), which 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

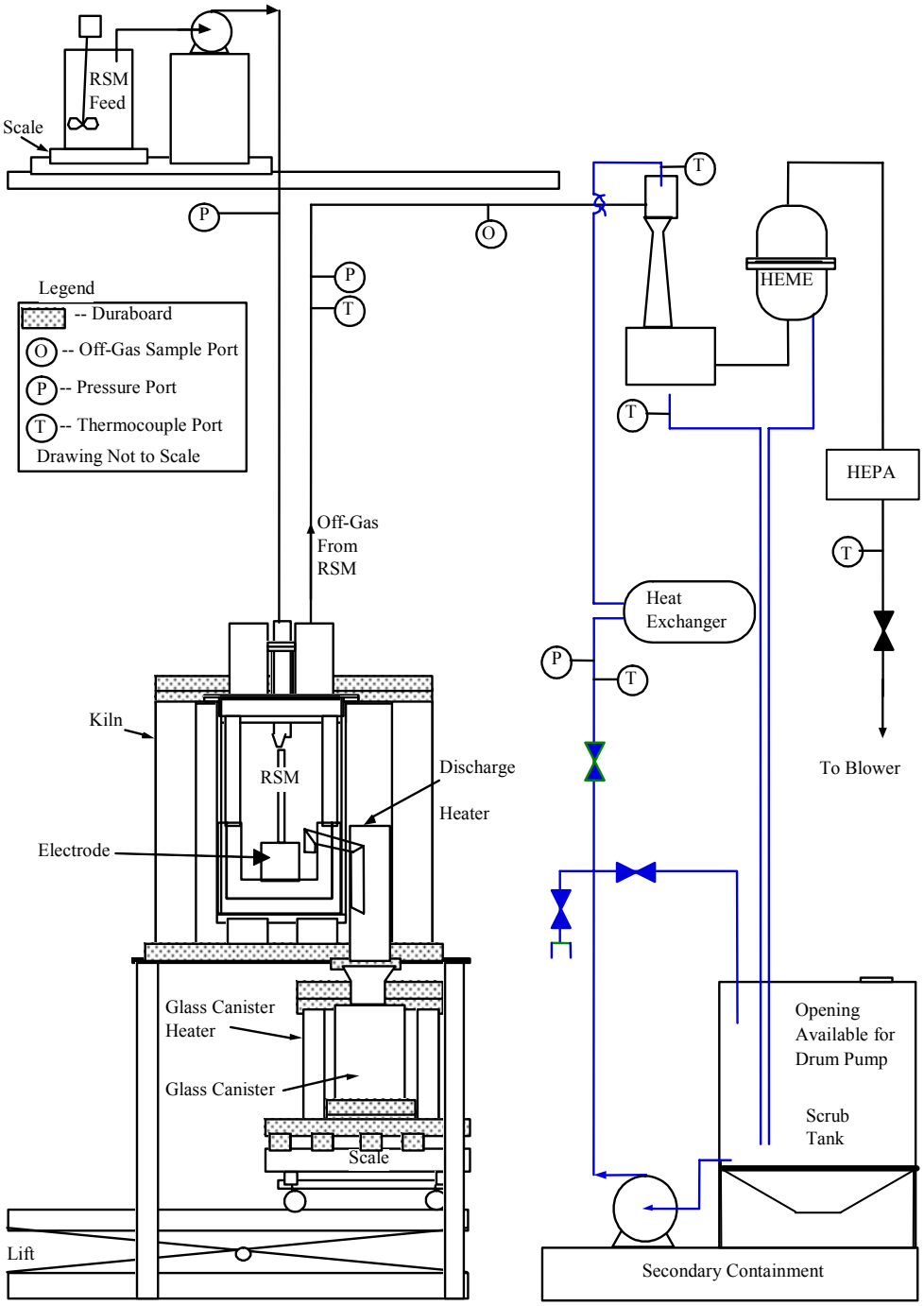


Figure 3.2. Research-Scale Melter Test Apparatus

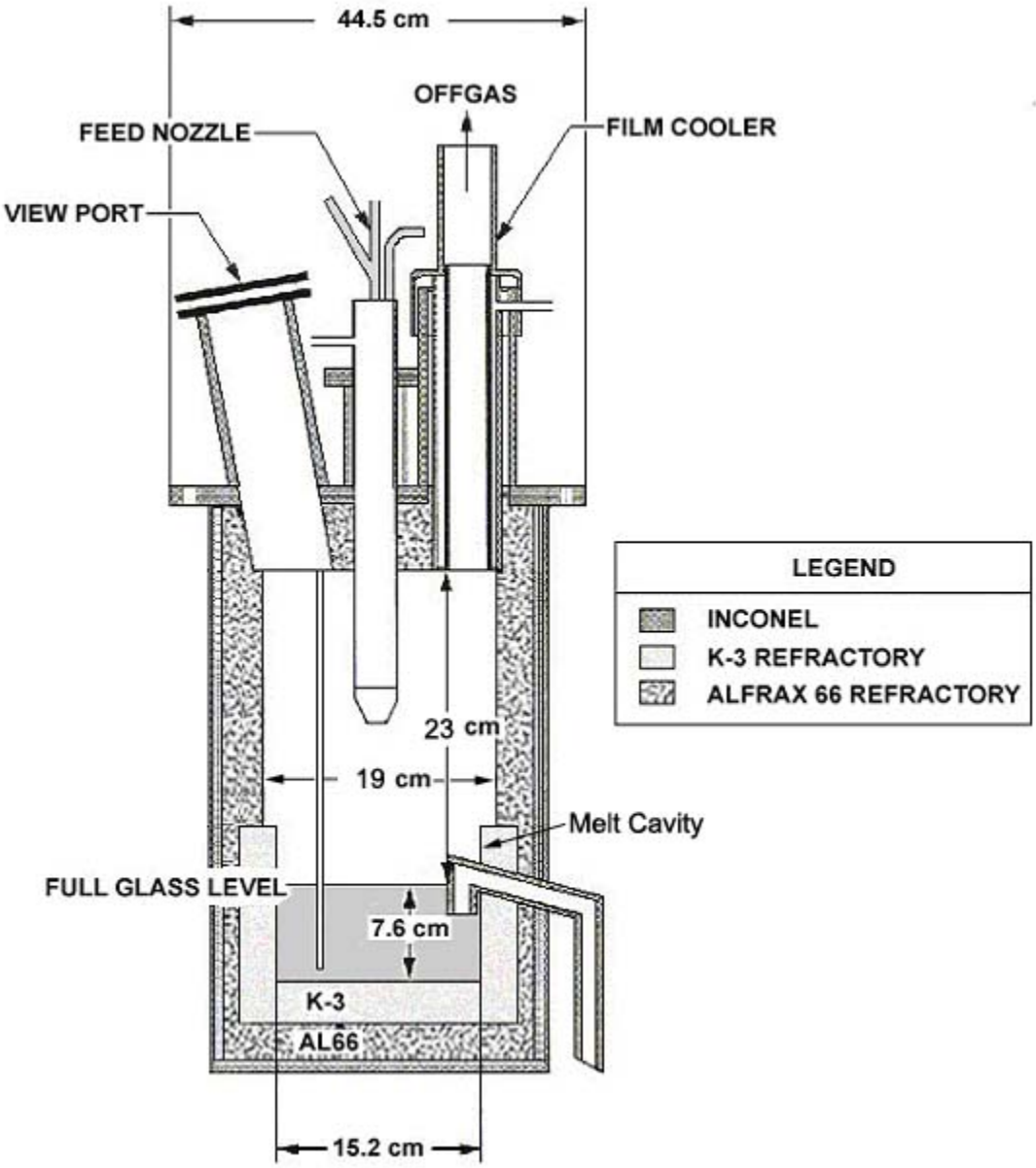


Figure 3.3. Schematic View of the Research-Scale Melter

Table 3.1. RSM Dimensions and Operational Features

Parameter	Value
Melt cavity diameter	15 cm
Melt cavity height	11 cm
Melt cavity volume	2.1 L
Glass-pool surface area	182 cm^2
Nominal glass depth	7.6 cm
Melter-glass-inventory volume	1.4 L
Nominal molten-glass mass	3.6 kg
Glass turnover rate @ nominal feed rate of 1.5 L/h of feed with 0.6 kg/L oxides	4.0 h
Maximum operating temperature	1,200°C
Nominal operating temperature	1,150°C
Electrode Dimensions	7.6 cm × 7.6 cm
Electrode Material	Inconel 690
Electrode melt-cavity bottom clearance	0 cm
Electrode current (average)	90 A
Electrode voltage (average)	25 V
Electrode current density (average/maximum)	1.6/2.0 A/cm^2

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 57 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 large ($^3/_4$-in. tube) peristaltic pump was used to recirculate agitated melter feed through a heat exchanger and provide a slip-stream extraction point for a second peristaltic pump that was used to directly feed the melter. The melter feed pump directed the feed 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 (using either a pressurized or nonpressurized water source) 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), high efficiency mist eliminator (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 pipe runs.

The EVS uses a high-pressure aqueous scrubbing liquor spray to contact the process exhaust stream to quench it and to remove steam, large-diameter aerosols, and some condensable and/or acid gases. A 97 L (~25 gal) charge of fresh water 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. A water-cooled heat exchanger located in the EVS's spray circuit was used to maintain nominal room-temperature scrubbing liquor conditions.

The HEME uses a deep, regenerable fibrous bed to remove both liquid aerosols generated by the high-pressure EVS spray and submicron condensed-phase aerosols that successfully penetrate the low-efficiency quench (EVS) scrubber. The demisted and relatively clean HEME exhaust is then heated to preclude condensation by the next stage of treatment, a certified HEPA filter that removes 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 (~80%) 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_2, He, N_2, CO, NO, O_2, CO_2, and N_2O
Nitrogen Oxide	NO and NO_2

Discrete sampling of process-generated aerosols and condensable/scrubbable effluents is normally 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. During the current test, however, only one scrubber vessel was used, and it was immersed in an ice bath to serve as the system's condenser. Consequently, only two sample components were extracted by this system - condensed matter, (aerosols) and condensable/acid-gas effluents (steam, boron, halogens etc.).

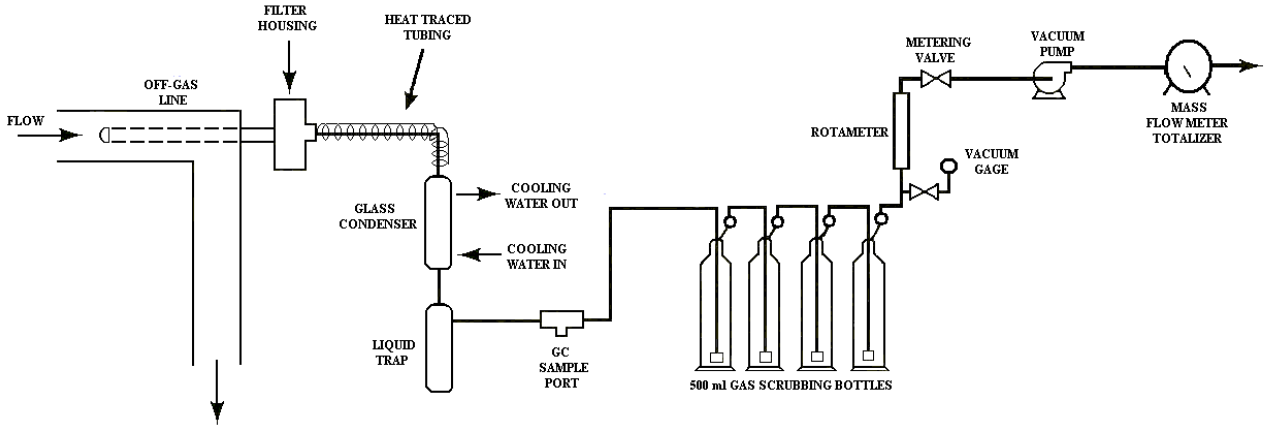


Figure 3.4. Schematic Arrangement of Off-Gas Sampling System Components

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 flowrates. 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 (0°C) by an ice bath condenser.

To establish isokinetic sampling conditions, the total off-gas flowrate has to be measured. During RSM testing, this was accomplished by injecting a helium tracer into the melter plenum at a fixed flowrate (2 L/m) and measuring its resultant off-gas concentration with the online gas chromatograph discussed above. These POG flowrate data were also of fundamental importance in establishing effluent 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 CY106/AY102 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 CY106/AY102 flowsheet, melter-feed material at 60 wt% waste loading was prepared in batches by mixing a preformulated, waste stock solution/slurry with appropriate quantities of glass-forming chemicals to which sucrose reductant was sometimes added as a glass-oxidation-state modifier. The following discussion provides detailed information concerning these waste and melter feed-stream additives.

4.1 CY106/AY102 Surrogate

Extensive simulant development efforts for the C-106/AY-102 waste envelope have been conducted in support of Hanford's Waste Treatment Plant.[(a)] The defined C-106/AY-102 waste composition, which includes low-activity waste (LAW) pretreatment product-stream additives, is summarized in Table 4.1 where the concentrations of selected trace components have been appropriately adjusted to accommodate analytical detection sensitivities. The chemicals employed to simulate the physical and chemical characteristics of actual C-106/AY-102 waste are detailed in Table 4.2.

Since it may be important in the future to compare the current flowsheet tests using an optimized glass formulation with baseline testing results, the current test used the same simulant and similar glass-former components prepared in an identical manner as in previous studies. The chemical forms of the waste and glass-former constituents defined by the previous simulant-development effort were also used during the current test.

Following the waste makeup procedure presented in Appendix B, 125 L (33 gal) of CY106/AY102 surrogate was prepared, and its resultant composition is compared to the defined CY106/AY102 target in Table 4.3. The large deviation of Mg and S from the target value is due to impurity concentrations in the 13 wt% $Fe(OH)_3$ slurry that is a major chemical constituent of the validated C-106/AY-102 surrogate formulation. Other large waste-compositional variances, e.g., Ba, Cd, K, La, Si, etc. are apparently due to errant manufacturer assay results that are demonstrated in Table 4.4. The variances of strontium and zirconium from their target values documented in Table 4.3 are due to analytical difficulties (Sect 4.5.2), as the waste-preparation documentation fully supported attainment of the targeted value. Other target variances are likely due to analytical limitations as many waste constituents were present at

(a) WK Kot, H Gan, and IL Pegg. 2000. *Physical and Rheological Properties of Waste Simulants and Melter Feeds for RPP-WTP HLW Vitrification*, Vitreous State Laboratory, The Catholic State University, Washington, D.C.

Table 4.1. Composition Summary for C-106/AY-102 Waste, LAW Pretreatment Products and C-106/AY-102 Simulant

	C-106/AY-102 Envelope D Waste	Analytical Adjustment to Envelope D (g/100g-Ox)	Pretreatment Products (wt% of Adjusted Envelope D Oxide)	C-106/AY-102 Simulant
Ag_2O	0.59 %	----	----	0.46 %
Al_2O_3	26.99 %	----	----	20.91 %
BaO	0.17 %	----	----	0.13 %
CaO	1.79 %	----	----	1.39 %
CdO	0.11 %	----	----	0.09 %
CeO_2	0.08 %	----	----	0.06 %
Cl	0.00 %	0.060	----	0.05 %
Cr_2O_3	0.41 %	----	----	0.32 %
Cs_2O	0.01 %	----	0.25 %	0.20 %
F	0.04 %	0.022	----	0.05 %
Fe_2O_3	29.37 %	----	----	22.76 %
K_2O	0.07 %	----	----	0.05 %
La_2O_3	0.28 %	----	----	0.22 %
MgO	0.55 %	----	----	0.43 %
MnO	1.34 %	----	9.75 %	8.61 %
Na_2O	20.35 %	----	0.60 %	16.23 %
NiO	0.39 %	----	----	0.30 %
P_2O_5	0.38 %	----	----	0.29 %
PbO	0.54 %	----	----	0.42 %
PdO	0.01 %	----	----	0.01 %
Rh_2O_3	0.01 %	----	----	0.01 %
RuO_2	0.03 %	----	----	0.02 %
SO_3	0.02 %	0.044	----	0.05 %
SiO_2	14.75 %	----	----	11.43 %
SrO	0.05 %	----	18.34 %	14.26 %
TiO_2	0.10 %	----	----	0.08 %
UO_2	1.30 %	----	----	1.01 %
ZrO_2	0.29 %	----	----	0.23 %
TOTAL	100 %	0.126	28.94 %	100 %
Volatiles (g/100 g oxides)				
CO_3	0.004	----	36.7	8.240
NO_2	0.003	----	----	0.002
NO_3	0.000	----	4.5	1.013
TOC	1.601	----	----	1.240

Table 4.2. Composition and Preparation Recipe for C-106/AY-102 Simulant

Oxide	C-106/AY-102 Simulant	Starting Materials	Amount in Simulant (kg)
Ag_2O	0.46 %	Ag_2O	0.11
Al_2O_3	21.13 %	$Al(OH)_3$	7.83
BaO	0.13 %	$Ba(OH)_2 \cdot 8H_2O$	0.06
CaO	1.40 %	$CaCO_3$	0.61
CdO	0.09 %	CdO	0.02
CeO_2	0.06 %	$Ce(OH)_4$	0.02
Cl	0.05 %	NaCl	0.02
Cr_2O_3	0.32 %	$Cr_2O_3 \cdot 1.5H_2O$	0.09
Cs_2O	0.20 %	CsOH (50% Solution)	0.10
F	0.05 %	NaF	0.03
Fe_2O_3	22.99 %	$Fe(OH)_3$ Slurry (13 %)	56.49
K_2O	0.05 %	KNO_3	0.03
La_2O_3	0.22 %	$La(OH)_3 \cdot 3H_2O$	0.08
Li_2O	----	----	----
MgO	0.43 %	$4MgCO_3 \cdot Mg(OH)_2 \cdot 5H_2O$	0.25
MnO	8.70 %	MnO_2	2.58
Na_2O	16.40 %	NaOH (50% Solution)	9.82
NiO	0.30 %	$NiOH_2$	0.09
P_2O_5	0.30 %	$FePO_4 \cdot xH_2O$	0.15
PbO	0.42 %	$(PbCO_3)_2 \cdot Pb(OH)_2$	0.12
SO_3	0.05 %	Na_2SO_4	0.02
SiO_2	11.55 %	SiO_2	2.80
SrO	14.41 %	$SrCO_3$	3.73
	----	$Sr(OH)_2 \cdot 8H_2O$	2.24
TiO_2	0.08 %	TiO_2	0.02
ZrO_2	0.23 %	$Zr(OH)_4 \cdot xH_2O$	0.08
	----	Water	10.71
TOTAL	100 %	----	----
Volatiles (g/100 g oxides)			
CO_3	8.240	----	----
NO_2	0.002	$NaNO_2$	0.001
NO_3	1.012	$NaNO_3$	0.313
TOC	1.240	$H_2C_2O_4 \cdot 2H_2O$	1.578
		TOTAL	100

Table 4.3. RSM C-106/AY-102 Waste Surrogate Composition

	Waste Constituent Wt%				
Oxide	**RSM-00**	**RSM-61**	**Average**	**Target**	**%Dev**
Ag_2O	----	0.255	0.255	0.460	-44
Al_2O_3	22.600	22.500	22.600	21.100	7
B_2O_3	0.140	0.118	0.129	0.000	----
BaO	0.260	0.239	0.249	0.130	92
CaO	1.410	1.420	1.410	1.400	1
CdO	0.064	0.093	0.079	0.090	-13
CoO	0.086	0.085	0.085	----	----
Cr_2O_3	0.349	0.329	0.339	0.320	6
Cs_2O	0.138	0.139	0.138	0.200	-31
Fe_2O_3	26.000	24.800	25.400	23.000	11
K_2O	0.208	0.248	0.228	0.050	356
La_2O_3	0.186	0.162	0.174	0.220	-21
Li_2O	0.030	0.031	0.031	0.000	----
MgO	1.410	1.220	1.320	0.430	207
MnO	9.410	8.560	8.980	8.700	3
Na_2O	13.900	16.800	15.300	16.400	-6
NiO	0.276	0.256	0.266	0.300	-11
P_2O_5	----	0.569	0.569	0.300	90
PbO	0.544	0.482	0.513	0.420	22
PdO	----	----	----	0.010	----
SO_3	1.640	1.630	1.630	0.050	3170
SiO_2	15.400	14.800	15.100	11.500	30
SrO	5.300	5.130	5.210	14.400	-64
TiO_2	0.046	0.031	0.038	0.080	-52
ZrO_2	0.201	0.154	0.177	0.230	-23
Sum	100.000	100.000	100.000	100.000	

concentrations <0.5 wt%. Recognizing the factors and limitations reported above, the make-up procedure and chemical certifications were reviewed to gain assurance that sufficient quantities of each material were indeed added to the CY106/AY102 simulant mixture. Based on these analytical data and a review of the CY106/AY102 preparation documentation, it was determined that the surrogate material prepared in support of the RSM's CY106/AY102 flowsheet adequately represented the waste-source surrogate, and no chemical adjustments were deemed necessary.

Table 4.4. Comparison of Analytical and Manufacturer Assay Reports

Element	$Fe(OH)_3$ Slurry Wt% PNNL	$Fe(OH)_3$ Slurry Wt% Manf.	%Dev	Element	$Fe(OH)_3$ Slurry Wt% PNNL	$Fe(OH)_3$ Slurry Wt% Manf.	%Dev
Ag	----	0.0001	----	Mn	0.0012	0.0089	-86
Al	0.0156	0.0040	290	Mo	----	0.0002	----
As	----	0.0003	----	Na	0.0539	0.0490	10
B	0.0035	0.0009	284	Ni	0.0014	0.0010	43
Ba	0.0009	0.0001	770	P	----	0.0009	----
Ca	0.0061	0.0045	36	Pb	----	0.0002	----
Cd	0.0017	0.0001	1473	Pd	----	0.0002	----
Ce	----	----	----	S	0.0096	0.0160	-40
Cl	0.0041	0.0098	-58	Se	----	0.0003	----
Co	0.0012	0.0004	193	Si	0.4990	----	----
Cr	0.0025	0.0012	112	Sn	----	0.0004	----
Cu	----	0.0006	----	Sr	----	0.0009	----
Fe	6.7900	6.7700	0	Ti	0.0155	0.0170	-9
K	0.0101	0.0020	405	V	----	0.0002	----
La	0.0020	0.0001	1940	Y	----	0.0001	----
Li	0.0059	0.0060	-1	Zn	0.0014	0.0036	-62
Mg	0.3210	0.2500	28	Zr	0.0026	0.0011	135

Beyond chemical composition, the physical properties of the C-106/AY-102 surrogate were also measured. Specifically, density and weight losses on drying (LOD), and on ignition (LOI) and equivalent oxides/L measurements were conducted on the C-106/AY-102 surrogate waste. These measured values and their averages are compared to baseline C-106/AY-102 values[(a)] in Table 4.5. The measured oxide loading data suggest a slightly diluted C-106/AY-102 surrogate, but only by ~9%.

Table 4.5. Physical Characteristics of C-106/AY-102 Surrogate Waste

Sample#	Sp. Grav.	LOD (%) (110°C)	Sample LOI (%) Dried	Sample LOI (%) Initial	Oxides (g/L)
RSM-00	1.29	70.0	3.1	73.1	347
RSM-61	1.29	71.4	2.7	74.1	334
Average	1.29	70.7	2.9	73.6	340
Baseline	1.25	71.3	-1.3	70.0	375

(a) WK Kot, H Gan, and IL Pegg. 2000. *Physical and Rheological Properties of Waste Simulants and Melter Feeds for RPP-WTP HLW Vitrification*, Vitreous State Laboratory, The Catholic State University, Washington, D.C.

4.2 Target Glass Composition

The target vitreous product composition for the current RSM flowsheet evaluation was established from a series of laboratory crucible tests involving C-106/AY-102 surrogate material and suitable glass-forming additives that were chosen to maximize product-glass waste loading (Peeler et al. 2002). Since these crucible tests suggested that the glass crystalinity would limit maximum achievable waste loadings, a highly durable glass with the lowest possible crystalinity was formulated for melter testing. The target composition of this glass, designated ICCM-13, is summarized in Table 4.6, where only major constituents are identified.

Table 4.6. Target Glass Composition

Oxide	Wt%
SiO_2	33.80
Fe_2O_3	13.80
Al_2O_3	12.70
B_2O_3	12.10
Na_2O	9.84
SrO	8.64
Li_2O	1.08
CaO	0.84
Balance	7.29

4.3 Glass Former and Chemical Additives

The glass formers used with the reference C-106/AY-102 surrogate waste during the current test are listed and compared to the baseline formulation in Table 4.7. To produce the new target glass formulation for 60 wt% waste-loading, calculated quantities of glass-forming chemicals were blended with a prespecified batch volume of C-106/AY-102 surrogate slurry. The actual glass-forming chemicals employed during this feed-batching operation are summarized in Table 4.8 along with their corresponding oxide-equivalent forms. 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 C-106/AY-102 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 during RSM testing appear in Appendix C along with the other test-data logging sheets.

Table 4.7. Comparative CY-106/AY-102 Glass-Former Additives

	Oxide Wt%		Chemical
Oxide	**Baseline**	**ICCM-13**	**Additive**
B_2O_3	10.00	30.20	H_3BO_3
Li_2O	1.67	2.70	$LiOH{\bullet}H_2O$
SiO_2	86.78	67.10	SiO_2
ZnO	1.55		ZnO

Table 4.8. Melter Feed Glass-Former Additives and Equivalent Frit Composition

Frit	Oxide		Glass Former		Mass Ratio
Oxide	**Wt%**	**MW**	**Chemical**	**MW**	**(g-chem /g-frit)**
B_2O_3	30.20	69.6	H_3BO_3	61.8	0.536
Li_2O	2.70	29.9	$LiOH{\bullet}H_2O$	42.0	0.076
SiO_2	67.10	60.1	SiO_2	60.1	0.671

As mentioned above, an organic reductant, sucrose, was added to some of 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 waste concentrations of sucrose used during the current test ranged from 0 g/L to 9 g/L. Reductant loadings were parametrically varied in ~2.5 g/L increments 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 C-106/AY-102 melter test. These feed samples were subsequently analyzed to determine their physical properties and chemical composition. Table 4.9 presents the physical properties associated with all the feed batches prepared during RSM testing. Also presented in this table, for comparison purposes, is the total-oxide target value derived from the C-106/AY-102 glass composition data previously discussed. Feed total-oxide values derived from laboratory loss-on-drying (LOD, at 110°C) and loss-on-ignition (LOI of wet waste at 1150°C) tests are found to satisfactorily meet expectations based upon the oxide loading of the waste that was previously characterized and summarized in Table 4.5.

Table 4.9. Physical Properties of Surrogate C-106/AY-102 Melter Feeds

		Weight Loss %		g-Oxide/L-Wst	
Sample #	**Sp. Grav.**	**LOD**	**LOI**	**Found**	**Target**
RSM-F1-6	1.41	58.0	65.0	551	567
RSM-F1-19	1.41	58.1	63.5	575	567
RSM-F1-34	1.41	57.8	65.2	548	567
RSM-F1-38	1.41	57.9	63.2	580	567
RSM-F1-51	1.41	57.7	62.2	595	567
Average	1.41	57.9	63.8	570	567

Feed total-oxide values derived from laboratory LOI tests agree quite well with the target expectation value. The above data along with similar waste-related values listed in Table 4.5 suggest that the glass-former additives create a ~12% waste-volume change. The total average oxide loading of the feed calculated from all the feed processed and the glass produced throughout the duration of RSM testing, 560 g-oxide/L, also agrees very well with the values derived from LOI measurements.

The oxide-equivalent compositions of each of the feed batches prepared during RSM testing are summarized in Table 4.10 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 5 days of testing. With the exception of impurity levels that significantly influenced Ba, K, Mg, and S concentrations, there were no trends or large variations of importance, and most components, except for Na, were reasonably close to their target values. The 25% variance for Na is due to analytical difficulties resulting from incomplete feed-sample KOH fusions from which the Na values had to be derived. Similarly, chronic analytical difficulties involving Sr dissolution, as discussed in 4.5.2, is responsible for the low recorded Sr values. In addition, the weight fractions of several trace constituents (Ce, Pd, and Ti) could not be reported due to detection-sensitivity limitations.

Apart from the problems associated with identifiable analytical biases, contaminants and trace constituents just discussed, the overall melter-feed composition data agrees 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 RSM pours, 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 sugar loadings were compositionally analyzed and subjected to toxicity characteristic leach procedure (TCLP) and Product Consistency Testing (PCT). The results of these measurements will now be discussed.

Table 4.10. Melter Feed Batch Composition

	Feed Sample Weight Percent Composition							
Oxide	**RSM-6**	**RSM-19**	**RSM-34**	**RSM--38**	**RSM--51**	**Average**	**Target**	**%Dev**
Ag_2O	0.324	0.324	0.300	0.299	0.426	0.335	0.276	21
Al_2O_3	13.300	12.900	13.700	14.000	13.500	13.500	12.700	6
B_2O_3	15.600	15.800	14.700	12.600	11.400	14.000	12.100	16
BaO	0.135	0.129	0.138	0.136	0.133	0.134	0.078	72
CaO	0.635	0.761	1.150	1.110	1.090	0.949	0.840	13
CdO	0.048	0.046	0.049	0.049	0.048	0.048	0.054	-11
CeO_2	----	----	----	----	----	----	0.036	----
CoO	0.044	0.052	0.043	0.044	0.046	0.046	----	----
Cr_2O_3	0.088	0.091	0.119	0.102	0.105	0.101	0.192	-47
Cs_2O	0.093	0.095	0.104	0.092	0.075	0.092	0.120	-23
Fe_2O_3	11.800	11.300	12.200	12.300	12.000	11.900	13.800	-13
K_2O	0.270	0.313	0.299	0.265	0.270	0.284	0.030	846
La_2O_3	0.094	0.096	0.106	0.102	0.112	0.102	0.132	-23
Li_2O	1.470	1.180	1.270	1.050	1.050	1.200	1.080	11
MgO	0.663	0.628	0.690	0.669	0.771	0.684	0.258	165
MnO	4.790	4.420	4.340	4.380	4.520	4.490	5.220	-14
MoO_3	----	----	----	----	0.104	----	----	----
Na_2O	7.220	9.960	5.370	6.590	7.910	7.410	9.840	-25
NiO	0.143	0.139	0.151	0.154	0.143	0.146	0.180	-19
P_2O_5	----	----	----	----	----	----	0.180	----
PbO	0.349	0.343	0.307	0.329	0.372	0.340	0.252	35
PdO	----	----	----	----	----	----	0.006	----
SO_3	4.870	4.610	4.870	5.390	6.170	5.180	0.030	17,200
SiO_2	35.100	33.500	36.300	36.700	36.200	35.500	33.800	5
SrO	2.800	3.100	3.660	3.550	3.530	3.330	8.640	-62
TiO_2	----	----	----	----	----	----	0.048	----
ZrO_2	0.117	0.145	0.084	0.121	0.129	0.119	0.138	-14

4.5.1 Oxidation State Results

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

A chemical method for measuring an Fe(II) complex colorimetrically at a wavelength of 515 ηm was used to establish the vitreous iron fraction in the +II valance state. This is accomplished by dissolving a powdered glass sample in H_2SO_4 and HF, buffering the resultant solution with sodium acetate/boric acid solution containing o-phenanthroline complexing agent, and conducting an Fe(II)-specific absorbance measurement at 515 ηm. 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 oxidation-state results obtained from glass samples obtained during current RSM testing are summarized in Table 4.11 along with the corresponding sugar-loading values being concurrently used. These sugar-loading and glass-oxidation-state results for the RSM test are also displayed graphically in Figure 4.1 as a function of date and time. These data suggest that sugar concentrations as high as 9 g/L can be tolerated in maintaining acceptable glass-oxidation conditions. Slightly higher sugar concentrations would almost certainly result in an overly reduced glass—a glass where the fraction of iron in the +II oxidation state is >0.3. It should be noted that the variation in Fe(II) percentage exhibited by glasses produced under identical sugar-loading conditions suggests a nominal variability in this Redox parameter of up to ±2%.

Table 4.11. C-106/AY-102 Sugar Loadings and Glass-Oxidation States

Sample	Date/Time	%Fe(II)	Sugar (g/L-Wst)
RSM-F1-3	7/28/02 13:00	1.6*	0
RSM-F1-8	7/29/02 4:12	26.2	9
RSM-F1-20	7/29/02 22:40	24.2	9
RSM-F1-35	7/30/02 21:54	16.7	7.5
RSM-F1-40	7/31/02 6:53	17.3	7.5
RSM-F1-52	7/31/02 22:02	0.1	0
RSM-F1-56	8/1/02 7:09	0.1	2.5
RSM-F1-62	8/1/02 14:51	4.2	5
*Transition glass			

4.5.2 Compositional Data

Glass-product compositional data associated with samples taken throughout all phases of melter processing are summarized in Table 4.12 along with the corresponding target value for the C-106/AY-102 surrogate waste at 60-wt% loading. Apart from the contaminant (Ba, K, Mg, S, etc.) driven variances that have been previously discussed (Section 4.1), all major oxide constituents compared favorably with their respective target values. However, like the waste and feed data discussed earlier, the glass-compositional data also indicate a much lower than expected strontium concentration. Since this result is at odds with the waste makeup data appearing in Appendix B, incomplete fusion digestion is a possible explanation for this ambiguity. Indeed, the average analytical oxide mass yield for the samples summarized in Table 4.12 was only 93%, which supports this supposition.

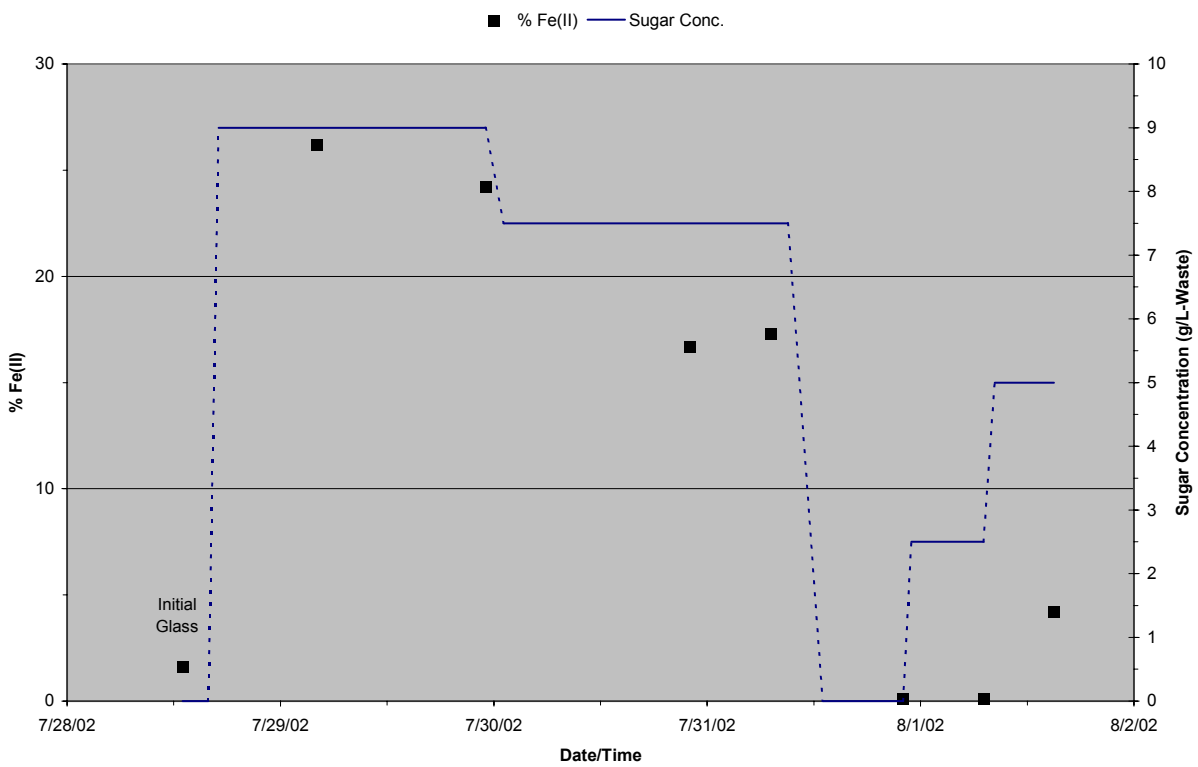


Figure 4.1. Historical Sugar Concentration and Glass-Oxidation-State Values

Additional glass samples that are nominally contemporaneous with those appearing in Table 4.12 were also analyzed at an independent laboratory at the Savannah River Site (SRS). The results of these analyses are summarized in Table 4.13. Although nominally equivalent to results previously presented, the average strontium result obtained is within 10% of the target value. Since the SRS total oxide yields are also in close agreement with expectation (100%), it appears that incomplete fusion digestion is responsible for the low reported PNNL strontium results, not only for the glasses, but also for waste and feed samples previously discussed. Since strontium compounds are difficult to digest, especially when sulfur is present, this analytical bias would not be expected to significantly affect many other waste constituents. And indeed, the comparative data in Table 4.12 and Table 4.13 corroborate this expectation, although the Table 4.13 cadmium and potassium values appear to be systematically biased low. Since, unlike cadmium, impurities are responsible for the potassium levels in the melter feed and no expectation value exits, the factor of 3 difference in glass compositional results for this element suggests an analytical problem associated with this element but doesn't identify the nature or source of error. Consequently, the actual concentration of K is an indeterminate quantity.

Table 4.12. Oxide Composition of Melter C-106/AY-102 Glass Samples

	Normalized Glass Oxide Composition (Wt%)								
Oxide	**RSM-3**	**RSM-8**	**RSM-20**	**RSM-35**	**RSM-40**	**RSM-52**	**Average**	**Target**	**% Dev.**
Ag_2O	0.047	0.223	0.270	0.294	0.303	0.308	0.294	0.276	6
Al_2O_3	8.220	12.400	12.900	12.900	12.700	12.900	12.900	12.700	1
B_2O_3	10.100	12.800	13.300	13.300	13.600	13.500	13.400	12.100	11
BaO	0.025	0.123	0.146	0.136	0.137	0.151	0.142	0.078	83
CaO	1.490	0.984	0.953	0.943	0.858	1.090	0.961	0.840	15
CdO	0.014	0.039	0.043	0.047	0.046	0.049	0.046	0.054	-14
CeO_2	----	----	----	----	----	----	----	0.036	----
CoO	----	0.043	0.047	0.049	0.049	0.055	0.050	----	----
Cr_2O_3	----	0.081	0.073	0.064	0.078	0.094	0.077	0.192	-60
Cs_2O	0.029	0.083	0.092	0.091	0.095	0.094	0.093	0.120	-22
Fe_2O_3	7.710	11.300	11.800	11.800	12.000	12.100	11.900	13.800	-13
K_2O	3.070	0.675	0.438	0.298	0.280	0.361	0.344	0.030	1050
La_2O_3	0.030	0.103	0.120	0.115	0.114	0.120	0.117	0.132	-11
Li_2O	4.180	1.770	1.320	1.250	1.230	1.330	1.280	1.080	19
MgO	0.801	0.757	0.759	0.713	0.725	0.764	0.740	0.258	187
MnO	1.070	3.830	4.240	4.300	4.350	4.430	4.330	5.220	-17
MoO_3	----	----	0.101	0.050	----	0.069	0.074	----	----
Na_2O	12.900	10.500	8.900	9.590	9.620	8.540	9.160	9.840	-7
NiO	0.177	0.163	0.187	0.153	0.163	0.175	0.170	0.180	-6
P_2O_5	1.420	0.396	----	----	----	----	----	0.180	----
PbO	----	0.250	0.298	0.287	0.283	0.309	0.294	0.252	17
PdO	----	----	----	----	----	----	----	0.006	----
SO_3	1.380	3.890	4.270	5.360	4.900	4.870	4.850	0.030	16100
SiO_2	42.200	35.600	36.000	34.400	34.900	35.200	35.100	33.800	4
SrO	0.608	3.120	3.220	3.570	3.350	3.190	3.330	8.640	-61
TiO_2	0.539	0.090	0.051	0.042	----	----	0.047	0.048	-3
V_2O_5	1.140	0.217	0.163	0.040	0.038	0.046	0.072	----	----
ZnO	0.060	0.019	0.012	----	0.012	0.016	0.013	----	----
ZrO_2	2.830	0.533	0.302	0.265	0.226	0.241	0.259	0.138	88

Table 4.13. Independent (SRS) Oxide Composition Data for Melter C-106/AY-102 Glass Samples

	Glass Oxide Composition (Wt%)							
Oxide	**RSM-10**	**RSM-15**	**RSM-30**	**RSM-37**	**RSM-53**	**Average**	**Target**	**% Dev.**
Ag_2O	----	----	----	----	----	----	0.276	----
Al_2O_3	11.400	11.400	11.800	11.800	11.700	11.800	12.700	-7
B_2O_3	12.000	12.800	13.300	13.400	13.300	13.300	12.100	10
BaO	----	----	----	----	----	----	0.078	----
CaO	1.100	1.040	0.935	0.886	0.890	0.904	0.840	8
CdO	0.014	0.012	0.017	0.019	0.020	0.019	0.054	-65
CeO_2	0.056	0.050	0.041	0.041	0.041	0.041	0.036	14
Cr_2O_3	0.179	0.160	0.141	0.140	0.147	0.143	0.192	-26
Cs_2O	----	----	----	----	----	----	0.120	----
Fe_2O_3	11.700	11.700	12.600	12.800	12.800	12.700	13.800	-8
K_2O	0.522	0.437	0.139	0.094	0.114	0.116	0.030	286
La_2O_3	0.131	0.129	0.139	0.144	0.139	0.141	0.132	7
Li_2O	1.590	1.550	1.200	1.200	1.250	1.220	1.080	13
MgO	0.617	0.598	0.556	0.570	0.549	0.558	0.258	116
MnO	3.840	3.920	4.390	4.390	4.450	4.410	5.220	-15
Na_2O	9.710	10.200	10.200	9.990	10.200	10.100	9.840	3
NiO	0.148	0.146	0.136	0.138	0.132	0.135	0.180	-25
P_2O_5	0.243	0.227	0.160	0.149	0.151	0.153	0.180	-15
PbO	0.180	0.182	0.195	0.206	0.204	0.202	0.252	-20
PdO	----	----	----	----	----	----	0.006	----
SO_3	----	----	----	----	----	----	0.030	----
SiO_2	39.200	38.300	36.600	36.200	36.000	36.300	33.800	7
SrO	6.870	6.970	7.820	7.850	7.640	7.770	8.640	-10
TiO_2	0.095	0.084	0.045	0.047	0.043	0.045	0.048	-6
ZrO_2	0.359	0.289	0.121	0.108	0.119	0.116	0.138	-16
Total	99.900	100.000	100.000	100.000	99.900	100.000		

The composition data in Table 4.12 and Table 4.13 also suggest that the melter's molten glass pool does not behave like an ideal stirred tank. Both sets of compositional data suggest that glass-composition transitioning was occurring for up to 36-h after startup: ~5 volume turnovers. Specifically, the temporal compositional behavior of potassium clearly showed a definite downward trend until sequential glass sample RSM-20 was taken. As a result, averages in Table 4.12 and Table 4.13 do not include the results obtained for the initial two samples in each set. Based on a simple comparison of product glass analytical and target compositions, the classic volatiles/semi-volatiles feed components (e.g., B, alkalis, etc.) do not appear to have partitioned significantly to the process exhaust. Partitioning values or melter decontamination factors (DFs) will be discussed in a later section dealing with off-gas emission characterization.

4.5.3 TCLP Results

Representative glass samples generated throughout all phases of testing were subjected to the U.S. Environmental Protection Agency's (EPA's) TCLP test (TCLP 1992). In that procedure crushed glass is leached with a sodium acetate buffer solution for 18-h at 22°C while under constant, end-over-end agitation. The leachate compositions were subsequently analyzed, and the results obtained are summarized in Table 4.14.

Table 4.14. TCLP Leachate Concentrations from C-106/AY-102 Glasses

	Leachate Concentration (mg/L)					
Item	**Ag**	**Ba**	**Cd**	**Cr**	**Pb**	**Sugar (g/L)**
UTS Limit	0.14	21	0.11	0.6	0.75	
RSM-H1-23	0.11	<0.50	<0.025	<0.50	<0.25	9
RSM-H1-44	0.13	<0.50	0.027	<0.50	<0.25	7.5
RSM-H1-58	0.14	<0.50	<0.025	<0.50	<0.25	0 & 2.5

The concentrations of most of the hazardous analytes were found to be well below their respective analytical reporting limits. Although silver concentrations never exceeded the Universal Test Standard (UTS) limit, they were nevertheless at of just slightly below this limit for all glasses tested. In addition, cadmium leachate concentrations for all glasses analyzed were slightly below or in one case just above the analytical reporting value (0.025 ppm) for this element; however in all cases the cadmium leachate concentrations were a factor of 4 lower than UTS limit for this element. It should be noted, however, that despite the high silver leaching characteristics, the glass produced from this new C-106/AY-102/ICCM-13 formulation still conforms with all existing Resource Conservation and Recovery Act (RCRA) land-disposal limits (40 CFR 268).

4.5.4 PCT Results

Representative glasses collected throughout the duration of RSM testing of the optimized C-106/AY-102 flowsheet were also subjected to product consistency testing (PCT; ASTM 1997), which, like TCLP testing, measures leach resistance of crushed (75 to 149 µm) glass samples. PCT leach conditions involve the use of deionized water held at 90°C without agitation. The results obtained, which are summarized in Table 4.15, were found, without exception, to be superior to the standard Environmental Assessment (EA) glass (Jantzen et al. 1993) that was run concurrently with the C-106/AY-102 glass samples. Moreover, these data are also found to be totally consistent with previous laboratory testing that supported the development of the glass formulation used during the current RSM flowsheet test (Peeler et al. 2002).

Table 4.15. PCT Leachate Results from C-106/AY-102 Glasses

Sample/Units	B	Li	Na	Si
Target Ox(%)				
All	12.1	1.1	9.8	33.8
PCT(ppm)				
RSM-F1-10	13.1	3.3	29.8	29.0
RSM-F1-15	13.9	3.0	30.8	26.3
RSM-F1-30	14.0	2.4	29.7	24.9
RSM-F1-37	15.7	2.7	33.0	28.1
RSM-F1-53	15.0	2.4	27.7	25.1
Norm (g/L)				
RSM-F1-10	0.350	0.666	0.409	0.184
RSM-F1-15	0.371	0.590	0.422	0.167
RSM-F1-30	0.374	0.473	0.407	0.158
RSM-F1-37	0.418	0.540	0.451	0.178
RSM-F1-53	0.401	0.483	0.380	0.159
Norm (g/m^2)				
RSM-F1-10	0.175	0.333	0.204	0.092
RSM-F1-15	0.186	0.295	0.211	0.083
RSM-F1-30	0.187	0.237	0.204	0.079
RSM-F1-37	0.209	0.270	0.226	0.089
RSM-F1-53	0.200	0.242	0.190	0.080

4.5.5 Spinel Secondary Phase

Pour-stream and core-drilled melt-cavity glass samples collected during and after melter testing of the optimized C-106/AY-102 glass formulation, respectively, were examined using a scanning electron microscope to establish the degree and extent of spinel secondary-phase formation. The pour-stream samples that were collected in small graphite boats cooled quickly and are directly comparable to laboratory crucible melt samples that were conducted over a range of temperatures to qualify the ICCM-13 glass formulation. Table 4.16 summarizes the temperature dependence of spinel crystal formation characterized in these laboratory tests and compares the crystallinity results obtained from three RSM glass pour samples produced at an invariant 1150°C melt temperature. These data show that glasses that are formed under dynamic processing conditions and are quickly cooled exhibit a lower concentration of spinel crystals than those produced under similar but static melting conditions.

To determine if there was any significant settling of spinel in the melter during the 5 days of testing, core samples of the frozen glass pool were collected from the center and periphery of the cylindrical melt cavity. Glass samples, collected from five separate locations along these glass cores that extended from the surface to the bottom of the melt cavity, were examined to determine spinel secondary-phase concentrations. The results obtained are plotted in Figure 4.2.

Table 4.16. Spinel Content of Laboratory and Melter Glasses

Sample	Wt% Spinel			
	1000 °C	1050 °C	1100 °C	1150 °C
Lab Crucible	5.4	3.8	4.1	1.8
RSM-F1-31	----	----	----	0.4
RSM-F1-46	----	----	----	0.4
RSM-F1-55	----	----	----	0.6

The significant cool down period associated with normal melter shut-down conditions is responsible for the overall increased spinel concentrations observed. However, these data do not indicate any significant stratification of spinel within the RSM's glass volume, although a significant scatter in these data is present. It should be noted that preliminary crucible tests for this glass formulation under canister centerline cooling conditions suggest a secondary spinel phase formation as high as 6.7 wt%.

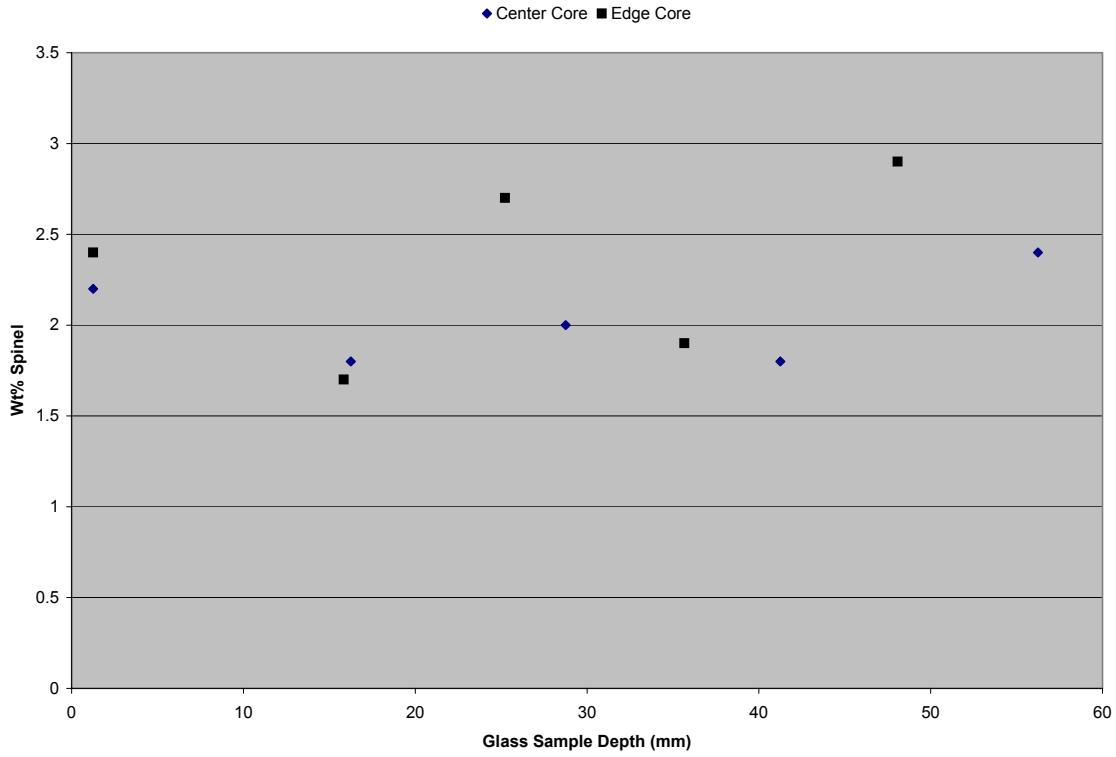


Figure 4.2. Spinel Concentrations Within the RSM's Frozen Glass Volume

5.0 Discussion of Results

RSM testing of the optimized C-106/AY-102 flowsheet was initiated on July 28, 2002, and concluded on August 1, 2002. During this 120-h period, C-106/AY-102 melter feeds containing varying reductant concentrations but at a fixed 60 wt% waste loading were successfully processed. In addition, the impact of varying the water content of the melter feed was also evaluated. The observations and experimental test results derived from all phases of C-106/AY-102 melter testing will now be discussed.

5.1 Processing Observations and Parameters

The melting characteristics of the C-106/AY-102 feed formulation under study were found to be dependent upon reductant loading. Feeds containing only the oxalic acid present in the Reference C-106/AY-102 waste processed extremely well until foaming limited heat-transfer rates between the molten glass pool and the melter cold-cap. The RSM test was initiated with feed containing no sugar. However, the transition glass began to foam after only ~3 h of processing whereupon the feed tank's waste concentration of sugar was adjusted to 9 g /L, which ultimately eliminated the melter foaming condition. Later in the test, the processing of feed containing no sugar was resumed but was terminated after 10-h of processing due to very active foaming conditions. Although ultimately limited by foaming instabilities, processing the C-106/AY-102 melter-feed formulation without sugar created a relatively well-behaved steady-state cold-cap structure that was less prone to accumulate feed than was the case when sugar was used to control the glass-oxidation state.

Four distinct melter-feed sugar loadings (2.5, 5.0, 7.5, and 9.0 g/L of waste) were evaluated during C-106/AY-102 flowsheet testing. Since the impact of reductant loading upon the glass oxidation state has been previously discussed (see Section 4.5.1), the current discussion will be limited to processing characteristics. As in the case of no supplemental reductant, feed containing 2.5 g of sugar/L of waste was not sufficiently reduced to eliminate foaming. Although not nearly as severe as when no sugar was added, the foam produced was sufficient to reduce heat transfer and related resultant processing rates. All other sugar feed concentrations were found to adequately reduce the glass to eliminate the processing impediment due to foam formation.

The addition of sugar to the C-106/AY-102 feed formulation not only influenced melter foaming behavior but also affected melter cold-cap characteristics. All feeds containing sugar exhibited a tendency to accumulate incoming feed in the form of a cone that developed beneath the feed nozzle. The material making up the cone was composed primarily of dry feed, which was friable and prone to spontaneous collapse, although the cone had to be physically pushed over several times during the test to eliminate feed-nozzle interference. The results of cone collapse usually resulted in 100% cold-cap coverage conditions that necessitated feed-rate reductions to accommodate the increased material in the melter reaction zone.

Beyond the influence of sugar upon the glass oxidation state (see Section 4.5.1) and cold cap characteristics, sugar at all concentration used during the current test appears to have efficiently reduced a significant quantity of the nitrate present in the waste to N_2. During both processing campaigns containing no sugar additive, NO dominated the melter's nitrogen oxide emission source term. However,

whenever sugar was present in the melter feed, off-gas concentrations of NO were significantly reduced. This subject will be discussed in greater detail is Section 6.1.

The processing characteristics documented for each of the feed batches prepared during RSM testing are summarized in Figure 5.1. These data reflect the average rates observed during both active feed-processing periods and non-feeding time intervals, and as such are conservative estimates. By combining all feed-batch dropout data, an average melter feeding rate for all C-106/AY-102 feeds over the entire processing campaign (feeding and nonfeeding) can be graphically derived as is also shown in Figure 5.1.

As is apparent from Figure 5.1, several of the batch-processing curves are composed of feeds containing differing sugar reductant concentrations. Appendix E contains graphical representations of all the components present in these overall batch processing curves, and Table 5.1 summarizes the processing rates and derived glass production rates for all individual feed-reductant conditions evaluated during RSM testing. The relationship between feed-processing and glass-production rates used in this table was derived from empirical feed property data previously discussed in Section 4.4.

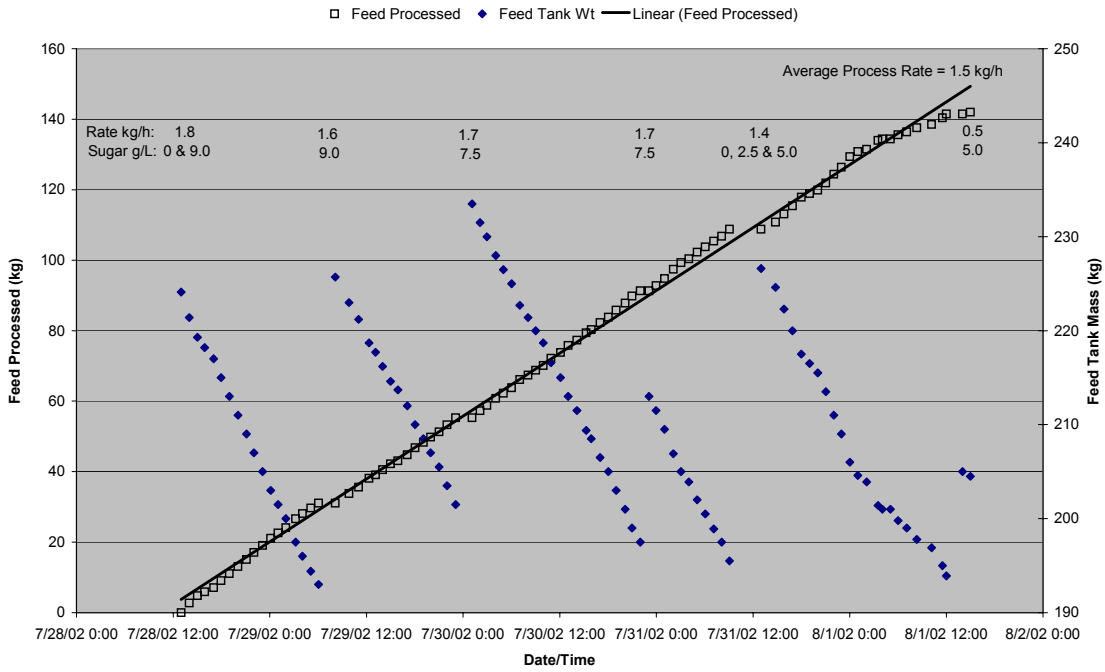


Figure 5.1. Process History of RSM Feed Batches and Average Overall Processing Rate

Although the data presented in Appendix E and Table 5.1 suggest that processing the C-106/AY-102 feed formulation without a sugar additive is advantageous, these data only reflect conditions that exist before the onset of glass foaming. Since foaming creates unstable processing conditions and is therefore undesirable, feed processing was terminated whenever foaming created processing difficulties. On this basis, processing of the C-106/AY-102 feed formulation without a sugar additive is not a viable option unless physical/mechanical methods are employed to eliminate foam layer development.

Table 5.1. C-106/AY-102 Feed Processing Rates and Derived Glass Production Rates

Feed Process Period		Fd Rate	Volumetric Rate (L/h)		Glass Production		Sugar
Start	Stop	kg/h	Feed	Waste	kg/h	Lbs/h/Ft^2	g/L-Wst
7/28/2002 13:00	7/28/2002 16:00	2.05	1.45	1.30	0.74	8.31	0.0
7/28/2002 17:00	7/29/2002 6:03	1.86	1.32	1.18	0.67	7.54	9.0
7/29/2002 8:08	7/29/2002 23:05	1.58	1.12	1.00	0.57	6.44	9.0
7/30/2002 1:07	7/30/2002 22:00	1.69	1.20	1.07	0.61	6.88	7.5
7/30/2002 23:05	7/31/2002 9:05	1.72	1.22	1.09	0.62	7.00	7.5
7/31/2002 13:00	7/31/2002 22:58	1.76	1.25	1.12	0.64	7.15	0.0
7/31/2002 22:58	8/1/2002 8:20	1.09	0.77	0.69	0.39	4.43	2.5
8/1/2002 8:20	8/1/2002 14:00	0.86	0.61	0.54	0.31	3.49	5.0
7/28/2002 13:00	8/1/2002 14:00	1.53	1.08	0.97	0.55	6.20	all
7/31/2002 13:00	8/1/2002 12:00	1.44	1.02	0.91	0.52	5.86	0, 2.5 & 5

The average processing rates for feeds containing waste sugar concentrations of 9 and 7.5 g/L were only slightly lower than the prefoaming rates obtained with feeds containing no sugar. However, processing conditions involving these feeds were stable, and subsequent changes in reductant concentrations during this test were only initiated to influence glass-oxidation conditions and not necessitated by processing instabilities. It should be noted that water dilution of the melter feed was to some extent responsible for the reduced average processing rate observed during the 2nd of two processing campaigns utilizing 9 g of sugar/L of waste. A ~10% water dilution of the incoming melter feed stream was somewhat successful in reducing cold-cap accumulation rates due to coning, but processing rates were also reduced.

Fairly good processing-rate reproducibility was observed for the sequential processing campaigns utilizing waste sugar concentrations of 7.5 g/L, which was nominally equivalent to both the average rate observed during previous processing of feeds containing 9 g of sugar/L of waste and the subsequent long-term processing of feed containing no sugar. The low processing rate observed for the feed containing 2.5 g of sugar/L of waste, as mentioned earlier, was due to chronic low-level foaming conditions resulting from an initial, strongly oxidized melt pool and a feed containing an inadequate sugar content.

Unlike the under-reduced feed containing 2.5 g of sugar/L of waste, the feed containing 5 g of sugar/L of waste was sufficiently reduced to eliminate the higher oxidation state of manganese in the product glass as previously discussed in Section 4.5.1. Although foaming was not a processing issue, cold-cap coning was. As is clearly shown in the graphical data presented in Appendix E, the very low average production rate associated with this feed was due to significant non-feeding periods occurring during the feed-processing campaign. Since systematic arguments can easily eliminate sugar content as a mitigating factor responsible for the cold-cap (enhanced coning) difficulties encountered with this feed, changes in feed-water content due to 4 days of evaporative losses is the likely cause of the cold-cap coning problems encountered.

Also included in Table 5.1 are specific glass-production rates derived from the experimentally determined total oxide loading of the feed (see Section 4.4), the glass pool surface area (6-in. dia.), and the corresponding melter feeding rates. These values comfortably exceed the reference (cold-lid) LFCM design production rate of 4 lbs/h/ft^2 that is often quoted and used for flowsheet and equipment-sizing

estimates (Perez et al. 2001). Indeed, this reference-normalized production rate is even exceeded when projections are based upon the overall average feeding rate that is inclusive of all idle batching periods. This derived overall average-production-rate value also agrees fairly well with the actual glass-accumulation information (0.55 vs 0.49 kg/h) 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 5.2 and in Table 5.2.

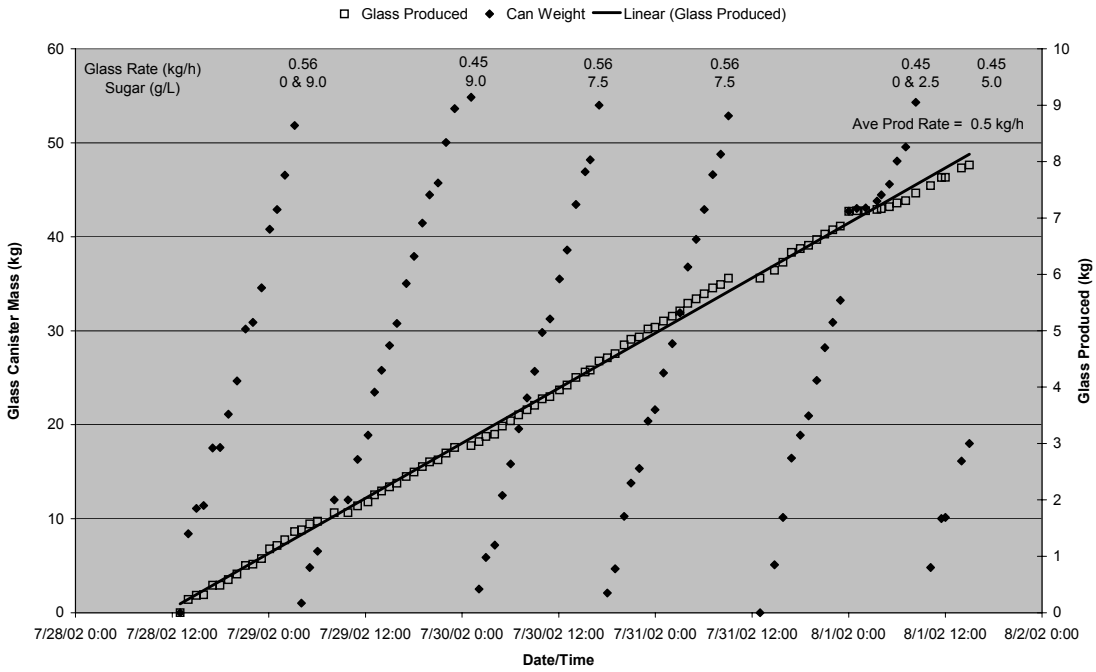


Figure 5.2. C-106/AY-102 Glass Accumulation Data

Table 5.2. Experimental C-106/AY-102 Glass Production Rate Data

Feed Process Period		Glass Production		Sugar
Start	Stop	kg/h	Lbs/h/Ft^2	g/L-Wst
7/28/2002 13:00	7/29/2002 3:12	0.56	6.3	0 & 9.0
7/29/2002 4:03	7/30/2002 1:07	0.45	5.1	9.0
7/30/2002 2:06	7/30/2002 17:01	0.56	6.3	7.5
7/30/2002 18:02	7/31/2002 9:05	0.56	6.3	7.5
7/31/2002 13:00	8/1/2002 8:20	0.45	5.1	0 & 2.5
8/1/2002 10:10	8/1/2002 15:00	0.45	5.1	5.0
7/28/2002 13:00	8/1/2002 15:00	0.49	5.5	all

Apart from the nominally good overall production-rate agreement between feed and glass data, the batch-production data differ markedly. The reason for this is that the glass data include nonfeeding periods between batches whereas only the overall average feed production value includes these nonproduction periods. Consequently, the glass data should significantly underestimate the actual average batch-production rates, while the feed-derived values should be much more representative.

Axiomatically, both feed and glass overall production rates are conservative values that are systematically biased low.

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 5.3 summarizes the maximum, minimum, and average temperatures of the melter's glass, plenum, and off-gas stream throughout the entire duration of C-106/AY-102 melter flowsheet evaluation. 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 5.4 provides similar daily tabular data for the melter's kiln, overflow spout, and canister oven.

All melter and off-gas process-temperature data collected during the C-106/AY-102 flowsheet evaluations are graphically presented in Appendix E. Unlike the plenum and melter off-gas (MOG) temperatures, the temperature variations of melter glass and EVS exhaust temperatures are, as expected, relatively small since both temperatures were actively controlled electrically or through use of a heat exchanger, respectively.

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.

Table 5.3. RSM's Operating Temperature Characteristics

Date/Item	Temperature (°C) Ave	Std Dev	Min	Max	Date/Item	Temperature (°C) Ave	Std Dev	Min	Max
7/28/2002					7/31/2002				
Glass	1150.0	5.20	1130.0	1160.0	Glass	1150.0	3.60	1140.0	1160.0
Plenum	574.0	38.80	480.0	705.0	Plenum	626.0	65.70	455.0	793.0
Mltr Exh	113.0	22.60	80.0	181.0	Mltr Exh	119.0	17.60	73.5	182.0
EVS Exh	33.2	1.26	30.5	36.0	EVS Exh	29.9	1.30	27.1	32.5
Scrub Liq	31.8	1.00	29.0	34.1	Scrub Liq	29.2	0.96	26.5	31.6
7/29/2002					8/1/2002				
Glass	1150.0	5.55	1100.0	1170.0	Glass	1150.0	4.85	1100.0	1170.0
Plenum	603.0	52.70	500.0	785.0	Plenum	561.0	62.10	340.0	699.0
Mltr Exh	113.0	24.00	64.0	191.0	Mltr Exh	105.0	24.60	53.5	168.0
EVS Exh	31.1	1.41	28.0	34.5	EVS Exh	29.0	1.48	25.5	32.5
Scrub Liq	29.9	0.88	27.5	31.9	Scrub Liq	28.5	1.10	25.5	30.5
7/30/2002					7/28 - 8/01				
Glass	1150.0	3.31	1140.0	1160.0	Glass	1150.0	4.57	1100.0	1170.0
Plenum	581.0	58.20	413.0	777.0	Plenum	593.0	62.00	340.0	793.0
Mltr Exh	102.0	13.60	70.0	156.0	Mltr Exh	111.0	21.40	53.5	191.0
EVS Exh	30.7	1.30	27.5	33.5	EVS Exh	30.6	1.78	25.5	36.0
Scrub Liq	29.7	0.83	27.5	31.6	Scrub Liq	29.7	1.30	25.5	34.1

Table 5.4. Melter Kiln, Pour Spout, and Canister Oven Temperatures

Date/Statistic	Temperature (°C) Kiln Bot	Kiln Mid	Kiln Top	Pour Spout	Can Oven	Date/Statistic	Temperature (°C) Kiln Bot	Kiln Mid	Kiln Top	Pour Spout	Can Oven
7/28/2002						7/31/2002					
Average	854.00	851.00	815.00	1150.00	750.00	Average	856.00	855.00	821.00	1150.00	749.00
Std Dev	2.63	3.70	4.37	2.77	3.34	Std Dev	4.85	7.12	7.21	3.53	18.10
Minimum	847.00	843.00	803.00	1140.00	730.00	Minimum	846.00	844.00	811.00	1130.00	463.00
Maximum	860.00	862.00	832.00	1160.00	771.00	Maximum	869.00	872.00	843.00	1170.00	785.00
7/29/2002						8/1/2002					
Average	854.00	851.00	818.00	1150.00	746.00	Average	851.00	851.00	813.00	1150.00	749.00
Std Dev	2.53	3.50	4.00	15.80	23.40	Std Dev	2.17	2.90	3.03	3.09	12.60
Minimum	845.00	843.00	811.00	880.00	491.00	Minimum	846.00	843.00	807.00	1130.00	585.00
Maximum	865.00	868.00	837.00	1160.00	769.00	Maximum	857.00	858.00	820.00	1170.00	781.00
7/30/2002						7/28 - 8/1					
Average	854.00	851.00	817.00	1150.00	748.00	Average	854.00	852.00	817.00	1150.00	748.00
Std Dev	2.32	3.59	4.18	3.38	18.00	Std Dev	3.44	4.89	5.52	8.28	17.80
Minimum	844.00	843.00	810.00	1130.00	516.00	Minimum	844.00	843.00	803.00	880.00	463.00
Maximum	862.00	865.00	834.00	1170.00	784.00	Maximum	869.00	872.00	843.00	1170.00	785.00

A graphical summary of these operating parameters extracted from the hourly process log is presented in Figure 5.3. Recognizing that automatic off-gas control based on plenum pressure was inoperable throughout the entire duration of RSM testing, it is apparent that vacuum control of the melter and its off-gas system was easily maintained throughout all phases of testing.

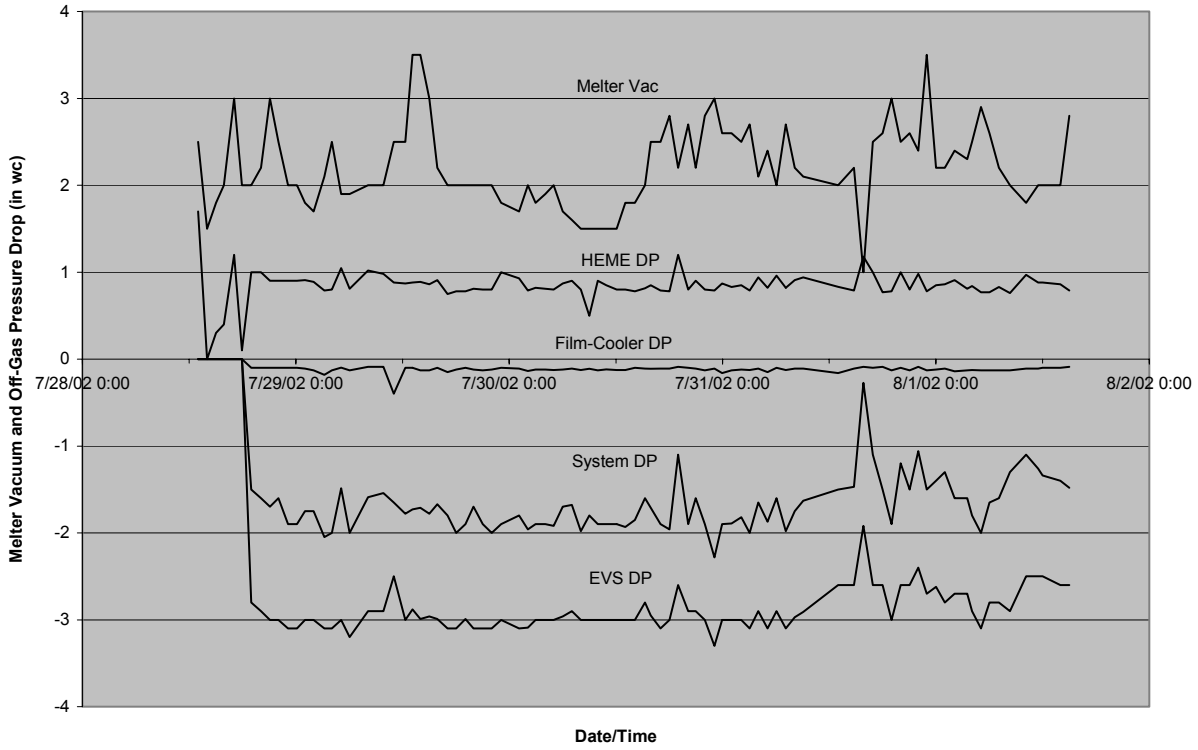


Figure 5.3. Melter Vacuum and Off-Gas Pressure Drop Data

As an explanatory note, the EVS, apart from all other off-gas system devices, assisted the blower in maintaining the melter-plenum vacuum. Specifically, the operation of this device created aspiration conditions that help pump gas from the melter's plenum. As is clear from the stable and nominally invariant film-cooler pressure-drop data, no off-gas obstructions formed within this device during C-106/AY-102 melter flowsheet testing. Nonuniform gas distribution within the previously used and replaced film cooler was apparently responsible for creating off-gas blockages during previous RSM testing (Goles et al. 2001).

It should be noted that the variability in melter vacuum throughout the RSM test resulted from the need to manually control melter vacuum. This resulted from a failure of an automatic control valve that is designed to maintain melter vacuum at an operator-determined setpoint. However, since no significant off-gas accumulations occurred during RSM testing, manual melter vacuum control was easily maintained throughout all phases of C-106/AY-102 testing.

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).

Figure 5.4 graphically summarizes all of these electrical quantities. 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 most of the melter test. Also apparent from these graphical data are strong correlations between voltage, power, and resistance, as is expected from their functional interrelationships. The large fluctuations in power levels toward the conclusion of testing resulted from previously described foaming events that affected bulk glass resistance and the ability of the control system to maintain the melter's glass temperature setpoint. Higher time-resolved plots of these electrode parameters that provide much more detail are displayed in Appendix E for the interested reader.

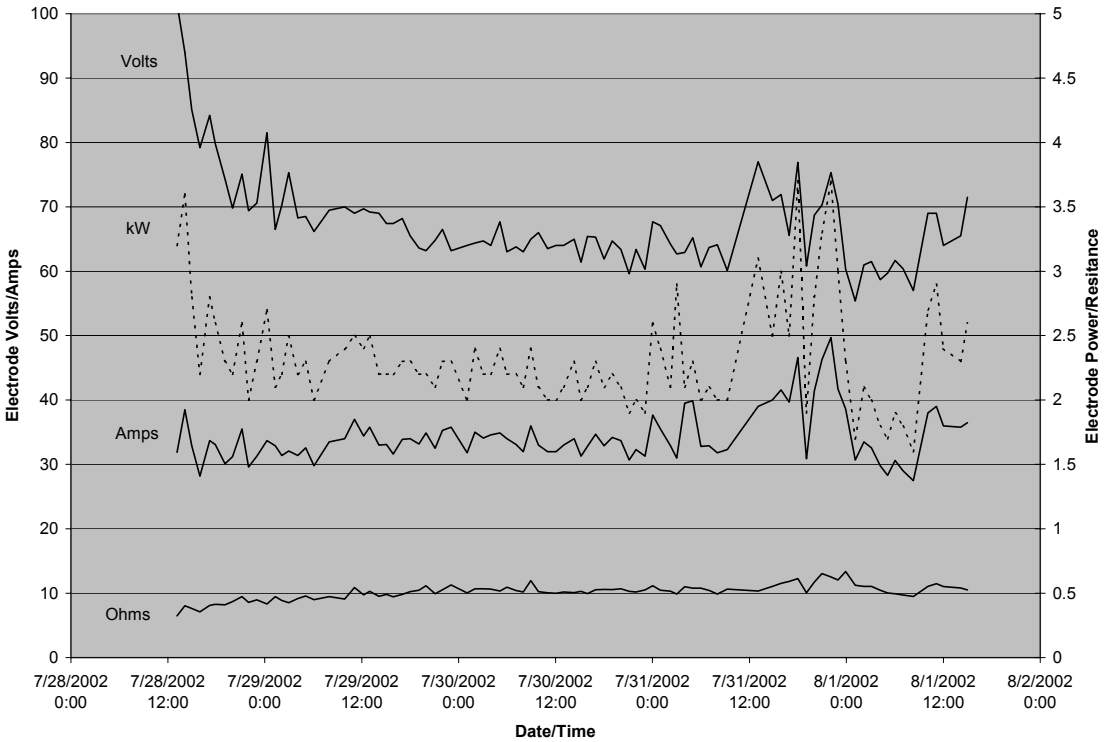


Figure 5.4. RSM Electrode Operating Characteristics

Table 5.5 summarizes the maximum, minimum, and average electrode operating parameters and their standard deviations during all active batch-processing periods (see Figure 5.1). 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 C-106/AY-102 feeds. These data are summarized in Table 5.6.

Table 5.5. RSM Electrode Circuit Operating Characteristics

Date/Sugar Statistic	RSM Electrode Parameter Volt	Amp	kVA	Ohm	Date/Sugar Statistic	RSM Electrode Parameter Volt	Amp	kVA	Ohm
7/28, 0					7/31, 0				
Average	33.60	89.90	3.07	0.37	Average	40.90	69.50	2.87	0.59
Std Dev	6.64	9.90	0.83	0.06	Std Dev	5.57	5.30	0.59	0.05
Min	24.80	70.60	1.75	0.29	Min	28.40	55.70	1.58	0.49
Max	54.10	107.00	5.39	0.54	Max	55.40	82.30	4.56	0.69
7/28, 9					7/31, 2.5				
Average	32.20	73.50	2.37	0.44	Average	32.02	59.87	1.93	0.53
Std Dev	1.89	4.96	0.25	0.03	Std Dev	3.93	4.71	0.41	0.04
Min	28.10	61.00	1.78	0.37	Min	27.42	49.70	1.43	0.46
Max	41.90	91.30	3.29	0.56	Max	59.71	97.61	5.83	0.68
7/29, 9					8/1, 5				
Average	33.80	66.80	2.26	0.51	Average	36.13	66.39	2.41	0.54
Std Dev	2.00	3.36	0.23	0.02	Std Dev	3.22	3.88	0.34	0.03
Min	28.80	57.30	1.70	0.45	Min	28.08	56.91	1.60	0.47
Max	40.90	77.30	3.06	0.59	Max	42.72	76.51	3.16	0.59
7/30, 7.5					7/28-8/1, All				
Average	33.50	64.20	2.15	0.52	Average	34.21	67.14	2.31	0.51
Std Dev	1.82	2.47	0.19	0.02	Std Dev	4.07	7.19	0.45	0.06
Min	29.00	52.50	1.65	0.47	Min	24.84	49.70	1.43	0.29
Max	39.70	72.70	2.82	0.63	Max	59.71	107.21	5.83	0.69
7/30, 7.5									
Average	33.40	63.60	2.13	0.53					
Std Dev	1.92	2.31	0.19	0.02					
Min	29.30	58.60	1.76	0.48					
Max	44.50	72.00	3.15	0.65					

Table 5.6. Specific Process Energy Requirements For C-106/AY-102 Feeds

Start Date/Time	Feeding Rate (l/h)	Glass Lbs/h/ft^2	Sucrose $(g/L)_{waste}$	Power (kW) Joule	Power (kW) Combust	Spec Engy (kW•h/kg) Joule	Spec Engy (kW•h/kg) Total
7/28/2002 13:00	1.45	8.3	0.0	3.1	0.000	4.1	4.1
7/28/2002 17:00	1.32	7.5	9.0	2.4	0.044	3.5	3.6
7/29/2002 8:08	1.12	6.4	9.0	2.3	0.038	3.9	4.0
7/30/2002 1:07	1.20	6.9	7.5	2.2	0.034	3.5	3.6
7/30/2002 23:05	1.22	7.0	7.5	2.1	0.034	3.4	3.5
7/31/2002 13:00	1.25	7.1	0.0	2.9	0.000	4.5	4.5
7/31/2002 22:58	0.77	4.4	2.5	1.9	0.007	4.9	4.9
8/1/2002 8:20	0.61	3.5	5.0	2.4	0.011	7.8	7.8
(a) Exclusive of 2.5 and 5 g-sugar/L-waste campaigns						Average[(a)] =	3.9
						Std Dev =	10%

Typical energy requirements for slurry-fed Joule-heated ceramic melters range from 2 to 4 kW•h/kg of glass produced (Perez et al. 2001). The average specific energy requirements determined for the C-106/AY-102 feeds processed fall nicely within this range. Recalling that dilution water was used during part of the 2nd campaign involving feed with a waste-sugar concentration of 9 g/L, Table 5.6 data illustrate very good specific energy reproducibility among campaigns having similar sugar and water loadings. The last two campaigns involving 2.5 and 5 g of sugar/L of waste were not included in the average specific energy value, as steady-state processing conditions were, for one reason or another, difficult to achieve. It should also be noted that in addition to the electrical power delivered to the melt pool, combustion energy was also being provided by the reductant feed-stream component. By combining this combustion energy source to the Joule heating component for each of the RSM's processing campaigns, total 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 C-106/AY-102 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 5.7.

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 5.3, the EVS's 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 5.5 provides a historical summary of the EVS's scrubbing liquor/condensate tank volume throughout the duration of the RSM test.

The 0.23 L/h condensate accumulation rate is 26% of the overall average rate to which water was fed to the melter (see Figure 5.1 and Table 4.9). If one directly compares the accumulated condensate volume (24 L [6.34 gal], neglecting solids contributions) and the total water volume (82 L [21.7 gal]) fed to the melter, a 29% steam recovery is obtained, which is in good agreement with the average accumulation-rate value.

Also presented in Figure 5.5 is the manually recorded Film Cooler, injection-air flowrate. The transient flow excursions illustrated by these graphical data are apparently real as these artifacts are also represented in total off-gas flowrate data. 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. Total process flow rates will be discussed and characterized in Section 6.1.

Table 5.7. Operational Characteristics of Melter Kiln and Overflow Heaters

	Kiln Electrical Parameter			OverFlow Heater Parameter		
Date/Item	**Volt**	**Amp**	**kVA**	**Volt**	**Amp**	**kVA**
7/28/2002						
Average	4.58	0.13	20.00	14.40	2.07	143.00
Std Dev	2.88	0.09	11.90	0.46	0.14	5.14
Min	0.00	0.00	1.25	13.20	1.71	130.00
Max	8.09	0.28	35.00	15.50	2.39	155.00
7/29/2002						
Average	5.43	0.15	23.50	14.70	2.17	147.00
Std Dev	2.31	0.08	9.62	1.16	0.25	11.90
Min	0.00	0.00	1.00	0.00	0.00	2.75
Max	8.10	0.28	35.00	18.90	3.43	182.00
7/30/2002						
Average	5.91	0.17	25.50	14.60	2.12	145.00
Std Dev	2.35	0.08	9.78	0.52	0.16	5.81
Min	0.00	0.00	1.00	13.20	1.73	130.00
Max	8.64	0.32	37.30	15.80	2.51	159.00
7/31/2002						
Average	3.30	0.08	14.70	14.60	2.13	145.00
Std Dev	2.95	0.08	12.10	0.66	0.21	7.42
Min	0.00	0.00	1.00	12.80	1.61	126.00
Max	7.49	0.24	32.30	16.10	2.60	162.00
8/1/2002						
Average	3.17	0.06	13.80	14.50	2.08	143.00
Std Dev	2.26	0.06	9.36	0.52	0.16	5.83
Min	0.00	0.00	0.75	13.20	1.72	130.00
Max	7.76	0.26	33.50	15.70	2.46	157.00
7/28 - 8/01						
Average	4.57	0.12	19.90	14.60	2.12	145.00
Std Dev	2.80	0.09	11.60	0.76	0.20	8.07
Min	0.00	0.00	0.75	0.00	0.00	2.75
Max	8.64	0.32	37.30	18.90	3.43	182.00

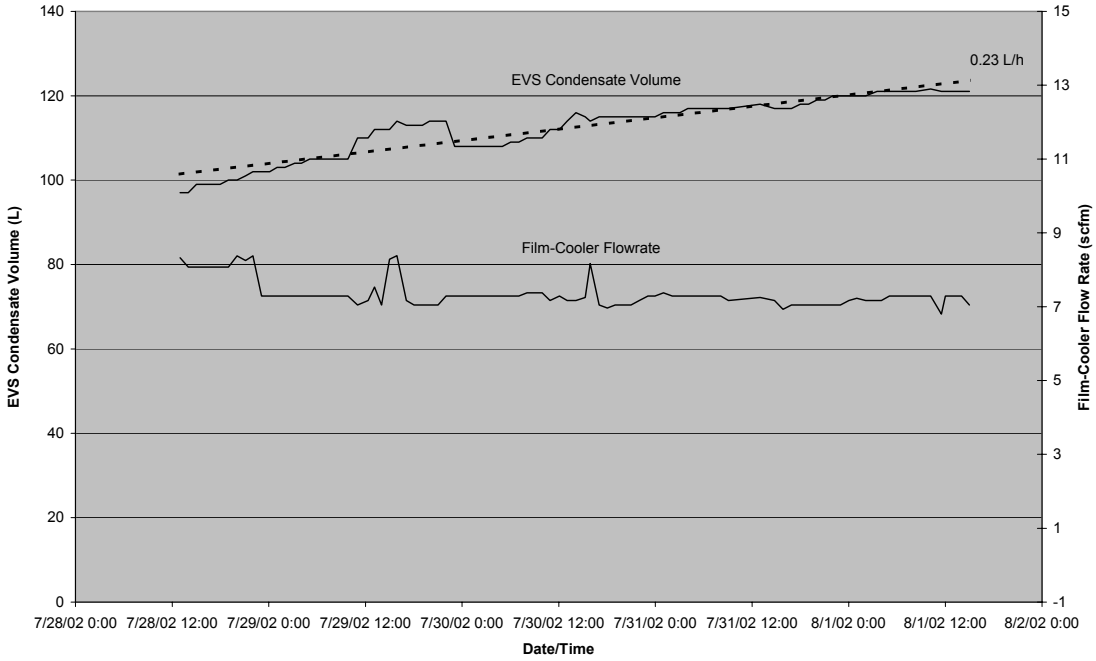


Figure 5.5. EVS Condensate Tank, Film-Cooler Operating History

6.0 Melter Off-Gas Emission Characterization

Off-gas effluent studies were conducted during C-106/AY-102 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 (25°C) as well as condensable effluents.

The composition of melter-generated, non-condensable effluent emissions was established using the gas analyzers described Table 3.2. The instruments used were designed to continuously (or quasi-continuously) monitor and record process-exhaust concentrations of H_2, He, N_2, CO, NO, O_2, CO_2, N_2O, and NO_2. In addition, 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 is usually 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 results of several previous sampling campaigns (Goles et al. 2001, 2002) have shown that an ice-bath immersed condenser is often more than sufficient to quantitatively capture the acid gases specific to C-106/AY-102 waste composition. Consequently, the sampling system employed during the current test did not employ chemical gas scrubbers.

The operational data and experimental results obtained from the melter off-gas studies conducted in support of the C-106/AY-102 flowsheet evaluations are discussed below.

6.1 Gaseous Effluent

For the surrogate C-106/AY-102 melter feed used during the July/August 2002 test, CO_2 and NO_x (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 Table 5.1). Also presented in this table are the standard deviations associated with the average concentrations listed, and off-gas flow-rate data.

For a non-condensable off-gas flow rate of ~10 scfm and a steady-state feeding rate of 1.1 L/h, the MOG concentrations of the major effluent gases (CO_2 and NO) were found to be, nominally, 0.22% and 0.001%, respectively. The combustible gas CO was not detectable (<250 ppm), and H_2 was only detectable at high sugar loading conditions. Throughout all phases of processing, the H_2 concentration averaged only 7 ppm, and the maximum concentration recorded, 0.018%, occurred during processing of feed containing 9 g of sugar/L of waste. These concentrations are well below the lower flammability limits of these combustible gases, 4.65% for H_2 and 15.5% for CO. Given that the film-cooler injection air flow rate averaged 7 scfm (see Figure 5.5) and the total off-gas flow rate averaged 10 scfm, which includes ~0.7 scfm of steam, the off-gas concentrations of these gases are clearly below individual or combined flammability limits even without the benefit of film-cooler dilution.

Table 6.1. Unquenched Melter Off-Gas Composition

	Post Film-Cooler Concentration (ppm)							Flw Rate
Off-Gas Statistics	**He**	**H_2**	**O_2**	**N_2**	**CO_2**	**NO**	**NO_2**	**(scfm)**
Sugar (g/L): 0								
Average	5,580	0	192,000	748,000	1,670	55	4	12.7
Std Dev	238	2	5,520	16,500	598	21	4	0.6
Minimum	5,010	0	172,000	700,000	743	17	0	11.7
Maximum	6,040	10	198,000	791,000	3,280	97	15	14.1
Sugar (g/L): 9.0								
Average	7,420	26	198,000	780,000	2,600	3	3	9.6
Std Dev	719	31	3,640	8,550	723	3	2	0.9
Minimum	6,010	0	180,000	737,000	1,310	0	0	8.1
Maximum	8,680	182	204,000	796,000	5,120	15	9	11.7
Sugar (g/L): 9.0								
Average	8,280	14	202,000	753,000	2,230	2	5	8.6
Std Dev	606	21	7,570	33,500	836	2	3	0.6
Minimum	6,740	0	186,000	703,000	438	0	0	7.3
Maximum	9,620	108	220,000	846,000	4,330	14	14	10.5
Sugar (g/L): 7.5								
Average	7,980	1	198,000	747,000	2,270	4	3	8.9
Std Dev	468	5	8,890	22,200	645	4	3	0.5
Minimum	6,440	0	180,000	685,000	493	0	0	8.0
Maximum	8,850	29	219,000	799,000	4,610	22	25	11.0
Sugar (g/L): 7.5								
Average	7,810	0	208,000	824,000	2,250	2	2	9.1
Std Dev	598	0	4,730	14,500	700	3	2	0.8
Minimum	4,680	0	199,000	782,000	411	0	0	7.7
Maximum	9,200	0	229,000	863,000	4,850	16	8	15.1
Sugar (g/L): 0								
Average	6,480	0	184,000	720,000	2,130	61	6	11
Std Dev	694	0	15,300	69,800	954	40	6	1
Minimum	5,060	0	154,000	581,000	0	0	0	9
Maximum	8,110	0	218,000	876,000	4,030	165	33	14

Table 6.1 (Contd)

Off-Gas Statistics	Post Film-Cooler Concentration (ppm)							Flw Rate
	He	H_2	O_2	N_2	CO_2	NO	NO_2	(scfm)
Sugar (g/L): 2.5								
Average	5,750	0	183,000	703,000	1,570	8	2	13
Std Dev	748	0	15,600	65,600	746	7	6	2
Minimum	3,210	0	153,000	579,000	0	0	0	10
Maximum	7,060	0	211,000	821,000	5,370	36	56	22
Sugar (g/L): 5.0								
Average	5,510	0	189,000	743,000	1,500	6	1	13
Std Dev	409	0	4,710	18,500	1,050	13	2	1
Minimum	4,980	0	182,000	711,000	510	0	0	11
Maximum	6,620	0	201,000	791,000	4,810	57	5	14
Sugar (g/L): 0- 9								
Average	7,350	7	196,000	756,000	2,180	10	3	10
Std Dev	1,100	19	12,300	50,700	813	22	4	2
Minimum	3,210	0	153,000	579,000	0	0	0	7
Maximum	9,620	182	229,000	876,000	5,370	165	56	22

The time-dependent behavior of gaseous process effluent emissions was recorded at nominally 5-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 and changes in film-cooler injection rates as well as during feed sampling and/or feed system repair. Figure 6.1 graphically presents the temporal behavior of melter off-gas effluents on a daily basis.

The only other noteworthy point to make regarding gaseous effluent behavior that has not already been discussed is the apparent effectiveness of even small amounts of sugar upon the melter's NO_x source term. Only during the processing of feeds containing no sugar is NO a significant byproduct of melter nitrate destruction. These data suggest that the sugar-feed additive is effectively reducing the nitrate feed component to N_2. Throughout RSM testing, NO_2 or N_2O (undetected, <2.5ppm) were found to be unimportant contributors to the melter's overall nitrogen oxide source term.

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.

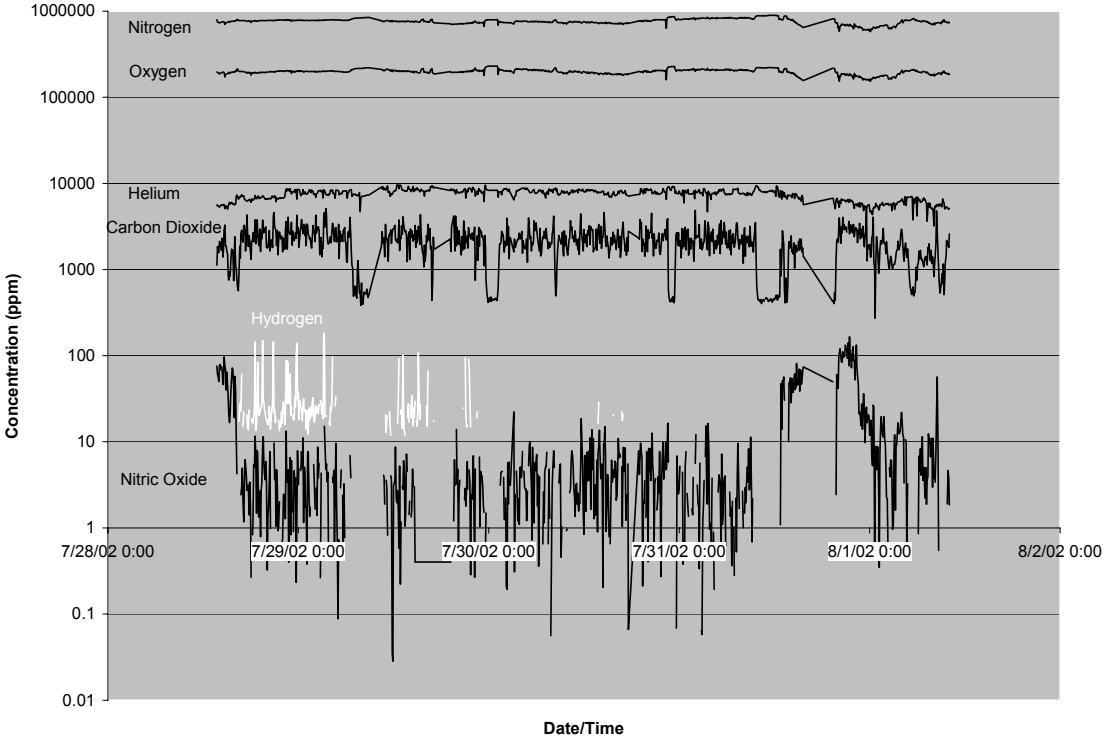


Figure 6.1. Temporal Behavior of Major Process Effluent Gases

Entrained aerosols typically have a mass median diameter of » 1 µm and are compositionally similar to the feed. Consequently, entrainment losses, to first approximation, will influence all feed components in the same way. Feed constituents that fume at melter vitrification temperatures, 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 only capable of forming refractory compounds. Effluent loss rates are traditionally expressed in terms of equipment decontamination factors or 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 6.2 for each distinct sample taken during RSM testing. All sampling data were taken during the processing of melter feed containing 7.5 g of sugar/L of waste. These melter aerosol mass DFs represent a 0.1% to 0.2% mass partitioning to the off-gas system, which is

entirely consistent with previous small-scale melter flowsheet tests and larger scale expectations. Also listed in this table are related, melter DFs and off-gas aerosol loading values. The internal consistency of these off-gas loading and DF data suggest that steady-state melter processing conditions appear to have been nominally achieved and maintained during all sampling periods.

It should be noted that quasi isokinetic sampling conditions were achieved during RSM particulate sampling campaigns. Consequently, a fairly representative sample of off-gas aerosols should have been collected by the sampling filters. However, a larger sampling nozzle was employed after the first sampling campaign to allow increased sampling flow rates to be used. The relative proportions of fuming to entrained effluents will be examined in the following section.

Table 6.2. Gross Melter Aerosol Emission Characteristics

	Feed		Sampling		MOG Flw	Aerosol Catch		Melter	
Date/Time	Sugar (g/L)	Rate (l/h)	Time (m)	Flw (slpm)	(scfm)	Mass (g)	Con (mg/L)	DF	Loss%
7/31/02 2:21	7.5	1.40	58	7.5	8.8	0.0116	0.047	1050	0.095
7/31/02 5:00	7.5	0.89	65	7.5	8.9	0.0492	0.100	450	0.222
7/31/02 7:16	7.5	0.97	79	7.5	9.2	0.0313	0.052	828	0.121

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 the oxide feed target values shown in Table 6.3 clearly illustrates the unmistakable influence of volatility upon the melter-emission source term. In each sample, the concentration of all the classic semivolatiles (B, Cd, Cr, alkali earths, etc.) are seen to be significantly enriched over their nominal feed-composition values. Non-fuming components, such as Al, Fe, Mn, etc., are present at much lower concentrations, while the anomalous values for Ba, Mg, and S are due to impurities that affected the actual feed composition as discussed in Section 4.4. It should be noted that the Si and Na could not be quantified because of quartz filter matrix interferences—analysis included dissolving both the aerosol sample and the filter containing it. Since Si and Na contributions were excluded, the oxide composition of all other components should be biased high. Furthermore, the analytical bias discussed in Section 4.5.2 is responsible for the low Sr (negative deviation) value. The general behavior of the effluents listed in Table 6.3 are in total conformity with generalized LFCM effluent-emission expectations developed from past melter-source-term characterization studies.

Table 6.3. Oxide Composition of Melter Generated Aerosols and Melter Feed

	Off-Gas Aerosol Sample Wt%				Target	
Oxide	**Filter #1**	**Filter #2**	**Filter #3**	**Average**	**Wt%**	**%Dev**
Ag_2O	2.310	1.650	1.890	1.950	0.276	606
Al_2O_3	12.400	13.400	9.210	11.700	12.700	-8
B_2O_3	34.400	30.900	29.200	31.500	12.100	161
BaO	0.545	0.371	0.452	0.456	0.078	485
CaO	5.360	2.510	3.360	3.740	0.840	346
CdO	2.030	3.960	3.470	3.150	0.054	5,740
Cr_2O_3	0.817	0.657	0.838	0.771	0.192	302
Cs_2O	1.630	2.500	2.730	2.290	0.120	1,810
Fe_2O_3	14.100	20.700	20.600	18.500	13.800	34
K_2O	5.230	1.790	1.830	2.950	0.030	9,750
Li_2O	6.670	1.760	2.410	3.610	1.080	234
MgO	3.770	1.900	2.510	2.730	0.258	959
MnO	4.570	7.390	6.670	6.210	5.220	19
MoO_3	1.510	0.479	0.567	0.854	----	----
NiO	1.760	0.850	1.230	1.280	0.180	611
PbO	----	1.450	1.700	1.570	0.252	524
SO_3	----	4.490	5.540	5.010	0.030	16,600
SrO	1.780	2.890	4.990	3.220	8.640	-63
ZrO_2	0.938	0.344	0.747	0.676	0.138	390

Using actual feed-composition and physical-property information provided earlier in Section 4, melter DFs associated with aerosol loss for individual elements can be calculated for the constituents listed in Table 6.3. These derived DF values are tabulated in Table 6.4. 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.

The reproducibility of melter-feed component DFs is, overall, quite good. Furthermore, the magnitudes of the DF values listed in Table 6.4 are well within expectations and are quite representative of average melter-performance behavior.

Table 6.4. Elemental Melter DFs Associated With Aerosol Emissions

	Melter Aerosol DF From Filter#			
Element	**F-01-GE-01**	**F-01-GE-02**	**F-01-GE-03**	**Ave[(a)]**
Ag	295	281	444	326
Al	2,220	1,400	3,670	2,090
B	828	631	1,200	828
Ba	502	504	744	564
Ca	360	526	708	493
Cd	48	17	35	28
Cr	251	213	301	250
Cs	115	51	84	75
Fe	1,720	801	1,450	1,190
K	110	220	387	185
Li	367	954	1,250	656
Mg	369	500	682	486
Mn	2,000	845	1,690	1,320
Ni	169	239	297	223
Pb	----	326	502	395
S	----	1,610	2,340	1,910
Sr	8,860	3,740	3,900	4,710
Zr	407	758	629	559
(a) Calculated from corresponding partitioning factors.				

6.2.3 Volatile Partitioning and Total Elemental DFs

Since only a very few feed components are lost to the off-gas processing system in the gaseous state, essentially all the aerosol performance values listed in Table 6.4 also represent total melter DFs for these elements. Notable exceptions to this statement include B, Cl, F, N, P, and S, whose volatility usually dominate melter off-gas system losses and determine their melter DFs. Table 6.5 summarizes the composition of the off-gas sampler's condensate fraction that is designed to condense (0°C) and/or remove melter (gaseous) effluents penetrating the upstream aerosol filter of the differential sampling system previously described (see Figure 3.4). With the exception of B, Cl, F, S, $C_2O_4^{-2}$, and nitrogen oxy-anions, these data show little evidence of other gas-phase effluents. The other cations in these condensate solutions most likely reflect normal blank or background values.

As was done for particulate matter, volatile melter DFs, which are partial DFs relating to only volatile melter losses, can be derived by combining the meaningful condensate data with actual feed-compositional and physical-property information previously discussed in Section 4.4. These volatile DFs are summarized in Table 6.6.

Table 6.5. Off-Gas Sampler Condensate Solution Composition (ppm)

	Sampler Condensate Composition (ppm)		
Analyte	**Sample #1**	**Sample #2**	**Sample #3**
Ag	0.08	0.073	0.033
Al	0.57	0.16	0.13
B	37	58	73
Ba	0.02	0.0067	0.0
Ca	4.4	1.4	1.7
Co	----	0.02	0.01
Cs	0.00074	0.00048	0.00052
Cu	0.12	0.023	0.024
Fe	0.11	0.013	----
K	1.2	0.39	0.26
Mg	0.62	0.32	0.37
Mn	0.04	0.07	0.017
Na	10	2.2	0.7
Ni	----	0.078	0.16
S	1.3	34	26
Si	0.067	0.62	0.6
Sr	----	0.011	0.011
Ti	----	0.01	
Zn	0.23	0.066	0.032
Br^-	< 1	< 1	< 1
Cl^-	0.98	1.5	2.3
F^-	12.9	8	15.8
NO_2^-	19	25	18
NO_3^-	24	3.8	3.4
PO_4^-	< 3	< 3	< 3
SO_4^{-2}	40	120	830
$C_2O_4^{-2}$	4.5	< 1.7	< 1.7

Table 6.6. Volatile Melter DFs for Boron, Halogen, and Sulfur Feed Constituents

	Melter Volatile DF From Condensate#			
Element	**F-01-Cond-01**	**F-01-Cond-02**	**F-01-Cond-03**	**Ave[(a)]**
B	735	469	372	485
Cl	46	30	20	28
F	8	13	7	9
S	9,970	381	498	634
(a) Derived from corresponding partitioning factors.				

By combining the aerosol-filter and corresponding condensate-sampler fractions for condensed and condensable effluents, total melter DFs for all feed constituents detected in the process exhaust can be derived. Table 6.7 summarizes total elemental melter DFs measured during the processing of C-106/AY-102 with 7.5 g of sugar/L of waste. 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. Volatility contributions to the melter-effluent source term were only significant for boron, the halogens, and sulfur; all other total DFs are nominally equivalent to their particulate values. These element-specific, total DFs, like their corresponding particulate values, are reasonably close to general expectations and are with few exceptions consistent with previous RSM melter-testing results.

Estimates of total melter, elemental DFs can also be extracted from off-gas system, secondary waste stream composition data, provided the volatiles are efficiently captured by emission-abatement equipment and that the resultant equipment effluent steams can be representatively sampled. These estimates will be discussed in the following Section.

Table 6.7. Total Individual Elemental Melter DF Values

	Total Melter DF @ 7.5 g-Sugar/L			
Element	**Smp#1**	**Smp#2**	**Smp#3**	**Ave[(a)]**
Ag	291	278	441	323
Al	2,150	1,400	3,630	2,060
B	389	269	284	306
Ba	495	501	740	559
Ca	262	448	551	382
Cd	48	17	35	28
Cl	46	30	20	28
Cr	251	213	301	250
Cs	114	51	84	75
F	8	13	7	9
Fe	1,710	800	1,450	1,190
K	101	208	363	172
Li	367	954	1,250	656
Mg	339	470	621	449
Mn	1,990	842	1,680	1,310
Ni	169	233	278	217
Pb	----	326	502	395
S	----	308	411	352
Sr	8,860	3,740	3,900	4,710
Zr	252	469	389	346
(a) Derived from corresponding partitioning factors				

6.2.4 Process-Waste-Stream Composition

The RSM's off-gas system, which is composed of an EVS quench scrubber, a high efficiency mist eliminator (deep bed regenerable filter), and a HEPA filter, acts like a multi-component sampler for process aerosols and condensable and/or reactive effluent gases (see Section 3.3). The EVS function is to

quench and condition the hot melter exhaust stream for subsequent cleanup stages. As such, it removes entrained (large-diameter) debris and condenses steam and other chemically reactive and/or condensable gases that 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. The HEME serves to efficiently demist the EVS's exhaust stream and to remove small diameter aerosols (<1 μm) that successfully penetrate the upstream EVS. The HEPA is a polishing filter that removes any remaining off-gas aerosols before the off-gas is released to the environment.

An aqueous secondary waste stream is generated by both the EVS and HEME off-gas processing devices. In the case of the EVS, both soluble and insoluble matter that accumulate within this device's condensate tank are readily collectable. The waste stream generated by the HEME, on the other hand, is composed primarily of soluble effluent species that dissolve in, usually, a highly acidic oxidizing aqueous media. Because of its extremely high surface area, the HEME provides efficient contacting between acid gasses (NO_x, SO_x, halogens, etc.) and the mist-generated aqueous run-off. Insoluble solids collected on the HEME are not directly recoverable as is the case with the HEPA filter, which is itself a secondary waste. Consequently, overall melter DFs can be approximated from the compositions and flow rates of the secondary waste streams generated by the EVS and HEME.

Although undissolved solids (UDS) were not collected from the EVS's condensate tank until the RSM test was complete, EVS supernatant was sampled throughout the duration of melter flowsheet testing. The time-dependent composition of this SBS scrubbing liquor during RSM testing is summarized in Table 6.8. The fact that only nominally soluble species (halogens, alkali-earths boron, etc.) exhibit a strongly increasing concentration while insolubles (Fe, Mg, Mn, Si, etc.) do not is a reflection of the absence of UDS in these samples. High concentrations of Na in the initial scrubbing liquor and in subsequently collected UDS are responsible for its nonsystematic behavior of this feed constituent in these EVS supernatant samples.

Table 6.9 contains the time-dependent behavior of the HEME's secondary waste stream. The concentration of all waste-stream components are flat or only slowly increasing, indicating little holdup of captured, soluble effluents. Since an initial blank sample cannot be taken from this device, it is assumed that the constituents eluted are associated with the current flowsheet test. The HEME filter element was soaked in a nitric acid bath twice and rinsed with water once prior to the C-106/AY-102 test in order to purge materials accumulated during previous testing and thus minimize the potential for stream contamination.

To complete the characterization of the EVS's waste stream, all undissolved solids were harvested from the condensate tank at the conclusion of testing. These solids were dried weighed and mill-blended before being sent to the analytical laboratory. The off-gas system was also disassembled, and all pipe deposits were collected and treated as discussed above. Significant pipe deposits were only found in pipe sections between the melter and the EVS quench scrubber. The compositions of the above described UDS and pipe deposits are summarized in Table 6.10.

Comparing the compositions of the EVS's UDS and the pipe deposits, it is fairly clear that the off-gas deposits are representative of material ordinarily collected by the EVS. Consequently, the pipe deposits

are found to have higher concentrations of nominally soluble constituent while insoluble constituents appear comparable in both samples.

Using these waste-stream compositions, volumes and/or masses with melter-feed-composition physical properties and processing-history information, overall average melter DFs can be derived for the C-106/AY-102 flowsheet test. Table 6.11 presents these DF approximations and compares them to the reference average values derived from the off-gas sampling campaigns. These data show that both off-gas sampling and secondary waste stream derived DF values exhibit the same relative trends for related groups of elements, and are of comparable magnitudes for most corresponding waste constituents. Not surprisingly, the greatest differences between these data sets are associated with volatile constituents (the halogens) that present unique and significant collection difficulties.

Table 6.8. EVS Condensate Soluble Effluent Composition

	Composition of EVS Supernatant Samples (ppm)						
Element	**RSM-F1-1**	**RSM-F1-9**	**RSM-F1-18**	**RSM-F1-33**	**RSM-F1-42**	**RSM-F1-54**	**RSM-F1-64**
Ag	0.04	0.37	1.0	1.8	2.1	2.0	2.4
Al	0.96	0.21	0.18	0.18	0.17	0.20	0.20
B	3	24	43	63	74	93	100
Ba	0.062	0.11	0.12	0.14	0.16	0.16	0.18
Ca	2.2	21	20	19	18	18	19
Cd	----	0.49	0.82	0.97	0.93	0.73	0.68
Cl	7.5	14	18	23	26	32	34
Cr	0.42	0.32	0.31	0.32	0.30	0.51	0.60
Cs	0.078	0.60	1.1	1.7	1.9	2.4	2.8
Cu	0.83	0.81	0.026	0.024	0.030	0.016	0.016
F	4.3	8.1	13	19	21	22	28
Fe	0.44	0.088	0.004	0.010	0.016	0..026	0.034
K	2.9	4.9	5.3	5.3	5.0	5.4	5.5
Li	0.4	1.0	1.5	1.9	2.2	2.6	3.0
Mg	5.4	5.4	5.3	5.3	5.6	6.0	6.3
Mn	0.15	0.18	0.16	0.18	0.19	0.25	0.28
Mo	----	----	----	----	0.080	0.085	0.094
Na	450	420	410	390	370	380	380
Ni	0.34	0.33	0.26	0.23	0.2	0.22	0.21
S	4.1	17	23	29	32	37	41
Si	6.0	6.3	6.3	6.7	7.4	8.2	9.1
Sr	0.53	3.7	5.6	6.7	9.6	12	13
Ti	0.01	0.01	0.01	0.01	0.01	0.01	0.01
V	0.050	0.020	0.010	0.010	----	0.010	0.012
Zn	0.14	0.026	0.006	----	----	----	----

Table 6.9. HEME Waste Stream Composition For Soluble Effluent

	Composition of HEME Aqueous Waste Stream Samples (ppm)				
Element	**RSM-F1-11**	**RSM-F1-29**	**RSM-F1-43**	**RSM-F1-60**	**RSM-F1-65**
Ag	----	0.33	0.23	0.17	0.13
Al	67	88	87	90	94
B	48	61	64	67	65
Ba	6.3	8.3	9.0	8.8	8.7
Ca	100	130	140	140	130
Cd	0.11	0.12	0.16	0.23	0.28
Cl	15	13	15	16	18
Cr	1.2	1.2	1.2	1.1	1.1
Cs	0.41	0.43	0.54	0.68	0.73
Cu	8.3	11	11	11	11
F	4.6	2.2	2.7	3.4	4.3
Fe	4.4	4.8	4.9	5.3	4.6
K	26	36	42	43	42
Li	1.8	2.9	3.4	4.1	3.3
Mg	62	82	87	87	82
Mn	0.21	0.20	0.21	0.22	0.21
Na	1300	1700	1800	1800	1800
Ni	0.91	0.88	0.91	0.89	1.0
P	2.4	3.0	3.0	2.9	2.7
Si	28	34	31	35	34
Sr	0.25	0.27	0.28	0.27	0.27
Ti	0.15	0.13	0.11	0.12	0.11
V	0.42	0.46	0.49	0.49	0.48
Zn	4.9	5.6	5.7	5.5	5.3

Table 6.10. Composition of EVS Undissolved Solids and Off-Gas Pipe Deposits

Element	Off-Gas Solids Wt%		Element	Off-Gas Solids Wt%	
	EVS	Pipe		EVS	Pipe
Ag	0.22	0.24	Li	0.12	0.52
Al	4.8	6.1	Mg	0.17	0.28
B	1.6	3.7	Mn	3.4	2.9
Ba	0.076	0.10	Mo	0.07	----
Ca	0.70	0.58	Na	5.8	6.4
Cd	0.80	0.21	Ni	0.21	0.19
Cl	0.340	4.00	P	0.19	0.19
Co	0.036	0.027	Pb	0.39	0.29
Cr	0.35	0.28	S	0.93	0.99
Cs	0.057	0.21	Si	17	13
Cu	0.088	0.011	Sr	1.0	2.2
F	0.257	1.690	Ti	0.037	0.03
Fe	8.8	7.2	V	0.02	0.03
K	0.28	0.57	Zn	0.028	0.043
La	0.089	0.080	Zr	0.097	0.14

Table 6.11. Off-Gas Sampler and Waste-Stream Total Melter DF Values

Element	Melter DF		Element	Melter DF	
	Off-Gas Sampler	2nd Wst Streams		Off-Gas Sampler	2nd Wst Streams
Ag	323	394	Li	656	581
Al	2,060	1,110	Mg	449	547
B	306	169	Mn	1,310	902
Ba	559	746	Na	----	173
Ca	382	132	Ni	217	474
Cd	28	47	P	----	287
Cl	28	1	Pb	395	747
Cr	250	152	S	352	188
Cs	75	111	Si	----	836
F	9	2	Sr	4,710	1,650
Fe	1,190	860	Ti	----	659
K	172	154	Zr	346	693
La	----	863			

The agreement between these DF data sets suggests that relatively stable operating conditions prevailed throughout most phases of this melter-flowsheet evaluation study. The element-specific DFs recorded during C-106/AY-102 testing are all reasonably close to general expectations. Moreover, the agreement between off-gas sampling results, taken during an 8-h period with feed only containing a single sugar loading, and cumulative overall test DF results suggests that sugar loading in the range used during the current test does not strongly affect the melter off-gas source term. The properties and magnitudes of the secondary waste stream discussed above are summarized in Table 6.12.

Table 6.12. Secondary Waste Stream Physical Properties

Waste Stream	Quantity	pH
EVS Supernatant (L)	122.0	8
EVS UDS (g)	41.6	----
HEME Volume (cc)	1530.0	1
Pipe Deposits (g)	18.4	----

7.0 Byproducts, Residuals, Mass Balance, and Volume Reduction

Beyond the analytical characterization of feed, glass, off-gas effluents, and process-system waste streams previously discussed, a physical accounting of off-gas pipe deposits, process products, and off-gas generated waste streams has also been conducted. The results of these evaluations as well as an overall process summary assessment are discussed below.

7.1 Off-Gas Line Deposits

At the conclusion of RSM testing, the off-gas line from the melter to the EVS was disassembled, inspected, and sampled. Figure 7.1 is a schematic of this segmented off-gas jumper that may be useful in providing perspective for understanding the interrelationship of these pipe segments and the samples obtained from them. As discussed briefly in Section 6.2.4, the cumulative accumulations within this off-gas jumper was quite small. The manner in which the deposits were distributed within this jumper is summarized in Table 7.1.

Although there was some buildup of condensed matter at the film-cooler inlet, the greatest accumulation of deposited material occurred in the 90° elbow where physical impaction enhanced overall losses of entrained debris. Settling in the 8-ft-long horizontal off-gas line segment accounted for only about a half of material collected by the previously mentioned elbow. All other pipe accumulation sites were not significant. Although inlet and outlet photographs of the off-gas jumper pipe segments were taken, they were of little qualitative value since visible accumulations did not influence the internal geometry of the pipe sections. In total, only about 18.4 g of material accumulated within the entire run of this off-gas line jumper over the ~ 96-h processing period.

As mentioned earlier, all harvested pipe deposits were homogenized and analyzed as a single sample. These analytical data were summarized and previously discussed in Section 6.2.4.

Table 7.1. Distribution of Melter Off-Gas Line Deposits

Pipe Segment	Deposit Mass	
	(g)	(%)
Film-Cooler	5.3	29
Elbow	8.8	48
Horizontal	4.0	22
Reducer	0.3	2
Total	18.4	

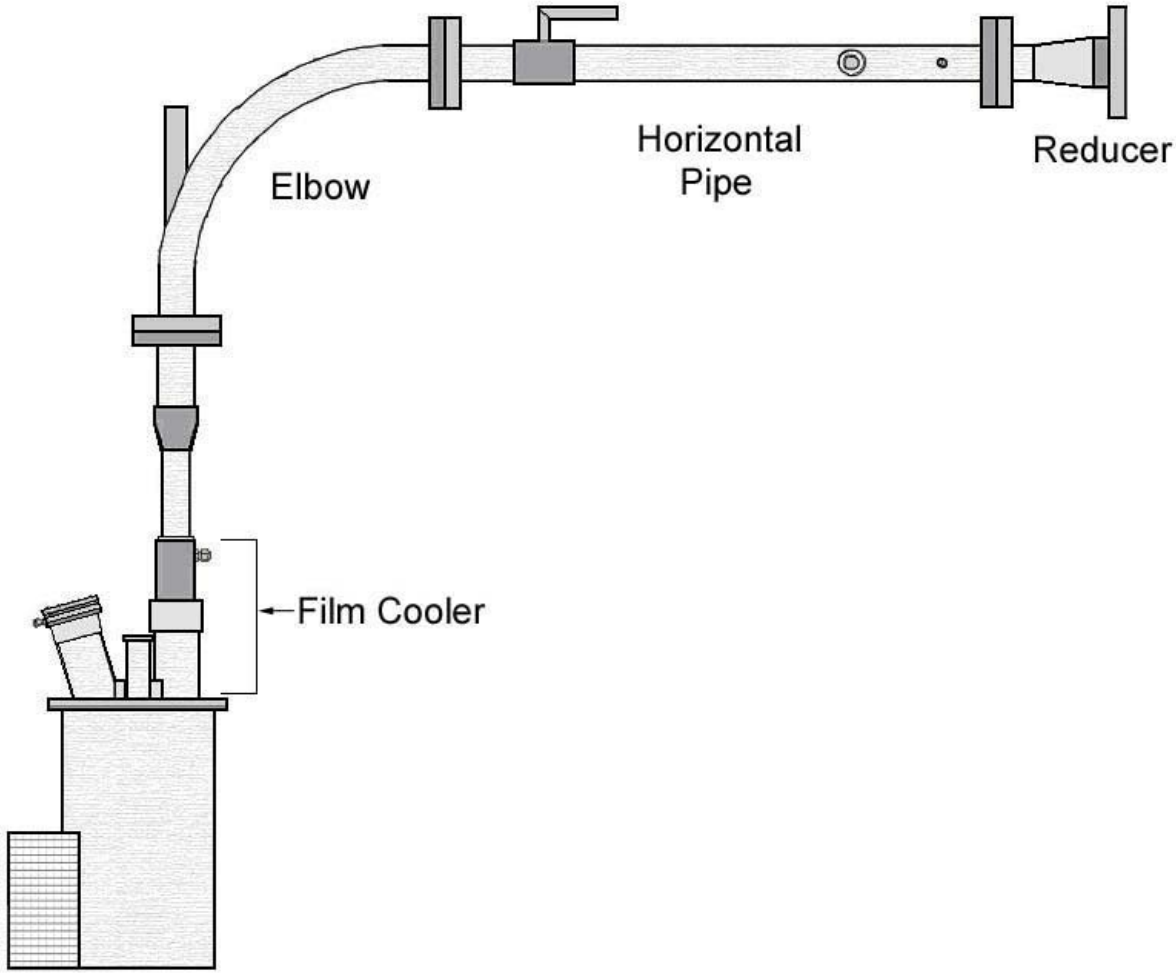


Figure 7.1. Melter Off-gas Jumper Configuration and Sample Site Locations

7.2 Process Mass Balance

The compositional data of process streams previously discussed and summarized were combined in an attempt to fully characterize the C-106/AY-102 vitrification process flowsheet. Analytical feed compositional data were used whenever possible or reasonable; otherwise, target values were employed. In addition, averaged PNNL and SRS glass-compositional data were used throughout this partitioning evaluation, except as noted. The mass-balance results for the overall test are summarized in Table 7.2. Recognizing the limitations imposed by analytical uncertainties, very reasonable mass closure is demonstrated for most of the feed constituents for which complete analytical data exist. The partial data existing for the volatile halogen feed constituents suggests an overall DF of ~ 2 for F, as discussed in Section 6.2.4, and an apparent accounting problem for Cl. Since the feed compositional data for chlorine represent only the soluble fraction of this element, the apparent over recovery of this element suggested by Table 7.2 is a reflection of the lower bound feed-concentration value used in this evaluation.

Table 7.2. Stream-Dependent %-Partitioning of C-106/AY-102 Melter Feed Constituents

			EVS				
Element	**Glass**	**Pipe Dep.**	**Solution**	**Solids**	**HEME**	**Total**	**Var (%)**
Ag	86.800	0.027	0.057	0.170	0.000	87.1	-12.9
Al	90.500	0.031	0.055	----	0.005	90.5	-9.5
B	94.100	0.031	0.029	0.526	0.006	94.7	-5.3
Ba	104.000	0.031	0.051	0.024	0.028	105.0	5.0
Ca	97.100	0.031	0.084	0.571	0.075	97.8	-2.2
Cd[(a)]	95.500	0.182	1.540	0.398	0.002	97.6	-2.4
Ce	98.800	----	----	----	----	98.8	-1.2
Cl	----	19.800	3.800	114.000	0.935	138.0	38.0
Cr	108.000	0.147	0.407	0.098	0.006	108.0	8.0
Cs	100.000	0.086	0.054	0.756	0.003	101.0	1.0
F	----	3.640	1.250	39.900	0.096	44.9	-55.1
Fe	102.000	0.031	0.085	----	0.000	102.0	2.0
K	80.100	0.087	0.095	0.400	0.068	80.8	-19.2
La	125.000	0.033	0.083	----	----	125.0	25.0
Li	103.000	0.033	0.018	0.119	0.002	103.0	3.0
Mg	93.700	0.025	0.033	0.047	0.077	93.9	-6.1
Mn	96.200	0.030	0.080	0.001	0.000	96.3	-3.7
Na[(b)]	96.700	0.031	0.065	0.387	0.095	97.3	-2.7
Ni	103.000	0.059	0.149	----	0.003	103.0	3.0
P	98.800	0.102	0.230	----	0.016	99.2	-0.8
Pb	72.100	0.033	0.101	----	----	72.2	-27.8
S	92.500	0.017	0.036	0.480	----	93.0	-7.0
Si	99.200	0.029	0.083	0.007	0.001	99.4	-0.6
Sr[(c)]	98.800	0.012	0.013	0.036	0.000	98.9	-1.1
Ti	98.800	0.038	0.111	0.002	0.002	99.0	-1.0
Zr[(c)]	96.200	0.055	0.089	----	----	96.4	-3.6

(a) Average PNNL glass value used
(b) Target feed composition value used
(c) Average SRS glass value used.

The other elements exhibiting poor mass closure but possessing complete analytical results include K, La, and Pb. Demonstrated (Section 4.5.2) analytical difficulties are responsible for the poor K mass balance results, and the highly variant waste, feed, and glass-compositional results obtained from La and Pb also suggest significant analytical limitations for these elements.

As discussed above, the Table 7.2 mass balance evaluation utilized all available empirical analytical data except in a few instances, which will now be described. For reasons discussed in Section 4.5.2, averaged compositional glass values were not used for Sr and Zr due to the demonstrated PNNL analytical bias associated with these elements. For similar reasons, only the averaged PNNL glass-compositional value was used for Cd partitioning evaluations. Due to incomplete potassium fusions of

C-106/AY-102 feed samples previously discussed in Section 4.4, the target feed-compositional value for Na was used in place of the biased (low) analytical result.

7.3 Process Statistics

During the July 2002 melter-flowsheet evaluation studies, 100 L (26 gal) of 60 wt% C-106/AY-102 simulated melter feed (~ 90 L-waste), having a total mass of 142 kg, were processed by the RSM, producing 20 L (5.3 gal) of glass having a total mass of 51 kg. Although vitrification results in both mass and volume waste reductions, only the volume-reduction parameter is meaningful since a major mass contributor to the waste (H_2O) is a nonvitrifiable, volatile effluent. On the other hand, since most of the hazardous and rad-waste components can be incorporated and immobilized in the melter's vitreous product and tank-waste volumes are a physical reality, volume reduction has important waste-disposal implications. During the current RSM test, an overall C-106/AY-102 waste-volume reduction factor of 4.6 was achieved.

7.4 Process Summary

The results obtained from C-106/AY-102 melter-flowsheet evaluations have demonstrated that acceptable operating conditions and adequate processing rates can be achieved for the newly formulated, 60 wt% C-106/AY-102 melter feed provided supplemental reductant (>2.5 g of sugar/L of waste) is used to control glass-oxidation conditions. PCT evaluations demonstrated that the newly formulated product glass, at all reductant loadings, was of higher quality than the EA glass to which it was compared. Although TCLP leaching results for Ag were borderline, the glass product was nevertheless sufficiently durable to comply with all existing RCRA land-disposal limits. Spinel formation at 1150°C (0.5 wt%) was found to be lower than laboratory-based expectations (Peeler et al. 2002), and no evidence for crystal stratification or settling in the melter could be found. Moreover, specific glass-production rates and process energy requirements for the C-106/AY-102 flowsheet were found, overall, to be in reasonably good agreement with generalized expectations for slurry-fed, Joule-heated ceramic melters.

Melter partitioning of individual feed constituents, derived from both off-gas sampling and secondary waste-stream analysis, revealed that with the exception of boron, sulfur, and the halogens, essentially all feed constituents (excluding C, N, H_2O, etc.) were found to be primarily in a condensed state downstream of the film cooler. Overall, the element-specific DFs recorded during RSM testing are reasonably close to general expectations and are generally consistent with previous RSM testing results. Indeed, the very reasonable mass closure demonstrated for most of the feed constituents for which complete analytical data exist suggests that the current melter test has successfully characterized the vitrification flowsheet of the optimized, 60 wt% C-106/AY-102 glass formulation.

8.0 References

40 CFR 268. Land Disposal Restrictions. Code of Federal Regulations. U.S. Environmental Protection Agency, Washington D.C.

American Society for Testing and Materials (ASTM). 1997. *Standard Test Methods for Determining Chemical Durability of Nuclear, Hazardous, and Mixed Waste Glasses: The Product Consistency Test (PCT),* ASTM C1285-97, West Conshohoken, PA.

Goles RW, JM Perez, BD MacIsaac, DD Siemer, and JA McCray. 2001. *Test Summary Report: INEEL Sodium-Bearing Waste Vitrification Demonstration RSM-01-1,* PNNL-13522, Pacific Northwest National Laboratory, Richland, WA.

Goles RW, JA Del Debbio, RJ Kirkham, BD MacIsaac, JA McCray, DD Siemer, and NR Soelberg. 2002. *Test Summary Report INEEL Sodium-Bearing Waste Vitrification Demonstration RSM-01-2*, PNNL-13869, Pacific Northwest National Laboratory, Richland, WA.

Jantzen CM, NE Bibler, DC Beam, CL Crawford, and MA Pickett. 1993. *Characterization of the DWPF Environmental Assessment Glass SRM,* WSRC-TR-92-346 REV 3, Savannah River, GA.

Peeler DK, TB Edwards, CC Herman, RJ Workman, IA Reamer, JD Vienna, JV Crum, DE Smith, and DS Kim. 2002. *Development of High Waste Loading Glasses for Advanced Melter Technologies*, WSRC-TR-2002-00426, Westinghouse Savannah River Company, Aiken, SC.

Perez JM, DF Bickford, DE Day, DS Kim, SL Lambert, SL Marra, DK Peeler, DM Strachan, MB Triplett, JD Vienna, and RS Wittman. 2001. High-Level Waste Melter Study Report, PNNL-13582, Pacific Northwest National Laboratory, Richland, WA.

Toxicity Characteristic Leaching Procedure (TCLP). 1992. Test Methods for Evaluating Solid Waste, Volume 1C: Laboratory Manual Physical/Chemical Methods, SW-846, Method 1311, Rev. 2, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington D.C.

Appendix A

Test Plan and Test Instructions for Vitrification Demonstration Tests of C-106/AY-102 Waste Simulant

Appendix A.1: Test Plan for Vitrification Demonstration Tests of C-106/AY-102 Waste Simulant

Tanks Focus Area

Vitrification Flowsheet Evaluation of a High-Capacity Hanford C-106/AY-102 Glass Formulation

PNNL Research-Scale Melter Test Plan – Test No.: RSM-02-1

R.W. Goles

July 2002

Prepared for the U.S. Department of Energy
under Contract DE-AC06-76RL018

APPROVALS

PREPARED BY:

_____ _____
PNNL RW Goles Date

APPROVED BY:

_____ _____
RW Goles, Lead PNNL Investigator Date

_____ _____
LM Peurrung, Line Manager, Advanced Proc. & App. Date

CONCURRENCE:

_____ _____
JM Davis, RSM Equipment Custodian Date

_____ _____
RM Gough, Safety Representative Date

_____ _____
PH Rojas, APEL Building Manager Date

RECEIPT ACKNOWLEDGED:

_____ _____
CR Allen, APEL Building Manager Date

Table of Contents

1.0 **Background** 1

2.0 **Test Objectives** 1

3.0 **Waste, Waste-Simulant, Glass Former and Target Glass Compositions** ... 2
- 3.1 Melter Feed Preparation Description 5

4.0 **Test Objectives** 5

5.0 **Equipment Description** 8

6.0 **Responsibilities** 12

7.0 **Quality Assurance** 12

8.0 **Hazards Assessment** 12
- 8.1 Environmental Releases 13
- 8.2 Flammability 13
- 8.3 Loss of Vacuum 14

9.0 **Personnel Safety** 14
- 9.1 Personnel Protective Equipment 15
- 9.2 Material Safety Data Sheet 15
- 9.3 Medical Requirements 15
- 9.4 Confined Space 15
- 9.5 Respiratory Protection Requirements 15

10.0 **Waste Minimization/Management** 15

11.0 **Associated Procedures, Test Instructions, and Safety Limits** 16

12.0 **Emergency Response** 16

13.0 **References** 16

List of Tables

3.1 Composition Summary for C-106/AY-102 Waste, LAW Pretreatment Products and C-106/AY-102 Simulant 3

3.2 Composition and Preparation Recipe for C-106/AY-102 Simulant 4

3.3 Glass Former Additives and Equivalent Oxide Composition 5

3.4 Target Glass Composition at 65 Wt% Waste Loading 6

4.1 RSM's Effluent Gas Analyzers 7

4.2 Electronically Logged Operational Data 7

4.3 Process Sample Frequency 8

5.1 RSM dimensions and operational features 11

8.1 Maximum RSM Noxious Gas Release Estimates Accompanying Off-Gas System Failure 14

List of Figures

4.1 Multi-Components Melter Off-Gas Sampling System 8

5.1 Schematic of the Research-Scale Melter Processing System 9

5.2 Cross Section View of the Research-Scale Melter 10

1.0 Background

The U.S. Department of Energy (DOE) Headquarters directed the Tanks Focus Area (TFA) to conduct a technical review of alternatives for solidification of high-level waste (HLW) that could achieve major cost reductions with reasonable long-term risks. In Response, the TFA chartered an independent Review Team that collected and analyzed data and performed specific analyses, and recommended a research and development program, as warranted, for future waste form and melter advancements.

The focus of the Review Team effort was on HLW at the Hanford site in Richland, Washington and on evaluating the impacts of modifications to product requirements, modifications of processing constraints potentially through alternate technologies, alternative waste forms and evaluation of alternate melter technologies to increase waste loading and reduce costs.

Based upon the results of the study, the review team addressed whether alternate glasses or glass-ceramic compositions including borosilicate glass could accommodate DOE HLW with greater efficiency, cost savings, and/or lower risks. One of the conclusions drawn from the Review Team evaluation of this subject area was that the borosilicate waste form has performed predictably and is firmly entrenched in the nuclear waste remediation program. However, it was felt that the vitrification process using this waste form has not been optimized.

In order to further the goal of optimizing Hanford's HLW borosilicate flowsheet, a glass formulation effort has been launched to develop an advanced high-capacity waste form exhibiting acceptable leach and crystal formation characteristics. A simulated C-106/AY-102 waste envelop inclusive of LAW pretreatment products was chosen as the subject of these nonradioactive optimization efforts.

To evaluate the processing, off-gas emission, and product glass characteristics of this advanced borosilicate waste formulation under continuous dynamic vitrification conditions, a research-scale Joule-heated ceramic melter will be used to demonstrate the advanced waste form's flowsheet. This test plan describes the research-scale melter (RSM) test to be performed in furtherance of the process characterization goals described above.

2.0 Test Objectives

The primary objective of the current RSM test is to characterize the melter-process flowsheet based upon the Hanford's C-106/AY-102 waste envelope D and an optimized target glass composition. The targeted vitreous-product composition was established from a series of laboratory crucible tests involving C-106/AY-102 surrogate material and suitable glass-forming additives that were chosen to maximize product glass waste loading. Although these crucible tests suggested that the optimized glass formulation with waste loading as high as 60 Wt% could be achieved while maintaining a durable vitreous waste product, continuous feeding and dynamic processing conditions under Joule-heating conditions are needed to validate this finding. 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 flowsheet assessments of processing conditions and glass-product quality, the experimental objectives of the current RSM C-106/AY-102 flowsheet test is to:

- Determine feasibility of vitrifying surrogate C-106/AY-102 at 55 wt% and 60 wt% waste loading using waste loading-dependent glass former compositions to produce a regulatory acceptable borosilicate glass waste form.
- Evaluate the glass products with regard to composition, crystal growth and concentration, phase separation, chemical durability, and oxidation state.
- Characterize the composition and concentration of condensed-phase and selected gaseous melter generated off-gas emissions and the composition of secondary off-gas system waste streams.
- Establish a maximum process rates at each waste loading based on stable operation at a glass temperature of 1150°C and a 95% cold-cap coverage.
- Access whether the melt rate or processing conditions can be optimized by changing reductant and/or melter-operating conditions.
- Measure the volume reduction factor for vitrified surrogate C-106/AY-102 waste stream.
- Quantify specific glass production rates and glass production-rate energy requirements.

3.0 Waste, Waste-Simulant, Glass Former and Target Glass Compositions

Extensive simulant development efforts for the C-106/AY-102 waste envelope have been conducted in support of Hanford's Waste Treatment Plant [1]. The defined C-106/AY-102 waste composition, that includes LAW pretreatment product stream additives, is summarized in Table 3.1, where the concentrations of selected trace components have been appropriately adjusted to accommodate analytical detection sensitivities. The chemicals employed to simulate the physical and chemical characteristics of actual C-106/AY-102 waste are detailed in Table 3.2.

The C-106/AY-102 simulated waste in combination with selected glass formers has been the subject of both research- [2] and pilot-scale [3] melter flowsheet testing. The identity and relative proportions of the baseline chemical glass formers used to produce a regulatory-compliant glass from C-106/AY-102 simulated waste constituents are summarized in Table 3.3. The maximum waste loading used during these previous melter tests was 51-Wt%. The current test involving an optimized glass formulation will use a waste loading of up to 60 Wt%.

Since it will be important to compare the current flowsheet tests using an optimized glass formulation with previous testing results, the current test will use the same simulant and similar glass former components prepared in an identical manner as in previous studies. The chemical forms of the waste and glass former constituents defined by the previous simulant development effort and summarized in Table 3.2 and Table 3.3, respectively, will also be used during the current test. The resultant target glass compositions for the 55wt% and 60 wt% waste loadings to be used during the current test using with waste and glass former compositions summarized in Tables 3.1 and 3.3 is detailed in Table 3.5

Table 3.1 Composition Summary for C-106/AY-102 Waste, LAW Pretreatment Products and C-106/AY-102 Simulant

	C-106/AY-102 Envelope D Waste	Analytical Adjustment to Envelope D (g/100g-Ox)	Pretreatment Products (wt% of Adjusted Envelope D Oxide)	C-106/AY-102 Simulant
Ag_2O	0.59 %			0.46 %
Al_2O_3	26.99 %			20.91 %
As_2O_3				
B_2O_3				
BaO	0.17 %			0.13 %
CaO	1.79 %			1.39 %
CdO	0.11 %			0.09 %
CeO_2	0.08 %			0.06 %
Cl	0.00 %	0.060		0.05 %
Cr_2O_3	0.41 %			0.32 %
Cs_2O	0.01 %		0.25 %	0.20 %
CuO				
F	0.04 %	0.022		0.05 %
Fe_2O_3	29.37 %			22.76 %
K_2O	0.07 %			0.05 %
La_2O_3	0.28 %			0.22 %
Li_2O				
MgO	0.55 %			0.43 %
MnO	1.34 %		9.75 %	8.61 %
Na_2O	20.35 %		0.60 %	16.23 %
NiO	0.39 %			0.30 %
P_2O_5	0.38 %			0.29 %
PbO	0.54 %			0.42 %
PdO	0.01 %			0.01 %
Rh_2O_3	0.01 %			0.01 %
RuO_2	0.03 %			0.02 %
SO_3	0.02 %	0.044		0.05 %
Sb_2O_5				
SeO_2				
SiO_2	14.75 %			11.43 %
SrO	0.05 %		18.34 %	14.26 %
TiO_2	0.10 %			0.08 %
UO_2	1.30 %			1.01 %
ZnO				
ZrO_2	0.29 %			0.23 %
TOTAL	100 %	0.126	28.94 %	100 %
Volatiles (g/100 g oxides)				
CO_3	0.004		36.7	8.240
NO_2	0.003			0.002
NO_3	0.000		4.5	1.013
TOC	1.601			1.240

Table 3.2 Composition and Preparation Recipe for C-106/AY-102 Simulant

Oxide	**C-106/AY-102 Simulant**	Starting **Materials**	Amount in **Simulant (kg)**
Ag_2O	0.46 %	Ag_2O	0.11
Al_2O_3	21.13 %	$Al(OH)_3$	7.83
As_2O_3			
B_2O_3			
BaO	0.13 %	$Ba(OH)_2*8H_2O$	0.06
CaO	1.40 %	$CaCO_3$	0.61
CdO	0.09 %	CdO	0.02
CeO_2	0.06 %	$Ce(OH)_4$	0.02
Cl	0.05 %	NaCl	0.02
Cr_2O_3	0.32 %	$Cr_2O_3*1.5H_2O$	0.09
Cs_2O	0.20 %	CsOH (50% Solution)	0.10
CuO			
F	0.05 %	NaF	0.03
Fe_2O_3	22.99 %	$Fe(OH)_3$ Slurry (13 %)	56.49
K_2O	0.05 %	KNO_3	0.03
La_2O_3	0.22 %	$La(OH)_3*3H_2O$	0.08
Li_2O			
MgO	0.43 %	$4MgCO_3*Mg(OH)_2*5H_2O$	0.25
MnO	8.70 %	MnO_2	2.58
Na_2O	16.40 %	NaOH (50% Solution)	9.82
NiO	0.30 %	$NiOH_2$	0.09
P_2O_5	0.30 %	$FePO_4*xH_2O$	0.15
PbO	0.42 %	$(PbCO_3)_2*Pb(OH)_2$	0.12
Rh_2O_3			
RuO_2			
SO_3	0.05 %	Na_2SO_4	0.02
SiO_2	11.55 %	SiO_2	2.80
SrO	14.41 %	$SrCO_3$	3.73
		$Sr(OH)_2*8H_2O$	2.24
TiO_2	0.08 %	TiO_2	0.02
UO_2			
ZnO			
ZrO_2	0.23 %	$Zr(OH)_4*xH_2O$	0.08
		Water	10.71
TOTAL	100 %		
Volatiles (g/100 g oxides)			
CO_3	8.240		
NO_2	0.002	$NaNO_2$	0.001
NO_3	1.012	$NaNO_3$	0.313
TOC	1.240	$H_2C_2O_4*2H_2O$	1.578
		TOTAL	100

Table 3.3 Glass Former Additives and Equivalent Oxide Composition

	Frit/Glass Former @ %WL			
	Baseline	Advanced		Chemical
Oxide	51 Wt%	55 Wt%	60 Wt%	Additive
B_2O_3	10.00%	6.67%	7.50%	H_3BO_3
K_2O				KOH
Li_2O	1.67%	3.50%		$LiOH*H_2O$
Na_2O		27.27%	32.53%	NaOH
P_2O_5				Na_3PO_4
SiO_2	86.78%	62.57%	59.97%	SiO_2
ZnO	1.55%			ZnO

3.1 Melter Feed Preparation Description

Appropriate amounts of glass formers (Table 3.3) and C-106/AY-102 simulant (Table 3.2) will be batched to provide enough melter feed (~150-liters) to support a nominal 5-day melter test. The glass additive/waste-simulant slurry will be allowed to mix for a minimum of 24 hours before feeding to the melter. During this mixing and reaction period, there is a chance that some reactions may produce small quantities of nitrogen oxide gases, so the melter feed vessel will be vented appropriately.

A 55-liter feed tank will be used to directly feed the melter unless parametric evaluations of reductant concentrations upon processing characteristics are performed. In this eventuality, the melter feed concentration of reductant (sucrose) will be altered in 20-liter day tank vessels that will temporarily become the melter's feed source. 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. However, during the current test, glass oxidation state, as indicated by the Fe^{+2}/Fe_{total} ratio, will be constrained to be $\leq$ 0. 3.

4.0 Test Description

Hanford's C-106/AY-102 envelope D waste simulant will be combined with an optimized glass former and processed over a 5-day period in a research-scale melter at the Pacific Northwest National Laboratory (PNNL). Two different C-106/AY-102 waste loadings , 55 wt% and 60 wt%, will be evaluated during this test period This test will be used to:

- evaluate the processability of optimized C-106/AY-102 glass formulations,
- test the product glass compositions to ensure that acceptable final products are produced,
- characterize the melter off-gas emissions, and
- determine secondary waste stream compositions.

Table 3.4 Target Glass Composition at 65 Wt% Waste Loading

		Waste Loading 55%		Waste Loading 60%	
Oxide	**Simulant Wt%**	**Glass Form Wt% Glass**	**Target Glass Wt%**	**Glass Form Wt% Glass**	**Target Glass Wt%**
Ag_2O	0.46%	----	0.25%	----	0.28%
Al_2O_3	21.12%	----	11.62%	----	12.67%
B_2O_3	----	3.00%	3.00%	3.00%	3.00%
BaO	0.13%	----	0.07%	----	0.08%
CaO	1.40%	----	0.77%	----	0.84%
CdO	0.09%	----	0.05%	----	0.05%
CeO_2	0.06%	----	0.03%	----	0.04%
Cl	0.05%	----	0.03%	----	0.03%
Cr_2O_3	0.32%	----	0.18%	----	0.19%
Cs_2O	0.20%	----	0.11%	----	0.12%
F	0.05%	----	0.03%	----	0.03%
Fe_2O_3	22.98%	----	12.64%	----	13.79%
K_2O	0.05%	----	0.03%	----	0.03%
La_2O_3	0.22%	----	0.12%	----	0.13%
Li_2O	----	1.57%	1.57%	----	0.00%
MgO	0.43%	----	0.24%	----	0.26%
MnO	8.70%	----	4.78%	----	5.22%
Na_2O	16.40%	12.27%	21.29%	13.01%	22.85%
NiO	0.30%	----	0.16%	----	0.18%
P_2O_5	0.30%	----	0.16%	----	0.18%
PbO	0.42%	----	0.23%	----	0.25%
PdO	0.01%	----	0.01%	----	0.01%
SO_3	0.05%	----	0.03%	----	0.03%
SiO_2	11.55%	28.15%	34.51%	23.99%	30.91%
SrO	14.41%	----	7.92%	----	8.64%
TiO_2	0.08%	----	0.04%	----	0.05%
ZrO_2	0.23%	----	0.13%	----	0.14%
TOTAL	100%	45%	100.00%	40%	100.00%

From processing observations, optimum operating conditions (such as melt rate) and process limits will be determined. The product glass will be sampled throughout the test and subjected to the Product Consistency Test (PCT), ASTM C-1285-94[4] which is the standard test method used for determining the durability of high-level waste glasses in the United States. In addition the Toxicity Characteristic Leaching Procedure[5] will be performed for all hazardous metals present in the glass product. Glass samples will also be taken to determine density, crystalline morphology, and viscosity and for archival for future testing.

Melter off-gas source term sampling will also be conducted periodically to establish the nature and magnitudes of melter effluent losses. A multi-component sampling system (see Figure 4.1) will be used to characterize both gaseous and condensed-phase melter exhaust emissions on a

periodic basis. Continuous emission analyzers will also be used to determine the concentrations of the gaseous off-gas constituents summarized in Table 4.1.

Table 4.1 RSM's Effluent Gas Analyzers

Analyzer	Targeted Effluent Gases
Gas Chromatograph	H_2, He, N_2, CO, NO, O_2, CO_2, & N_2O
Nitrogen Oxide	NO & NO_2

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 redox can be determined.

In addition to the observations mentioned above, process data sheets, illustrated in the attached Test Instruction, will be filled in at hour intervals throughout the duration of melter testing. Process data will also be electronically collected for many of the more important operational variables identified in Table 4.2. Sample collection of process samples will be conducted at the specific frequencies summarized in Table 4.3 and manually recorded on Sample Log data sheets, that are illustrated in the attached RSM Test Instruction.

Table 4.2 Electronically Logged Operational Data

Operational Parameter	Units
Melt Temperature	° C
Plenum Temperature	° C
Feed pump setting	%
Electrode Potential	Volts
Electrode Current	Amps
Electrode Power	kW
(Electrode Power) Output	%
(Electrode) Control Mode	A or M
Melt Resistance	Ω
Melt (Electrode) Set point Temp	° C
Kiln Power	kW
Kiln Temp Set point	° C
Kiln Actual (Middle) Temp	° C
Kiln Power Output	%
Kiln Control Mode	A or M
Discharge Can Power	kW
Discharge Can Temp Set point	° C
Discharge Can Actual Temp	° C
Discharge Can Power Output	%
Overflow Heater Power	kW
Overflow Heater Set point	° C
Overflow Heater Power Output	%

Table 4.3 Process Sample Frequency

Sample	Frequency	Quantity
Melter Feed	One per Shift	100 cc
Melter Glass	Two per Shift	Graph Boat
Condensate Tank	One per Shift	100cc
HEME Run-Off	Once per Day	100cc

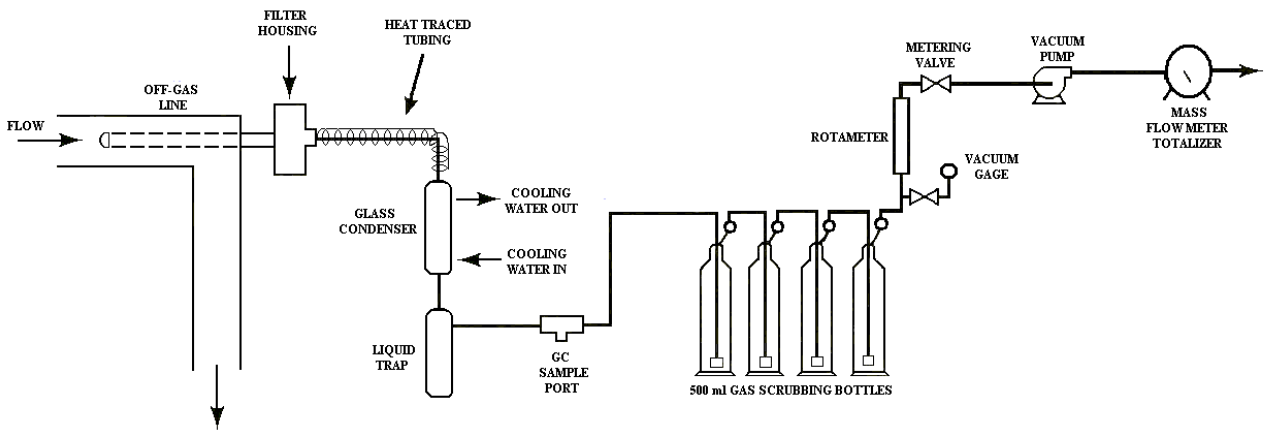


Figure 4.1 Multi-Components Melter Off-Gas Sampling System

At the completion of the test, the melter will be shut down according to PNNL procedures. Any devitrification in the containers of glass produced will be noted. In addition, the melter tank, other melter components, and the off-gas system will be examined for any wear, pitting, or corrosion. Any unusual observations will be documented.

5.0 Equipment Description

The Research Scale Melter is located in the Applied Process Engineering Laboratory (APEL) building in Richland, Washington. Figure 5.1 shows the RSM processing system. Figure 5.2 illustrates the construction of the melter vessel and Table 5.1 documents RSM dimensions and other operational features.

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 composition and the ability to perform 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 55-liter 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.

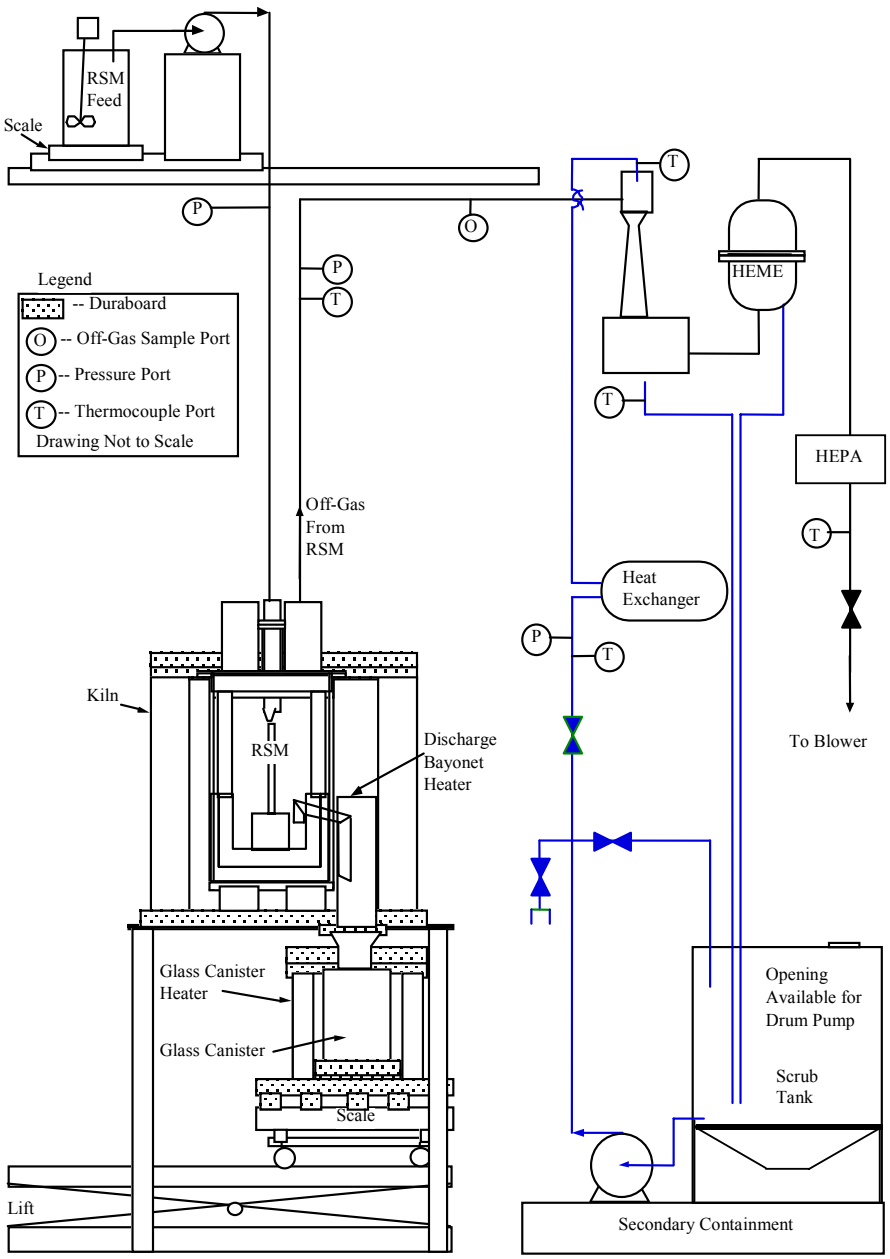


Figure 5.1. Schematic of the Research-Scale Melter Processing System

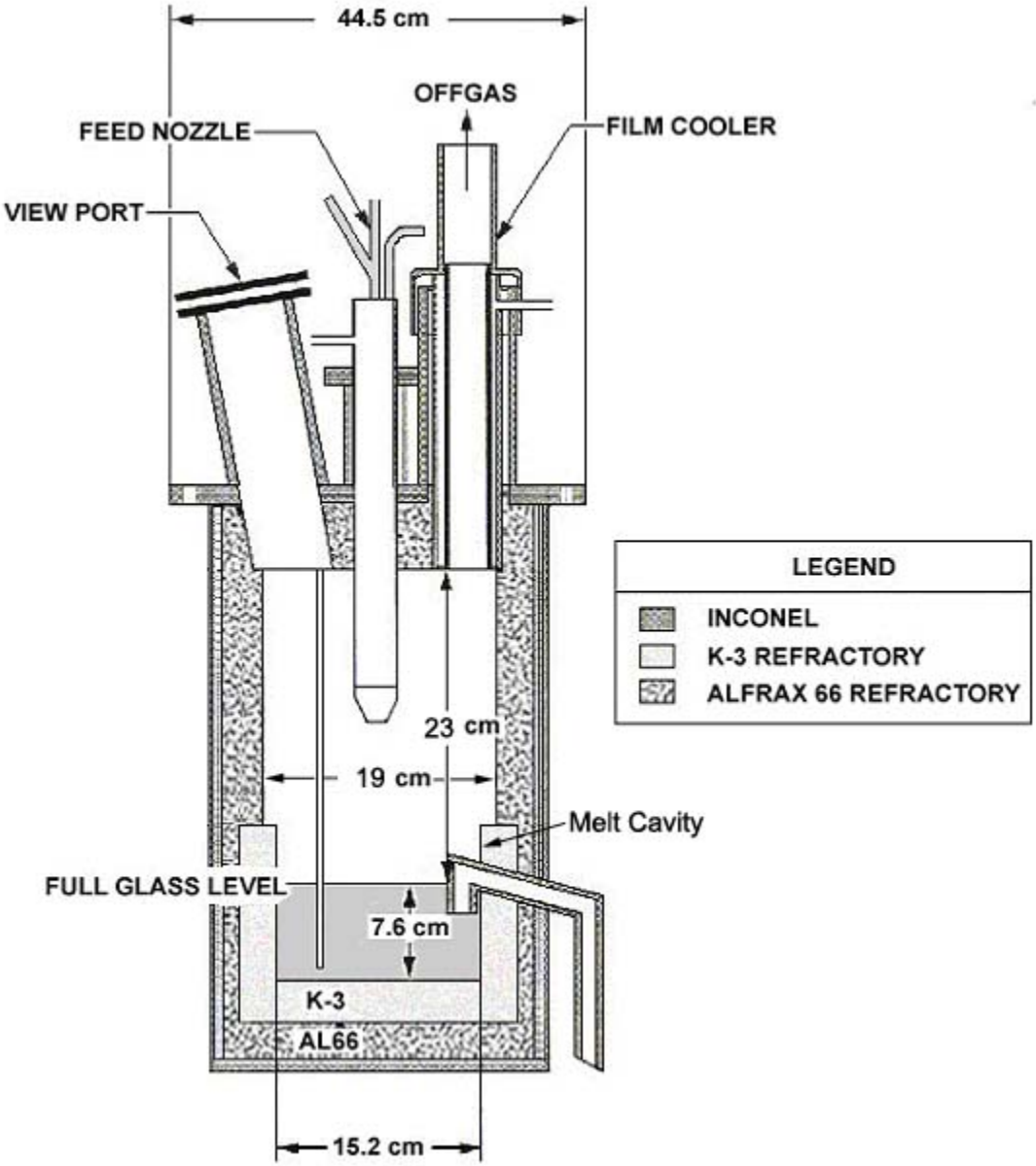


Figure 5.2. Cross-Section View of the Research-Scale Melter (not drawn to scale)

Table 5.1 RSM dimensions and operational features

Parameter	Value
Melt cavity diameter	15 cm
Melt cavity height	11 cm
Melt cavity volume	2.1 L
Glass pool surface area	182 cm^2
Nominal glass depth	7.6 cm
Melter inside volume	1.4 liters
Nominal molten glass mass	3.6 kg
Glass turnover rate @ nominal feed rate 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^2

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 resting on the crucible floor supply joule-heating power to the RSM.

As is shown in Figure 5.1 the RSM's exhaust 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 heater for the discharge section, and the peristaltic pump for the feed system. The data collected includes the voltage and current for major electrical components, temperature at various locations in the system (*e.g.*, molten glass, plenum space in melter, melter kiln, off-gas treatment system), pressure in the melter, and weight of the feed tank. Data are typically archived every minute but are displayed at more frequent intervals to assist the operators.

6.0 Responsibilities

The cognizant PNNL project manager/principal investigator (RW Goles) has the responsibility of preparing this test plan and associated test instructions, and ensuring that the test plan is executed in accordance with the TFA task requirements and applicable quality assurance requirements. The RSM Equipment Custodian (JM Davis) will provide reviews on all procedures and plans to ensure compliance with the RSM SOP. He is responsible for ensuring the safe operation of the RSM equipment.

The cognizant PNNL line manager (L.M. Peurrung) shall review and approve this test plan for technical adequacy, Environmental Health and Safety compliance, and resource availability.

The cognizant safety engineer (R.M. Gough) has the responsibility of reviewing this test plan for compliance with laboratory safety requirements.

The cognizant building manager (P.H. Rojas) has the responsibility of reviewing this test plan for compliance with building regulations and limits.

This document will be peer reviewed by a Senior Scientist, Engineer, or Engineering Associate familiar with the work but not directly involved in the generation of this test plan.

7.0 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. An explanation of any changes should be noted in the LRB. PNNL standard laboratory practices will be followed throughout the testing.

8.0 Hazards Assessment

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.

8.1 Environmental Releases

Worst-case air emissions have been estimated and project staff have worked with PNNL environmental management staff to verify that sufficient controls will be in place to assure that all emissions fall within permit limits. Written documentation of this fact has been placed in the project records documenting projected test compliance with facility permit requirements.

The melter feed stream that will be thermally processed by the RSM contains the reductant oxalic acid. However, due to the low reducing capacity of this reductant, sucrose may also have to be used. Although CO_2 and H_2O will be the dominant thermal byproducts of reductant oxidation, the off-gases generated could possibly contain pyrolytic and incomplete products of combustion such: as CO, H_2, and volatile organic compounds (VOCs). However, the oxidizing conditions expected in the plenum will virtually eliminate the flammability risks posed by pyrolytic byproducts such as H_2, and VOCs. CO, on the other hand, is expected to be present at measurable concentrations.

8.2 Flammability

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

Beyond the use of plenum temperature control of flammables, the RSM test will comply with the NFPA 69 standard. According to NFPA 69, the lower flammable limit (LFL) of a vapor must be maintained below 25% using engineering controls if automatic interlocks based on the flammable gas concentration are not used. This means that to maintain a guaranteed safe condition in the off-gas system, sufficient dilution air must be added in a controlled manner such that the LFL does not exceed 25%. However, the worst-case off-gas system concentration of CO (where all available carbon is converted to CO) that could occur under maximum melter glass production (10-lbs/h/Ft^2) and reductant loading conditions and minimum film-cooler flow rate (6 scfm) is: 0.14% (~1% of LFL). 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 gas chromatograph for H_2 and CO.

In the event that H_2, CO, or H_2+CO should exceed 25% of their flammability limits (4%, 12.5%, and 8.25%, respectively) under baseline operating conditions, film cooler dilution air will be increased, and/or feed rates will be reduced to control off-gas concentration at safe operating limits (25% of LFL). 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.

8.3 Loss Of Vacuum

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 contains totally un-reacted feed with a 1-kg equivalent oxide content (~1/3 that present in the melt cavity), the maximum possible volumetric releases of all anticipated noxious, room-temperature gases are summarized in Table 8.1.

Table 8.1. Maximum RSM Noxious Gas Release Estimates Accompanying Off-Gas System Failure

Volatiles	Max Vol (Std Liters)	Vol (%)	High Bay Conc (ppm)
NO and/or NO_2	4.9	7.9	0.22
CO	15.0	24.2	0.68
CO_2	42.3	67.9	1.93

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 3 ppm. However, the cross ventilation that will result from opening 2 or more (as appropriate) 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.

9.0 Personnel Safety

To ensure the safe operation of the RSM and general safety of operators, staff responsible for operating test equipment, recording data, and making process changes will be trained by the cognizant PNNL project manager (RW Goles). At a minimum, operators will be required to read and understand the following documents:

1. Research Scale Melter SOP #80, latest version
2. The Research Scale Melter Test Plan
3. The applicable Test Instruction
4. All pertinent Material Safety Data Sheets

These activities will be documented by signing-off training instruction and a record sheet as each activity is completed. After completion of all reading assignments, operating staff will be required to attend a complete system walk through. The walk through will include a review of the Test Plan, and the RSM SOP.

There may be times when the melter will need to be opened (to clear the off-gas line, probe for salt, cold-cap sampling etc.). Prior to opening the melter, feeding will be stopped, the melter vacuum will be increased to 2"wc and the cold cap will allowed to dry or to be fully incorporated into the melt if at all possible. This will ensure that no noxious or hazardous gases are present in the system when it is opened. The melter must be maintained at a slight vacuum so that no gases are expelled into the work area.

9.1 Personnel Protective Equipment

During test operations involving the RSM, its feed system, off-gas hardware and secondary waste streams, required personnel protective equipment (PPE) 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. Latex gloves should be worn when conducting sampling or transfer operations involving the off-gas condensate and melter feed. A full-face shield is also required when performing maintenance or disassembling the melter feed system if the line could be under pressure, e.g., a plugged feed line. PPE required for routine activities including data collection and operation of the computer controller are governed by Laboratory 177 requirements. Equipment needed for other activities is discussed in SOP #80.

9.2 Material Safety Data Sheet (MSDS)

Material Safety Data Sheets 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.

9.3 Medical Requirements

N/A

9.4 Confined Space

N/A

9.5 Respiratory Protection Requirements

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

10.0 Waste Minimization/Management

The following waste minimization practices will be followed:

- 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.
- Simulant preparation procedures will be designed to reduce the volume of toxic chemical containing stock.

11.0 Associated Procedures, Test Instructions, and Safety Limits

The following documents are pertinent to this test activity.

- SOP #80, Safe Operating Procedure for the RSM.

12.0 Emergency Response

Actions for Emergency Response are detailed in SOP #80. Personnel working to SOP #80 shall be trained and understand the appropriate actions to be taken in the event of an emergency and an audible/visual alarms that may be encountered 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, and in the RSM's Test Plan and Test Instructions.

13.0 References

Kot, Wing K., Hao Gan and Ian L. Pegg, October 2000, *Physical and Rheological Properties Of Waste Simulants and Melter Feeds for RPP-WTP HLW Vitrification,* VSL-00R2520-1, Vitreous State Laboratory, The Catholic University of America Washington, DC 20064.

Matlack, Keith S., Wing K. Kot, and Ian L. Pegg, June 2000, *Screening Tests on DuraMelter 10 with C-106/AY-102 Simulant in Support of DuraMelter 1000 Throughput Tests,* VSL-00R2501-2, Vitreous State Laboratory, The Catholic University of America Washington, DC 20064.

Matlack, Keith S., Wing K. Kot, F. Perez-Cardenas and Ian L. Pegg, April 2000, *Determination of the Processing Rate of RPP-WTP HLW Simulants Using a DuraMelter 1000 Vitrification System,* VSL-00R2590-1, Vitreous State Laboratory, The Catholic University of America Washington, DC 20064..

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

Toxicity Characteristic Leaching Procedure (TCLP). 1992. *Test Methods for Evaluating Solid Waste, Volume 1C: Laboratory Manual Physical/Chemical Methods,* SW-846, Method 1311, Rev. 2, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington D.C.

Appendix A.2: Test Instructions for Vitrification Demonstration Tests of C-106/AY-102 Waste Simulant

This test instruction provides additional guidance to testing staff during the execution of the RSM C-106/AY-102 flowsheet test that will be referred to, from this point forward, as RSM-H1.

Objectives

Waste Processing: The primary objective of the current RSM test is to characterize the melter-process flowsheet based upon the Hanford's C-106/AY-102 waste envelope D and an optimized target glass composition. Melter feed with 55 Wt% C-106/AY-102 waste loading will be processed using sugar and oxalic acid as feed stream reductants. If steady-state processing at 55% waste loading is successfully achieved and Test Plan objectives have been achieved, the processing of characteristics at 60% waste loading will be evaluated as a secondary objective. Although the feed concentration of oxalic will be fixed, the sugar content of the melter feed may be varied to produce reference processing conditions where reduced Fe (II) can just be detected. Glass oxidation state will be determined by wet chemical, colorimetric procedure.

The melter feeds to be used in RSM-H1 were specifically formulated to maximize C-106/AY-102 waste loading in order to increase waste throughput rates at a reference processing temperature of 1150 °C. Therefore the major goal of the test will be to establish maximum steady state processing rates at 95% cold cap coverage. Since validation of steady-state cold-cap coverage conditions requires long-term observations of melter behavior, maintaining stable fixed operating conditions during the testing is a high priority. Consequently the ramping of melter feeding rates to achieve 95% cold cap conditions needs to be conducted slowly over a reasonably long period of time to preclude overfeeding and resultant reactive oscillatory (up/down) feeding conditions that are not representative of steady-state processing.

The reductant concentrations to be used to achieve glass oxidation levels previously discussed will be defined by the glass laboratory prior to the test. Based on subsequent testing of the glass produced, some adjustment of the sucrose concentration may be necessary if the glass is too oxidizing. A wet-chemical, colorimetric laboratory procedure will be used during initial phases of RSM-H1 testing to establish the oxidation state of the product glass and to determine the impact of resultant incremental changes in reductant concentrations.

Melter Off-Gas Characterization: At three separate times during RSM-H1 testing, discrete sampling of melter generated aerosols and condensable effluents will be conducted. This will occur, without exception, during steady-state processing periods. It is

the operator's task to maintain these conditions throughout the sampling campaign. The timing of the sampling to be conducted will be established during testing by the Project manager, and executed by non-shift staff.

To initiate sample collection, a sampling probe connected to a filter and a series of impinger bottles will be inserted into the off-gas sampling port located just down stream of the film cooler. An isolation valve equipped with inlet slip fittings will be used to reduce the impact of sample probe insertion and extraction activities upon melter vacuum. However, prior to probe insertion or extraction, the melter vacuum should be manually increased, if necessary, to 2"wc to compensate for a temporary increase in off-gas system inleakage. The off-gas vacuum control valve should be manually adjusted during these probe insertion and extraction periods to maintain this melter vacuum. Normally the melter vacuum is controlled at 1"-2"wc.

Secondary Waste Stream Evaluation: Secondary aqueous waste streams generated by the EVS and HEME off-gas components will be sampled throughout RSM-H1 testing as specified in the Test Plan. In addition at the conclusion of testing the EVS condensate will be harvested as discussed in the Test Activity section of these instructions. The collected EVS' secondary waste stream volume will be measured and all settled undissolved matter be segregated from the supernatant. This separation will be conducted in a secondary containment within the feed preparation hood. This can be accomplish by decanting or the condensate supernatant can be drawn off the settled solids with peristaltic pump that delivers this liquid directly to a waste drum. The settled solids along with residual supernatant will then be transferred to an appropriately sized vessel for subsequent analysis.

In addition to the HEME run-off collected during testing, the HEME filter element and vessel will be soaked in water to extract residual soluble effluent remaining on the filter. The volumes and concentrations of HEME run-off and soak water will be subsequently measured to support mass balance evaluations.

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 of C-106/AY-102 waste and glass formers will be prepared in ~20-liter volume increments. The first hours of operation will establish the average volumetric feed rate, e.g., one to two liters per hour. This value will be used to calculate when subsequent volumes of feed

need to be prepared to maintain adequate feed inventories in the melter feed tank. The attached Feed Batching Sheet (See Attachment) will be used and completed as part of the preparation of each feed batch. The preparation steps are as follows:

a. Test engineer to enter into the Feed Batching Calculation Sheet:
   i. A sequential feed batch number (Starting with "RSM-H1-F-1")
   ii. Name, date and time.
   iii. The C-106/AY-102 target waste batch volume
b. Transfer target volume of C-106/AY-102 simulant to the waste transfer tank - The C-106/AY-102 "master batch" will be stored in the large RSM feed tank. A poly line connected to the bottom of the transfer tank will be used to dispense feed to the make-up tank. It is required that splash protection including: lab coat, full-face shield or safety goggles and chemical resistant gloves be used for this task.
c. Estimate the transferred C-106/AY-102 volume and measure gross weight (tare weight is written on the transfer tank; 2.30 kg).
d. Use waste density and weight to confirm/calculate the actual C-106/AY-102 transfer volume.
e. Calculate the glass former/reductant weights by entering these data into the Batch Calculation Sheet and print out the Feed Batching Sheet.
f. Weigh glass formers/reductant and combine into the glass former containers and sign Feed Batching Sheet.
g. Shift engineer or lead test engineer review and sign off the Feed Batching Sheet.

2. <u>Melter Feed Tank Transfer:</u> 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 Lab coat or other protective over garments).
   c. Note the melter feed tank weight and time and record on the batch sheet and in the LRB. Note: be sure to remove all tools, buckets, etc. from the scale before recording the feed tank weight.
   d. Place the C-106/AY-102 transfer tank on the transfer stand, secure it, introduce an agitator if necessary, initiate agitation and record platform scale weight in LRB.
   e. Extend the C-106/AY-102 transfer tank hose into the lid opening of the melter feed tank and open the valve at

the base of the 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.**

f. 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 C-106/AY-102 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.
g. Note the time and melter feed tank weight after waste addition and record on the batch sheet **and the logbook.**
h. Place the glass former containers within easy access.
i. Place the copus blower intake hose at the feed station.
j. Rotate the agitator shaft angle to the vertical position and secure. This orientation allows a larger vortex to form.
k. Increase the agitator speed to develop a vortex around the shaft. **IF SPLASHING DEVELOPS REDUCE THE SPEED UNTIL SPLASHING DOES NOT OCCUR**
l. Remove the tank lid and slide it into the plastic bag next to the tank to prevent any feed splatter on the tank lid from spreading.
m. Turn on the copus blower and hold the intake at the tank edge.
n. 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. Continue until all feed additives are dispensed.
o. 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).
p. Inspect each other for any signs of feed splash stains and wipe with a damp paper towel.
q. Realign the melter feed tank agitator and adjust its speed to maximize agitation without creating splashing.
r. Note the time and melter feed tank weight after glass former addition and record these values on the batch sheet **and the logbook.**

3. Melter Feed Tank Change-Out: To completely change feed Tank contents follow steps in 2. after completing the following
   a. Turn feed tank agitator and feed circulator off.
   b. Close feed recirculator return line valve and move line to an appropriately sized receiver vessel.
   c. Open return line valve and gravity drain or turn on feed recirculator and pump melter feed tank contents into an appropriately sized vessel
   d. Close drain valve, return feed line to melter feed tank, open valve and remove receiver tank to a hood and initiate agitation
   e. Follow steps in 2. above.

4. EVS Condensate Tank Volume Adjustment/Sampling: At the beginning of testing, the EVS condensate tank will be charged with 100-liters of water adjusted to a pH of 11 by adding 4g of NaOH. If condensate accumulations increase condensate tank volume to 200-liters, 100-liters of condensate should be transferred to appropriate sized vessels by:
   a. Connecting receiver vessels to the condensate-sampling valve.
   b. Slowly open the sampling valve to begin the transfer.
   c. Adjust the EVS/condensate-tank bypass valve to maintain the spray nozzle pressure between 50 psig and 55 psig.
   a. Close condensate-sampling valve and readjust bypass valve to maintain nozzle pressure between 50 psig and 55 psig when the transfer has been completed.

   At the conclusion of the testing after the EVS has been shut down all condensate and undisolved solids will be harvested. The following steps should be conducted to collect the EVS' waste stream.

   a. Suspend feeding to the melter and water flush the feed nozzle.
   b. When the cold cap has been completely consumed reduce the melter vacuum to ½"wc.
   c. Turn off EVS pump.
   d. Close EVS drain line.
   e. Position condensate receiver vessel(s) next to the condensate tank and connect drain. (An exhaust line or a Copus blower in the proximity of the vessels lid can be used if appropriate to exhaust displaced gases).
   f. Drain condensate tank using a spray lance to wash out insoluble.
   g. Close tank drain valve, remove receiver vessel(s) to the feed makeup hood and allow suspended solids to settle.

5. HEME Runoff Sampling: At the conclusion of testing, the final HEME runoff sample should be collected. In addition post-test, water soaking of the HEME vessel will be conducted according to PNNL's safe operating procedure "APEL_INEEL_102" in order to harvest soluble effluents held up on the high surface area filter element.

6. Off-Gas Condensate Tank pH Adjustment: The pH of the condensate will be measured hourly. If the pH drops to 9, the solution pH should be adjusted to 11 by adding an appropriate quantity of NaOH pellets. Record the amount of NaOH added in the laboratory record book and the NaOH log sheet.

7. Test Conditions: The attached Run Guidance form will be used to document current test conditions and identify any necessary changes to the Test Plan and/or Test Instruction.

8. Sample Identification: Record samples in Sample Log (see attachments) and mark sample containers with the following:

    a. RSM-H1-x, where "x" is a sequential sample number
    b. Date & Military Time
    c. Sample description, e.g., *Feed, Condensate, Glass Sample, etc.*
    d. Initial of operations staff obtaining sample

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

Note: Appropriate adjustments should be made to ensure operating parameters, if identified on these sheets, remain in range. Descriptive comments regarding as-found conditions, necessary adjustments and other observations should be made in the LRB

FEED BATCHING CALCULATION SHEET

Batch Sheet No. RSM-H1-Fd-: _____

Prepared by: _____

Date: _____ Time: _____

Waste target volume, Liters:	15		Note: shaded areas require value to be input
Waste transfer tank gross wt:	20.95	kg	
Waste transfer tank tare wt:	2.30	kg	
Waste transfer tank net wt:	18.65	kg	
Waste Density:	1.25	kg/L	

Calculated volumed transferred: Net wt. / density = 14.92 Liters

Target glass waste oxide fraction:	0.55

Glass Former Addition Calculation

Glass former weights: 375 gm WO/L * (1 gm glass/ B gm WO) * (C gm GFO/ gm glass) * D-L Wst * (gm GF chemical / gm total GFO)

Waste oxide loading, g/L:	374.9	= A
Fraction waste oxide loading:	0.55	= B
Fraction glass former loading:	0.45	= C
C-106/AY-102 Volume, liters	14.92	= D
(A/B) * C * D =	4576.51	= E

Glass formers	F		Batch totals: (E * F)	Initial after weighing
gm silica / gm total GFO =	0.626	=>	2863.3 grams	
gm boric acid / gm total GFO =	0.118	=>	542.0 grams	
gm Na_2O / gm total GFO =	0.352	=>	1610.8 grams	
gm LiOH / gm total GFO =	0.098	=>	449.5 grams	
gm sugar/L-Waste	135	=>	2014.2 grams	

Initial melter feed tank weight: _____ kg Time: _____

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

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

Completed by (sign & data): _____

Reviewed & Approved by: _____

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:

Initial Sugar Addition Level:

Second Sugar Addition Level:

Third Sugar Addition Level:

Additional Guidance:

1. _____

2. _____

3. _____

4. _____

5. _____

6. _____

7. _____

8. _____

RSM-F1 Sample Data Log

Sample Number	Date	Time	Initials	Sample Source	Size	ARF #	Archived	Comments
RSM-F1-1								
RSM-F1-2								
RSM-F1-3								
RSM-F1-4								
RSM-F1-5								
RSM-F1-6								
RSM-F1-7								
RSM-F1-8								
RSM-F1-9								
RSM-F1-10								
RSM-F1-11								
RSM-F1-12								
RSM-F1-13								
RSM-F1-14								
RSM-F1-15								
RSM-F1-16								
RSM-F1-17								
RSM-F1-18								
RSM-F1-19								
RSM-F1-20								
RSM-F1-21								
RSM-F1-22								
RSM-F1-23								
RSM-F1-24								
RSM-F1-25								
RSM-F1-26								
RSM-F1-27								
RSM-F1-28								
RSM-F1-29								
RSM-F1-30								
RSM-F1-31								
RSM-F1-32								
RSM-F1-33								
RSM-F1-34								
RSM-F1-35								

Data Sheet #1: Priority & Electrical Data

RSM-F1	By:									
	Date:									
	Time									
DESCRIPTION	UNITS	Range								
Priority Data										
Melt Temperature (T1, control)	° C	1125 - 1175								
Melt Temperature (T2)	° C	1125 - 1175								
Plenum Temperature	° C	400 - 600								
Feed pump setting	%									
Cold Cap Coverage	%	> 75								
Slurry Pool Coverage of Cap	%									
Cold Cap Flexibility Visible	y or n									
Number of Vents	#									
Cold Cap Thickness	inch									
Phase Separation (Note in LRE	y or n									
Glass Pouring	y or n									
Electrical Data										
Electrode Potential	Volts									
Electrode Current	Amps									
Electrode Power	kW									
Melt Resistance	?									
Melt (Electrode) Setpoint Temp	° 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-F1		By:								
		Date:								
		Time:								
DESCRIPTION	**units**	**range**								
Feed Nozzle Cooling Flow	gpm	.5 - 1(reg FNT)								
Film Cooler Air Supply	scfm	1-10 (reg OGT)								
Melter Vacuum-Magnehelic	in. H_2O	0.1 - 2.0								
EVS ?P	in. H_2O									
Film Cooler ?P	in. H_2O									
HEME ?P	in. H_2O									
System ?P	in. H_2O									
EVS HX Cooling Flow	gpm	1-5 (reg SLT)								
EVS Scrub Tank Volume	gallon	35 - 50								
EVS Nozzle Pressure	psi	50 - 55								
EVS Scrub Solution pH	pH	> 9								
OG Control Valve position	%									
OG Control Valve mode	A or M									
Feed Pump (tubing) Condition										
Agitator Setting	%									
Blower Cooling Flow	gpm	1 - 1.5								
Feed Nozzle Temp (FNT)	° C	< 40								
Off-Gas Temp (OGT)	° C	< 250								
Post EVS Off-Gas Temp	° C	< 50								
Scrub Liquid Temp (SLT)	° C	<40								
Heat Xer Temp	° C	< 30								
Feed Pump Setting	%									
Feed Pump Control Mode	A or M									
Feed Pressure	psi	<1.0								
Feed Tank Wt	Kg	decreasing								
Overflow Temp	° C	1000 - 1100								
Discharge Can Temp	° C	750 - 850								
Glass Scale	Kg	< 10								
Alarm Condition	On/Off	off								
time	hh:mm									

Data Sheet #3: Off-Gas Monitoring Data

RSM-F1		By:								
		Date:								
		Time:								
DESCRIPTION	**units**	Range								
Gas Chromatograph										
He	ppm									
H_2	ppm	<1e4								
O_2	ppm									
N_2	ppm									
NO	ppm									
CO	ppm	<3E4								
CO_2	ppm									
N_2O	ppm									
NOx	ppm									
H_2 + CO	ppm	<2E4								
NOx Analyzer Computer										
Time	hh:mm									
NOx	ppm									
NO	ppm									
NOx Voltage	Volts									
NOx Instrument										
Sample Pressure	psig									
Sample bypass flow	sccm									
Ozone Pressure	psig									
MKS Mass Flow										
Ar Carrier, Chan-1	lpm									
He Tracer, Chan-2	lpm									
Total Sample , Chan-3	lpm									
Sample Pump*										
Vac Gauge	"Hg									
Pres Gauge	psig	<15								
Gas Cylinders Tank Pres#										
O_2	psig	>200								
Ar GC	psig	>201								
Ar Sweep	psig	>202								
He GC	psig	>203								
He Tracer	psig	>204								
time	hh:mm									

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

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

Appendix B

Test Instructions for Preparing C-106/AY-102Waste Simulant

Appendix B: Test Instructions for Preparing C-106/AY-102Waste Simulant

TEST INSTRUCTION FOR PREPARING 125L OF C-106/AY-102 HIGH-LEVEL WASTE SIMULANT

Objective: To prepare a C-106/AY-102 high-level waste (HLW) simulant to use in PNNL RSM testing.

Start Date: July 27, 2002

End Date: July 27, 2002

Laboratory Record Book Used _____, pp. _____

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

Author: Renée Russell

Responsible Scientist: Ronald W. Hahn

Test Operator(s): Renée Russell

William C Buchmiller

Balance Calibration Record:

Balance Calibration # 2810009 Last Cal. 2/02 Due 8/02

Balance Calibration # M28819 Last Cal. 2/02 Due 8/02

Chemical Inventory List:

Chemical	Manufacturer	Lot #	Amount Used
Ag_2O	Noah Tech	80136/1.1	173.24g
$Al(OH)_3$	Noah Tech	101476/1.1	12,322 g
$Ba(OH)_2 \cdot 8H_2O$	Noah Tech	11881/3.1	94.5g
$CaCO_3$	Noah Tech	85823/1.1	951g
CdO	Mallinckrodt	ACY	31.51g
$Ce(OH)_4$	Noah Tech	57758/0.0	31.51g
NaCl	Baker	149357	17.12g
$Cr_2O_3 \cdot 1.5H_2O$	Noah Tech	73653/0.0	139.91g
CsOH(50% soln)	Noah Tech	99133/1.1	157.51g
NaF	Baker	949110	47.24g
$Fe(OH)_3$ (13wt% slurry)	Noah Tech	111849/1.1	~89 Kg
KNO_3	Noah Tech	22804/1.1	47.25g
$La(OH)_3 \cdot 3H_2O$	Noah Tech	111783/1.1	126.0g
MnO_2	Noah Tech	94942/1.1	4051g
NaOH(50% soln)	Noah Tech	69510/1.1	15.4 Kg
$Ni(OH)_2$	Noah Tech	99122/1.1	140.35g
$FePO_4 \cdot xH_2O$	Noah Tech	96031/1.1	235.72g

Chemical Inventory List:

Chemical	Manufacturer	Lot #	Amount Used
$(PbCO_3)_2 \cdot Pb(OH)_2$	Noah Tech	111981/1.1	189.01g
SiO_2	Alfa Aesar	H08K26	4410g
$SrCO_3$	Noah Tech	75967/1.1	5875g
$Sr(OH)_2 \cdot 8H_2O$	Noah Tech	73184/1.1	3528g
TiO_2	Spectrum	42018A04	6.30g
$Zr(OH)_4 \cdot xH_2O$	Noah Tech	101630/1.1	124.11g
$NaNO_2$	Baker	H47937800120	1.23g
$NaNO_3$	Noah Tech	37467/3.1	492.6g
$H_2C_2O_4 \cdot 2H_2O$	Noah Tech	79091/1.1 ~~2485 g~~ RR 7-27-02	2485g

1. Add 88.97 kg of 13 wt% $Fe(OH)_3$ slurry to an appropriate sized vessel.

   Amount of $Fe(OH)_3$ slurry added: ~89 Kg

2. Add the following salts to the $Fe(OH)_3$ slurry in the amounts indicated:

Chemical	Target Wt.	Actual Wt.	Initial	Date
Ag2O	173.25 g	173.24g	RR	7-27-02
Al(OH)3	12.32 kg	12,322g	RR	7-27-02
$Ba(OH)2 \cdot 8H_2O$	94.5 g	94.50g	RR	7-27-02
CaCO3	950.75 g	951g	WCB	7/27/02
CdO	31.5 g	31.51g	RR	7-27-02
Ce(OH)4	31.5 g	31.51g	RR	7-27-02
NaCl	17.12 g	17.12g	RR	7-27-02
$Cr2O_3 \cdot 1.5H_2O$	139.91 g	139.91g	RR	7-27-02
CsOH (50% soln)	157.5 g	157.51g	RR	7-27-02
NaF	47.25 g	47.24g	RR	7-27-02
KNO3	47.25 g	47.25g	WCB	7/27/02
$La(OH)3 \cdot 3H_2O$	126.00 g	126g	RR	7-27-02
MnO2	4051 g	4051g	WCB	7/27/02
NaOH (50% soln)	15.47 kg	15.4 Kg	WCB	7/27/02
Ni(OH)2	140.34 g	140.35g	RR	7-27-02
$FePO4 \cdot xH_2O$	236.25 g	235.72g	RR	7-27-02
$(PbCO3)2 \cdot PB(OH)2$	189.0 g	189.01g	RR	7-27-02

Chemical	Target Wt.	Actual Wt.	Initial	Date
SiO2	4410 g	4410g	WCB	7/27/02
$SrCO_3$	5874.8 g	5875g	WCB	7/27/02
$Sr(OH)2 \cdot 8H_2O$	3528 g	3528g	WCB	7/27/02
TiO_2	6.3 g	6.30g	RR	7-27-02
$Zr(OH)_4 \cdot xH_2O$	124.10 g	124.11g	RR	7-27-02
NaNO2	1.23 g	1.23g	WCB	7/27/02
$NaNO_3$	492.55 g	492.6g	WCB	7/27/02
$H2C2O4 \cdot 2H_2O$	2485.4 g	2485g	WCB	7/27/02

2. Add 16.87 kg of DI water to the slurry.

   Actual amount of DI water added: 16.90 Kg

3. Stir the slurry until thoroughly mixed and uniform.

4. Final slurry volume: 126L

Waste Fraction
~~Feed~~ Makeup For 7/02 RSM Test

	Chemical	Amount Needed, g	Amount Added	Manufacturer	Lot #
	Ag2O	173.25	120g + 53.24g	Noah Tech	80136/1.1
	Al(OH)3	12322	10kg + 2322g	Noah Tech	10476/1.1
	Ba(OH)2*8H2O	94.5	94.50g	Noah Tech	11881/3.1
☆	CaCO3	950.75	890 + 61	Noah	85823/1.1 *3
	CdO	31.5	31.51g	Mallinckrodt	ACY
	Ce(OH)4	31.5	31.51g	Noah Tech	57758/0.0
	NaCl	17.12	17.12g	Baker	149357
	Cr2O3*1.5H2O	139.91	100g + 39.91g	Noah Tech	73653/0.0
	CsOH (50% soln)	157.5	157.51g	Noah Tech	99133/1.1
	NaF	47.25	47.24g	Baker	949110
	Fe(OH)3 Slurry (13%)	88972	~89 Kg	Noah Tech	111849/1.1
Don't add ?	KNO3 ☆	47.25	47.25	Noah Tech	22804/1.1
	La(OH)3*3H2O	126	126.00g	Noah Tech	111783/1.1
	4MgCO3*Mg(OH)2*5H2O	○ ~~393.75~~			
☆	MnO2	4051.0	3825 + 226	Noah	94942/1.1 *2
Add 10kg ?	NaOH (50% soln) ☆	15467	15.4 Kg	Noah	69510/1.1
	Ni(OH)2	140.34	140.35g	Noah Tech	99122/1.1
	FePO4*xH2O	236.25	235.72g	Noah Tech	96031/1.1
	(PbCO3)2*Pb(OH)2	189	189.01g	Noah Tech	111981/1.1
	Na2SO4	○ ~~31.5~~			
☆	SiO2	4410	4410	Alfa Aeser	H08K26
☆	SrCO3	5874.8	5875	Noah	75967/1.1
☆	Sr(OH)2*8H2O	3528	3528	Noah	73184/1.1
	TiO2	6.3	6.30g	Spectrum	42018AC4
	Zr(OH)4*xH2O	124.10	124.11g	Noah Tech	101630/1.1
don't add ?	Water ☆	16868.3	16.90 Kg	NA	NA
don't add ?	NaNO2 ☆	1.575	1.23	Baker	#47937800 12D
don't add ?	NaNO3 ☆	492.98	492.6	NOAH	37467/3.1
☆	H2C2O4*2H2O	2485.4	2485	Noah	79091/1.1

Tare Weight of Mixing Vessel	NA
Final Weight of Simulant + Mixing Vessel	NA
Final Weight of Simulant	NA

*2 226g Baker Lot# 38276

*3 61g Fisher Lot# 007112

☆ added by WCBuchmiller

WCBuchmiller 7/27/02

WCB for Renee Russell 7/27/02

Water total = 16916 g
NaOH soln total = 15371 g
$NaNO_2$ total = 1.23 g
$NaNO_3$ total = 492.55 g
KNO_3 total = 47.25 g

*4 amounts to be added was called out on makeup sheet ... due to $Fe(OH)_3$ slurry analysis
WCB 7/27/02

Appendix C

RSM Feed Batching and Manual Data Sheets

RSM Feed Batching Data Sheets

FEED BATCHING CALCULATION SHEET

Batch Sheet No. RSM-H1-Fd-: 1

Prepared by: RW Goles

Date: 7/28/2002 Time: 11:00

Waste target volume, Liters: 30

Note: shaded areas require value to be input

Waste transfer tank gross wt: 38.95 kg
Waste transfer tank tare wt: 0.00 kg
Waste transfer tank net wt: 38.95 kg
Waste Density: 1.29 kg/L

Calculated volumed transferred: Net wt. / density = 30.193798 Liters

Target glass waste oxide fraction: 0.60

Glass Former Addition Calculation

Glass former weights: 375 gm WO/L * (1 gm glass/ B gm WO) * (C gm GFO/ gm glass) * D-L Wst * (gm GF chemical / gm total GFO)

Waste oxide loading, g/L: 374.9 = A
Fraction waste oxide loading: 0.6 = B
Fraction glass former loading: 0.4 = C
C-106/AY-102 Volume, liters 30.193798 = D

(A/B) * C * D = 7546.44 = E

Glass formers	F		Batch totals: (E * F)	Initial after weighing	
gm silica / gm total GFO =	0.671	=>	5063.5 grams	5065	WCB
gm boric acid / gm total GFO =	0.536	=>	4048.3 grams	4050	WCB
gm LiOH / gm total GFO =	0.076	=>	572.8 grams	572.8	
gm sugar/L-Waste	9	=>	271.7 grams	No Sugar	RWG

Initial melter feed tank weight: 0. kg Time: 09:46
Melter feed tank wt. after Waste addition: 38.95 kg Time: 10:30
Melter feed tank wt. after glass formers addition: 48.64 kg Time: 11:30

Completed by (sign & data): WCBuchmiller 7/28/02

Reviewed & Approved by: RW Goles 7/28/02

NOTE POWER OFF TO LOAD CELL TARE WT LOST, CHEMICAL ADDITION CALCULATED! RWG

FEED BATCHING CALCULATION SHEET

Batch Sheet No. RSM-H1-Fd-: 2

Prepared by: JS Tixier

Date: 7/29/02 Time: 5:00 AM

Waste target volume, Liters: 20

Note: shaded areas require value to be input

Waste transfer tank gross wt: 25.8 kg
Waste transfer tank tare wt: 0.00 kg
Waste transfer tank net wt: 25.80 kg
Waste Density: 1.29 kg/L

JJ

Calculated volumed transferred: Net wt. / density = 20 Liters

Target glass waste oxide fraction: 0.60

Glass Former Addition Calculation

Glass former weights: 375 gm WO/L * (1 gm glass/ B gm WO) * (C gm GFO/ gm glass) * D-L Wst * (gm GF chemical / gm total GFO)

Waste oxide loading, g/L: 374.9 = A
Fraction waste oxide loading: 0.6 = B
Fraction glass former loading: 0.4 = C
C-106/AY-102 Volume, liters 20 = D

(A/B) * C * D = 4998.67 = E

Glass formers	F		Batch totals: (E * F)	Initial after weighing
gm silica / gm total GFO =	0.671	=>	3354.0 grams	[illegible]
gm boric acid / gm total GFO =	0.536	=>	2681.5 grams	[illegible]
gm $LiOH \cdot H_2O$ / gm total GFO =	0.076	=>	379.4 grams	[illegible]
gm sugar/L-Waste	9	=>	180.0 grams	[illegible]

Initial melter feed tank weight: 193.0 kg Time: 0732

Melter feed tank wt. after Waste addition: 218.5 kg Time: 0740

Melter feed tank wt. after glass formers addition: 224.5 kg Time: 0812

Completed by (sign & data): [illegible] 7/29/02

Reviewed & Approved by: [illegible] 7/29/02

FEED BATCHING CALCULATION SHEET

Batch Sheet No. RSM-H1-Fd-: 3

Prepared by: RW Goles

Date: 7/29/2002 Time: 23:32

Waste target volume, Liters: 20

Note: shaded areas require value to be input

Waste transfer tank gross wt: 25.8 kg
Waste transfer tank tare wt: 0.00 kg
Waste transfer tank net wt: 25.80 kg
Waste Density: 1.29 kg/L

Calculated volumed transferred: Net wt. / density = 20 Liters

Target glass waste oxide fraction: 0.60

Glass Former Addition Calculation

Glass former weights: 375 gm WO/L * (1 gm glass/ B gm WO) * (C gm GFO/ gm glass) * D-L Wst * (gm GF chemical / gm total GFO)

Waste oxide loading, g/L: 374.9 = A
Fraction waste oxide loading: 0.6 = B
Fraction glass former loading: 0.4 = C
C-106/AY-102 Volume, liters 20 = D

(A/B) * C * D = 4998.67 = E

Glass formers	F		Batch totals: (E * F)	Initial after weighing
gm silica / gm total GFO =	0.671	=>	3354.0 grams	3355
gm boric acid / gm total GFO =	0.536	=>	2681.5 grams	2680
gm $LiOH \cdot H_2O$ / gm total GFO =	0.076	=>	379.4 grams	379.40
gm sugar/L-Waste	7.5	=>	150.0 grams	127.25

Initial melter feed tank weight: 201.0 kg Time: 23:26

Melter feed tank wt. after Waste addition: 227.0 kg Time: 23:57

Melter feed tank wt. after glass formers addition: 233.5 kg Time: 00:29

Completed by (sign & data): [signature]

Reviewed & Approved by: [signature] 7/30/02

FEED BATCHING CALCULATION SHEET

Batch Sheet No. RSM-H1-Fd-: 4

Prepared by: RW Goles

Date: 7/30/2002 Time: 21:57

Waste target volume, Liters: 10

Note: shaded areas require value to be input

Waste transfer tank gross wt:	12.9	kg
Waste transfer tank tare wt:	0.00	kg
Waste transfer tank net wt:	12.90	kg
Waste Density:	1.29	kg/L

Calculated volumed transferred: Net wt. / density = 10 Liters

Target glass waste oxide fraction: 0.60

Glass Former Addition Calculation

Glass former weights: 375 gm WO/L * (1 gm glass/ B gm WO) * (C gm GFO/ gm glass) * D-L Wst * (gm GF chemical / gm total GFO)

Waste oxide loading, g/L:	374.9	= A
Fraction waste oxide loading:	0.6	= B
Fraction glass former loading:	0.4	= C
C-106/AY-102 Volume, liters	10	= D
(A/B) * C * D =	2499.33	= E

Glass formers	F		Batch totals: (E * F)	Initial after weighing
gm silica / gm total GFO =	0.671	=>	1677.0 grams	1675
gm boric acid / gm total GFO =	0.536	=>	1340.8 grams	1340
gm $LiOH \cdot H_2O$ / gm total GFO =	0.076	=>	189.7 grams	189.70
gm sugar/L-Waste	7.5	=>	75.0 grams	75.03

Initial melter feed tank weight: 196.5 kg Time: 2217

Melter feed tank wt. after Waste addition: 209.5 kg Time: 2227

Melter feed tank wt. after glass formers addition: 213.0 kg Time: 2251

Completed by (sign & data): JM Davis

Reviewed & Approved by: RW Goles

FEED BATCHING CALCULATION SHEET

Batch Sheet No. RSM-H1-Fd-: 5

Prepared by: RW Goles

Date: 7/31/2002 Time: 9:47

Waste target volume, Liters: 30

Note: shaded areas require value to be input

Waste transfer tank gross wt:	38.7	kg
Waste transfer tank tare wt:	0.00	kg
Waste transfer tank net wt:	38.70	kg
Waste Density:	1.29	kg/L

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

Target glass waste oxide fraction: 0.60

Glass Former Addition Calculation

Glass former weights: 375 gm WO/L * (1 gm glass/ B gm WO) * (C gm GFO/ gm glass) * D-L Wst * (gm GF chemical / gm total GFO)

Waste oxide loading, g/L:	374.9	= A
Fraction waste oxide loading:	0.6	= B
Fraction glass former loading:	0.4	= C
C-106/AY-102 Volume, liters	30	= D

(A/B) * C * D = 7498.00 = E

Glass formers	F		Batch totals: (E * F)	Initial after weighing
gm silica / gm total GFO =	0.671	=>	5031.0 grams	5.030 Kg WCB
gm boric acid / gm total GFO =	0.536	=>	4022.3 grams	4.025 Kg WCB
gm $LiOH \cdot H_2O$ / gm total GFO =	0.076	=>	569.1 grams	569.1 g WCB
gm sugar/L-Waste	7.5	=>	225.0 grams	No SUGAR RWG 7/31/02

Initial melter feed tank weight: 178.5 kg Time: 1002 7/31/02 WCB

Melter feed tank wt. after Waste addition: 216.8 kg Time: _____

Melter feed tank wt. after glass formers addition: 226.8 kg Time: _____

Completed by (sign & data): _____

Reviewed & Approved by: RW Goles 8/1/02

FEED BATCHING CALCULATION SHEET

Batch Sheet No. RSM-H1-Fd-: 6 RWB

Prepared by: LK Jagoda

Date: 7/29/2002 Time: 5:00 AM

Waste target volume, Liters: 20

Note: shaded areas require value to be input

Waste transfer tank gross wt: 10.15 kg
Waste transfer tank tare wt: 0.00 kg
Waste transfer tank net wt: 10.15 kg
Waste Density: 1.29 kg/L

Calculated volumed transferred: Net wt. / density = 7.8682171 Liters

Target glass waste oxide fraction: 0.60

Glass Former Addition Calculation

Glass former weights: 375 gm WO/L * (1 gm glass/ B gm WO) * (C gm GFO/ gm glass) * D-L Wst * (gm GF chemical / gm total GFO)

Waste oxide loading, g/L: 374.9 = A
Fraction waste oxide loading: 0.6 = B
Fraction glass former loading: 0.4 = C
C-106/AY-102 Volume, liters 7.8682171 = D

(A/B) * C * D = 1966.53 = E

Glass formers	F		Batch totals: (E * F)	Initial after weighing
gm silica / gm total GFO =	0.671	=>	1319.5 grams	1320
gm boric acid / gm total GFO =	0.536	=>	1054.9 grams	1055
gm $LiOH \cdot H_2O$ / gm total GFO =	0.076	=>	149.3 grams	149.4
gm sugar/L-Waste	0	=>	0.0 grams	—

Initial melter feed tank weight: _____ kg Time: _____

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

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

Completed by (sign & data): _____

Reviewed & Approved by: R W Gale 8/1/02

RSM Manual Data Sheets: Priority and Electrical Data

Data Sheet #1: Priority & Electrical Data

RSM-F1	By:		WCB	WCB	WCB	WCB	GD	[illegible]	[illegible]	[illegible]
	Date:		7/28/02	7/28/02	7/28/02	7/28/02	7/28/02	7/28/02	7/28/02	7/28/02
	Time		1308	1407	1458	1558	1710	1753	1905	2001
DESCRIPTION	UNITS	Range								
Priority Data										
Melt Temperature (T1, control)	° C	1125 - 1175	1152	1150	1156	1154	1153	1148	1153	1150
Melt Temperature (T2)	° C	1125 - 1175	1135	1147	1145	1152	1170	1155	1154	1147
Plenum Temperature	° C	400 - 600	643	538	531	530	639	613	548	583
Cold Cap Coverage	%	> 75	70	80	75	50	25	90	98	95
Slurry Pool Coverage of Cap	%		10	25	10	Ø	50	25	25	20
Cold Cap Flexibility Visible	y or n		Y	Y	Y	N	N	Y	N	N
Number of Vents	#		4	4	1	0	4+	2	2	1
Cold Cap Thickness	inch		1/4	3/8	1/4	1/2	1/2	1/2	1/2-1"	1"
Phase Separation (Note in LRE	y or n		N	N	N	N	N	N	N	N
Glass Pouring	y or n		N	N	N	N	N	N	N	N
Electrical Data										
Electrode Potential	Volts		31.9	38.5	32.9	28.2	33.7	33.1	30.1	31.2
Electrode Current	Amps		101.6	94	85.1	79.2	84.2	79.8	74.3	69.8
Electrode Power	kW		3.2	3.6	2.8	2.2	2.8	2.6	2.3	2.2
Melt Resistance	Ω		.324	.408	.382	.356	.406	.414	.410	.435
Melt (Electrode) Setpoint Temp	° C		1150	1150	1150	1150	1150	1150	1150	1150
(Electrode Power) Output	%		50	48	42	40	42	41	38	36
(Electrode) Control Mode	A or M		A	A	A	A	A	A	A	A
Kiln Power	kW		0	0.1	0.0	0.2	0	0.2	0.2	0.2
Kiln Temp Setpoint	° C		850	850	850	850	850	850	850	850
Kiln Actual (Middle) Temp	° C		855	847	860	846	848	848	848	851
Kiln Power Output	%		0	20	0	26	3	26	30	29
Kiln Control Mode	A or M		A	A	A	A	A	A	A	A
Discharge Can Power	kW		0.2	0.3	0.2	0.3	0.3	0.3	0.3	0.3
Discharge Can Temp Setpoint	° C		750	750	750	750	750	750	750	750
Discharge Can Power Output	%		46	55	53	58	55	~~58~~ 50	56	~~7~~ 55
Overflow Heater Power	kW		2.8	1.9	1.9	2.1	2.3	2.3	2.2	2.2
Overflow Heater Setpoint	° C		1150	1150	1150	1150	1150	1150	1150	1150
Overflow Heater Power Output	%		69	69	68	72	76	75	73	74
Time	hh:mm		1310	1409	1501	1600		1757	1908	2002

Reviewed and Approved: [illegible] Date: 8/1/02

sheet 1 of 12

Data Sheet #1: Priority & Electrical Data

RSM-F1	By:		SL	JW	JW	SL	CRA	CRA	JJ	CRA
	Date:		7/28/02	7/28/02	7/28/02	7/29/02	7/29/02	7/29/02	7/29/02	7/29/02
	Time		2112	2159	2302	0016	0118	0206	0258	0406
DESCRIPTION	UNITS	Range								
Priority Data										
Melt Temperature (T1, control)	° C	1125 - 1175	1150	1152	1154	1144	1154	1149	1147	1152
Melt Temperature (T2)	° C	1125 - 1175	1149	1141	1150	1175	1161	1141	1154	1149
Plenum Temperature	° C	400 - 600	599	590	590	539	603	546	507	600
Cold Cap Coverage	%	> 75	50	85	98	92	90	92	? Bridge	95
Slurry Pool Coverage of Cap	%		50	25	20	20	10	15	?	10
Cold Cap Flexibility Visible	y or n		Y	N	N	N	Y	N	Y	Y
Number of Vents	#		5	3	1	2	2	2	1	1
Cold Cap Thickness	inch		1/2	1/2	1/2-1 1/2	1/2-1	1/4-1 1/2	1/2-1	?	1/4-1 1/2
Phase Separation (Note in LRB	y or n		N	N	N	N	N	N	—	—
Glass Pouring	y or n		N	N	N	N	Y ~~Stop~~	N	N	Y
Electrical Data										
Electrode Potential	Volts		35.5	29.6	31.3	33.7	32.9	31.4	32.1	31.4
Electrode Current	Amps		75.1	69.4	70.6	81.5	66.5	70.3	75.3	68.3
Electrode Power	kW		2.6	2.0	2.3	2.7	2.1	2.2	2.5	2.2
Melt Resistance	Ω		.473	.429	.449	0.417	0.473	0.444	.427	0.459
Melt (Electrode) Setpoint Temp	° C		1150	1150	1150	1150	1150	1150	1150	1150
(Electrode Power) Output	%		38	35	35	41	33	35	38	35
(Electrode) Control Mode	A or M		A	A	A	A	A	A	A	A
Kiln Power	kW		0.2	0.2	0.2	0.3	0.2	0.3	0.2	0.1
Kiln Temp Setpoint	° C		850	850	850	850	850	850	850	850
Kiln Actual (Middle) Temp	° C		850	851	850	851	851	851	850	849
Kiln Power Output	%		29	29	29	33	25	33	31	20
Kiln Control Mode	A or M		A	A	A	A	A	A	A	A
Discharge Can Power	kW		0.2	0.3	0.2	0.2	0.2	0.2	0.2	0.3
Discharge Can Temp Setpoint	° C		750	750	750	750	750	750	750	750
Discharge Can Power Output	%		49	55	50	~~47 750~~	50	52	50	62
Overflow Heater Power	kW		2.2	2.2	2.1	2.1	2.0	2.2	2.0	2.0
Overflow Heater Setpoint	° C		1150	1150	1150	1150	1150	1150	1150	1150
Overflow Heater Power Output	%		74	73	73	72	68	74	71	70
Time	hh:mm		2116	2201	2306	0019	0124	0208	0304	0410

Reviewed and Approved: [signature] Date: 7/29/02 sheet 2 of 12

Feed Off

Data Sheet #1: Priority & Electrical Data

RSM-F1	By:		JJJ	CRH	WCB	TDD	TDP	WCB	WCB	TDP
	Date:		7/29/02	7/29/02	7/29/02	7/29/02	7/29/02	7/29/02	7/29/02	7/29/02
	Time		0504	0605	0759	0955	1106	1215	1300	1409
DESCRIPTION	UNITS	Range								
Priority Data										
Melt Temperature (T1, control)	° C	1125 - 1175	1152	1152	1140	1150	1149	1150	1147	1148
Melt Temperature (T2)	° C	1125 - 1175	1153	1150	1133	1155	1146	1156	1154	1160
Plenum Temperature	° C	400 - 600	582	566	743	659	601	627	576	556
Cold Cap Coverage	%	> 75	90	98	Ø	30	50	70	80	90
Slurry Pool Coverage of Cap	%		10	20		30	50	20	25	
Cold Cap Flexibility Visible	y or n		Y	N		Y	Y	Y	N	N
Number of Vents	#		2	1		3	2	3	3	1
Cold Cap Thickness	inch		1/2 - 1 1/2	1/2		—	3/4	1	~1"	
Phase Separation (Note in LRB	y or n		—	—		—	N	N	N	
Glass Pouring	y or n		N	Y	N	N	Y	N	N	Y
Electrical Data										
Electrode Potential	Volts		32.6	29.8	33.5	34	37	34.4	35.8	33
Electrode Current	Amps		68.5	66.2	69.5	70	69	69.7	69.2	69
Electrode Power	kW		2.3	2.0	2.3	2.4	2.5	2.4	2.5	2.2
Melt Resistance	Ω		0.478	0.450	.473	0.485	0.544	0.487	0.514	0.477
Melt (Electrode) Setpoint Temp	° C		1150	1150	1150	1150	1150	1150	1150	1150
(Electrode Power) Output	%		35	34	35	35	35	35	36	35
(Electrode) Control Mode	A or M		A	A	A	A	A	A	A	A
Kiln Power	kW		0.2	0.2	0	0	0.2	0.1	0.2	0.1
Kiln Temp Setpoint	° C		850	850	850	800	850	850	850	850
Kiln Actual (Middle) Temp	° C		850	850	856	850	850	851	848	847
Kiln Power Output	%		29	28	0	0	29	22	29	24
Kiln Control Mode	A or M		A	A	A	A	A	A	A	A
Discharge Can Power	kW		0.3	0.2	0.3	0.4	0.2	0.3	0.2	0.2
Discharge Can Temp Setpoint	° C		750	750	750	756	750	750	750	750
Discharge Can Power Output	%		60	52	61	68	51	58	50	52
Overflow Heater Power	kW		2.2	2.0	2.6	2.4	2.1	2.3	2.3	2.1
Overflow Heater Setpoint	° C		1150	1150	1150	1150	1150	1150	1150	1150
Overflow Heater Power Output	%		73	69	80	77	71	76	76	71
Time	hh:mm		0586	0607	0801	0959	1109	1219	1307	1409

Reviewed and Approved: RW Schulz Date: 8/1/02 sheet 3 of 12

Data Sheet #1: Priority & Electrical Data

RSM-F1	By:		WCB	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	Date:		7/29/02	7/29/02	7/29/02	7/29/02	7/29/02	7/29/02	7/29/02	7/29/02
	Time		1504	1554	1701	1800	1905	1957	2106	2159
DESCRIPTION	UNITS	Range								
Priority Data										
Melt Temperature (T1, control)	° C	1125 - 1175	1150	1146	1150	1149	1149	1153	1149	1147
Melt Temperature (T2)	° C	1125 - 1175	1159	1147	1157	1145	1152	1147	1154	1157
Plenum Temperature	° C	400 - 600	547	585	603	541	590	610	548	585
Cold Cap Coverage	%	> 75	95%	75	85	80	80	50	95	70
Slurry Pool Coverage of Cap	%		10%	25	35	20	25	50	20	30
Cold Cap Flexibility Visible	y or n		N	Y	N	N	Y	Y	N	Y
Number of Vents	#		2	5	3	2	3	5	2	3
Cold Cap Thickness	inch		<1"	<1	½"	3/4	3/4	½	3/4	3/4
Phase Separation (Note in LRE	y or n		N	N	N	N	N	N	N	N
Glass Pouring	y or n		N	N	N	N	N	N	N	N
Electrical Data										
Electrode Potential	Volts		33.1	31.6	33.9	34.0	33.2	34.9	32.5	35.3
Electrode Current	Amps		67.4	67.4	68.2	65.5	63.6	63.2	64.8	66.5
Electrode Power	kW		2.2	2.2	2.3	2.3	2.2	2.2	2.1	2.3
Melt Resistance	Ω		.491	.472	.491	.512	0.524	.559	0.495	.527
Melt (Electrode) Setpoint Temp	° C		1150	1150	1150	1150	1150	1150	1150	1150
(Electrode Power) Output	%		34	34	34	34	33	32	33	34
(Electrode) Control Mode	A or M		A	A	A	A	A	A	A	A
Kiln Power	kW		0.2	0.1	0.1	0.2	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	853	851	848	850	851	849	851
Kiln Power Output	%		27	21	16	31	26	26	28	28
Kiln Control Mode	A or M		A	A	A	A	A	A	A	A
Discharge Can Power	kW		0.2	0.3	0.3	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 Power Output	%		53	55	54	49	50	50	50	49
Overflow Heater Power	kW		1.9	2.4	1.9	2.1	2.2	1.9	2.2	2.0
Overflow Heater Setpoint	° C		1150	1150	1150	1150	1150	1150	1150	1150
Overflow Heater Power Output	%		69	77	69	73	74	69	73	71
Time	hh:mm		1508	1600	1704	1803	1908	1958	2109	2201

Reviewed and Approved: R W [illegible] Date: 8/1/02

sheet 4 of 12

Data Sheet #1: Priority & Electrical Data

RSM-F1	By:		[illegible]	GRH	[illegible]	[illegible]	[illegible]	GRH	[illegible]	GRH
	Date:		7/29/02	7/30/02	7/30/02	7/30/02	7/30/02	7/30/02	7/30/02	7/30/02
	Time		2305	0103	0202	0304	0400	0504	0559	0707
DESCRIPTION	UNITS	Range								
Priority Data										
Melt Temperature (T1, control)	° C	1125 - 1175	1152	1150	1149	1151	1152	1148	1152	1149
Melt Temperature (T2)	° C	1125 - 1175	1143	1144	1142	1144	1145	1139	1148	1144
Plenum Temperature	° C	400 - 600	609	774	646	625	578	562	578	561
Cold Cap Coverage	%	> 75	50	0	50	90	95	95	80	85
Slurry Pool Coverage of Cap	%		50	0	20	20	10	20	10	20
Cold Cap Flexibility Visible	y or n		Y	N/A	Y	Y	Y	Y	Y	Y
Number of Vents	#		4	—	lots	3	2	1	2	2
Cold Cap Thickness	inch		1/2	0	1/4	1/2	1/2 - 1	1/2 - 1 1/2	0 - 1	1/4 - 1
Phase Separation (Note in LRE	y or n		N	N	—	—	—	—	—	—
Glass Pouring	y or n		N	N	Dripping	Dripping	Y	N	N	N
Electrical Data										
Electrode Potential	Volts		35.8	31.8	35.0	34.1	34.6	34.9	34.0	33.1
Electrode Current	Amps		63.2	64.0	64.4	64.7	64.0	67.7	63.0	63.8
Electrode Power	kW		2.3	2.0	2.4	2.2	2.2	2.4	2.2	2.2
Melt Resistance	Ω		.564	0.501	.534	.534	0.532	0.518	.548	0.520
Melt (Electrode) Setpoint Temp	° C		1150	1150	1150	1150	1150	1150	1150	1150
(Electrode Power) Output	%		33	34	34	33	33	36	32	33
(Electrode) Control Mode	A or M		A	A	A	A	A	A	A	A
Kiln Power	kW		0.1	0	0	0.2	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		850	850	852	851	851	851	850	851
Kiln Power Output	%		23	0	0	32	30	31	30	32
Kiln Control Mode	A or M		A	A	A	A	A	A	A	A
Discharge Can Power	kW		0.2	0.3	0.4	0.3	0.3	0.3	0.3	0.3
Discharge Can Temp Setpoint	° C		750	750	700	750	750	750	750	750
Discharge Can Power Output	%		50	54	67	62	55	56	56	55
Overflow Heater Power	kW		2.3	2.4	2.0	2.3	2.0	2.2	2.3	2.2
Overflow Heater Setpoint	° C		1150	1150	1150	1150	1150	1150	1150	1150
Overflow Heater Power Output	%		76	77	69	75	70	74	75	73
Time	hh:mm		2308	0107	0204	0306	0400	0521	0600	0711

Reviewed and Approved: [illegible] Date: 8/1/02 sheet 5 of 12

Data Sheet #1: Priority & Electrical Data

RSM-F1	By:		TDP	TDP	TDP	WCB	TDP	WCB	TDP	WCB
	Date:		7-30-02	7-30-02	7-30-02	7/30/02	7/30/02	7/30/02	7/30/02	7/30/02
	Time		800	855	953	1100	1204	1300	1418	1509
DESCRIPTION	UNITS	Range								
Priority Data										
Melt Temperature (T1, control)	° C	1125 - 1175	1150	1150	1149	1150	1150	1150	1147	1149
Melt Temperature (T2)	° C	1125 - 1175	1145	1140	1140	1150	1152	1149	1152	1146
Plenum Temperature	° C	400 - 600	527	634	566	550	549	538	535	556
Cold Cap Coverage	%	> 75	95	25%	80	90	90	90	95	~~130~~ 95
Slurry Pool Coverage of Cap	%		20	50	20	20	20	20	20	20
Cold Cap Flexibility Visible	y or n		N	Y	N	N	N	N	N	N
Number of Vents	#		0	4	1	2	3	2	1	Ø
Cold Cap Thickness	inch		—	—	—	—	—	—	—	—
Phase Separation (Note in LRB	y or n		—	—	—	—	—	—	—	—
Glass Pouring	y or n		N	N	Y	N	N	N	Y	N
Electrical Data										
Electrode Potential	Volts		32	36	33	32	32	33	34	31.3
Electrode Current	Amps		63	65	66	63.5	64	64	65	61.4
Electrode Power	kW		2.1	2.4	2.1	2.0	2	2.1	2.3	2.0
Melt Resistance	Ω		0.51	0.547	0.513	0.503	0.50	0.51	0.505	0.515
Melt (Electrode) Setpoint Temp	° C		1150	1150	1150	1150	1150	1150	1150	1150
(Electrode Power) Output	%		32	33	33	32	33	33	35	32
(Electrode) Control Mode	A or M		A	A	A	A	A	A	A	A
Kiln Power	kW		~~200.2~~	0	0.2	0.2	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	852	858	848	850	849	849	848
Kiln Power Output	%		20	0	30	29	32	29	30	29
Kiln Control Mode	A or M		A	A	A	A	A	A	A	A
Discharge Can Power	kW		0.2	0.3	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 Power Output	%		52	56	48	51	52	49	48	48
Overflow Heater Power	kW		2	2.2	1.8	2.1	2.2	1.9	2.1	2.4
Overflow Heater Setpoint	° C		1150	1150	1150	1150	1150	1150	1150	1150
Overflow Heater Power Output	%		69	73	67	72	73	69	71	77
Time	hh:mm		801	855	955	1102	1205	1302	1419	1511

Reviewed and Approved: RW Jones Date: 8/1/02 sheet 6 of 12

Data Sheet #1: Priority & Electrical Data

RSM-F1	By:									
	Date:		7/30/02	7/30/02	7/30/02	7/30/02	7/30/02	7/30/02	7/30/02	7/30/02
	Time		1557	1657	1801	1901	2005	2103	2158	2305
DESCRIPTION	UNITS	Range								
Priority Data										
Melt Temperature (T1, control)	° C	1125 - 1175	1149	1149	1150	1149	1151	1152	1149	1150
Melt Temperature (T2)	° C	1125 - 1175	1152	1152	1142	1146	1150	1150	1141	1148
Plenum Temperature	° C	400 - 600	547	547	534	570	542	556	525	684 Feed off
Cold Cap Coverage	%	> 75	90	85	90	90	80	95	95	~~80~~ 0
Slurry Pool Coverage of Cap	%		25	25	20	20	20	25	25	0
Cold Cap Flexibility Visible	y or n		N	N	N	N	N	N	N	—
Number of Vents	#		1	1	0	3	2	0	2	—
Cold Cap Thickness	inch		1	3/4	3/4	3/4	3/4	1	3/4	—
Phase Separation (Note in LRE	y or n		N	N	N	N	N	N	N	—
Glass Pouring	y or n		N	N	N	N	N	N	N	—
Electrical Data										
Electrode Potential	Volts		32.8	34.7	32.9	34.2	33.7	30.7	32.3	31.3
Electrode Current	Amps		65.4	65.3	61.9	64.7	63.4	59.6	63.4	60.3
Electrode Power	kW		2.1	2.3	2.1	2.2	2.1	1.9	2.0	1.9
Melt Resistance	Ω		.497	.527	.530	0.528	.535	0.514	.510	.526
Melt (Electrode) Setpoint Temp	° C		1150	1150	1150	1150	1150	1150	1150	1150
(Electrode Power) Output	%		33	33	32	33	32	31	32	31
(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	0.2	0.1	0.4
Kiln Temp Setpoint	° C		850	850	850	850	850	850	850	850
Kiln Actual (Middle) Temp	° C		851	849	849	852	850	850	849	849
Kiln Power Output	%		27	29	25	27	30	28	23	14
Kiln Control Mode	A or M		A	A	A	A	A	A	A	A
Discharge Can Power	kW		0.2	0.2	0.2	0.3	0.2	0.3	0.2	0.2
Discharge Can Temp Setpoint	° C		750	750	750	750	750	750	750	750
Discharge Can Power Output	%		51	47	43	60	51	58	43	53
Overflow Heater Power	kW		2.1	2.1	1.8	2.3	2.1	2.3	1.9	2.0
Overflow Heater Setpoint	° C		1150	1150	1150	1150	1150	1150	1150	1150
Overflow Heater Power Output	%		73	72	67	75	71	75	68	70
Time	hh:mm		1600	1700	1802	1905	2007	2106	2200	2308

Reviewed and Approved: R W Gelsz Date: 8/1/02

sheet 7 of 12

Data Sheet #1: Priority & Electrical Data

RSM-F1	By:		[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	Date:		7/31/02	7/31/02	7/31/02	7/31/02	7/31/02	7/31/02	7/31/02	7/31/02
	Time		0001	0058	0214	0300	0400	0500	0600	0700
DESCRIPTION	UNITS	Range								
Priority Data										
Melt Temperature (T1, control)	° C	1125 - 1175	1150	1147	1152	1149	1150	1148	1150	1149
Melt Temperature (T2)	° C	1125 - 1175	1154	1148	1153	1142	1147	1145	1145	1145
Plenum Temperature	° C	400 - 600	629	588	587	547	565	581	591	580
Cold Cap Coverage	%	> 75	40	60	95	100	90	90	85	90
Slurry Pool Coverage of Cap	%		50	20	20	20	20	20	25	20
Cold Cap Flexibility Visible	y or n		N	Y	Y	N	Y	Y	Y	Y (1/2)
Number of Vents	#		3	4	3	0	2	3	2	2
Cold Cap Thickness	inch		1/4	0 - 1"	1/4	?	1/4 - 1"	1/2 - 1"	1/4 - 1"	1/4 - 1"
Phase Separation (Note in LRE	y or n		N	–	–	–	–	–	–	–
Glass Pouring	y or n		N	N	N	N	N	slow drips	N	steady drips
Electrical Data										
Electrode Potential	Volts		32.7	35.5	32.9	31.0	34.5	34.9	32.8	32.9
Electrode Current	Amps		67.7	67.1	64.2	62.7	62.9	65.2	60.6	63.7
Electrode Power	kW		2.6	2.4	2.1	1.9	2.1	2.3	2.0	2.1
Melt Resistance	Ω		.558	0.523	.516	.494	.550	.539	.539	.522
Melt (Electrode) Setpoint Temp	° C		1150	1150	1150	1150	1150	1150	1150	1150
(Electrode Power) Output	%		34	34	33	32	31	33	31	32
(Electrode) Control Mode	A or M		A	A	A	A	A	A	A	A
Kiln Power	kW		0	0.2	0.2	0.2	0.2	0.2	.2	0.2
Kiln Temp Setpoint	° C		850	850	850	850	850	850	850	850
Kiln Actual (Middle) Temp	° C		850	850	850	851	851	849	851	844
Kiln Power Output	%		0	31	25	25	27	25	25	25
Kiln Control Mode	A or M		A	A	A	A	A	A	A	A
Discharge Can Power	kW		0.2	0.3	0.3	0.3	0.3	0.3	0.2	0.2
Discharge Can Temp Setpoint	° C		750	750	750	750	750	750	750	750
Discharge Can Power Output	%		48	56	56	55	59	53	52	47
Overflow Heater Power	kW		2.0	2.2	2.3	2.1	2.4	2.0	2.4	2.0
Overflow Heater Setpoint	° C		1150	1150	1150	1150	1150	1150	1150	1150
Overflow Heater Power Output	%		70	73	75	71	77	69	78	70
Time	hh:mm		0004	0101	0215	0303	0402	0504	0602	0701

Reviewed and Approved: [signature] Date: 7/31/02 sheet 8 of 12

Data Sheet #1: Priority & Electrical Data

RSM-F1	By:		WCB	WCB	TDP	TDP	SAP	SAP	SAP	SAP
	Date:		7/31/02	7/31/02	7/31/02	7/31/02	7/31/02	7/31/02	7/31/02	7/31/02
	Time		0801	0915	1305	1451	1556	1655	1801	1908
DESCRIPTION	UNITS	Range								
Priority Data										
Melt Temperature (T1, control)	° C	1125 - 1175	1149	1152	1148	1152	1149	1150	1147	1152
Melt Temperature (T2)	° C	1125 - 1175	1150	1153	1154	1159	1155	1142	1152	1158
Plenum Temperature	° C	400 - 600	592	566	698	619	630	600	595	724
Cold Cap Coverage	%	> 75	80	90	20	35	40	80	90	0
Slurry Pool Coverage of Cap	%		30	20	10	10	20	10	20	0
Cold Cap Flexibility Visible	y or n		N	N	Y	Y	Y	Y	Y	Y
Number of Vents	#		2	2	3	3	5	4	4	0
Cold Cap Thickness	inch		—	—	—	—	—	—	—	—
Phase Separation (Note in LRE	y or n		N	N	—	—	N	N	N	N
Glass Pouring	y or n		N	N	N	Y	N	N	N	N
Electrical Data										
Electrode Potential	Volts		31.8	32.3	39	40	41.6	39.7	46.6	30.9
Electrode Current	Amps		64.1	60.1	77.0	71	71.9	65.6	76.9	60.8
Electrode Power	kW		2.0	2.0	3.1	2.8	3.0	2.5	3.7	1.9
Melt Resistance	Ω		0.494	0.532	0.518	0.552	0.578	0.592	0.614	0.509
Melt (Electrode) Setpoint Temp	° C		1150	1150	1150	1150	1150	1150	1150	1150
(Electrode Power) Output	%		32	31	39	34	37	34	40	31
(Electrode) Control Mode	A or M		A	A	A	A	A	A	A	A
Kiln Power	kW		0.1	0.2	0	0	0.2	0.1	0.1	0
Kiln Temp Setpoint	° C		850	850	850	850	850	850	850	850
Kiln Actual (Middle) Temp	° C		851	851	865	848	851	849	849	860
Kiln Power Output	%		24	29	0	2	27	17	18	0
Kiln Control Mode	A or M		A	A	A	A	A	A	A	A
Discharge Can Power	kW		0.3	0.2	0.2	0.2	0.3	0.3	0.2	0.2
Discharge Can Temp Setpoint	° C		750	750	750	750	750	750	750	750
Discharge Can Power Output	%		58	50	48	48	56	56	52	50
Overflow Heater Power	kW		2.4	2.3	2	1.9	2.4	2.0	2.0	1.9
Overflow Heater Setpoint	° C		1150	1150	1150	1150	1150	1150	1150	1150
Overflow Heater Power Output	%		76	75	69	69	76	70	71	69
Time	hh:mm		0806	0918	1306	1451	1558	1659	1804	1908

Reviewed and Approved: R.W. Hale Date: 8/1/02 sheet 9 of 12

Data Sheet #1: Priority & Electrical Data

RSM-F1	By:									
	Date:		7/31/02	7/31/02	7/31/02	7/31/02	7/31/02	8/1/02	8/1/02	8/1/02
	Time		2002	2059	2206	2258	2356	0106	0211	0307
DESCRIPTION	UNITS	Range								
Priority Data					*		*			
Melt Temperature (T1, control)	° C	1125 - 1175	1150	1152	1154	1149	1154	1149	1145	1147
Melt Temperature (T2)	° C	1125 - 1175	1156	1145	1156	1136	1138	1149	1140	1155
Plenum Temperature	° C	400 - 600	648	628	672	622	542	529	604	474
Cold Cap Coverage	%	> 75	50	75	60	90	90	95	40	100
Slurry Pool Coverage of Cap	%		50	25	25	20	20	20	10	5
Cold Cap Flexibility Visible	y or n		Y	N	Y	Y	N	N	Y	N
Number of Vents	#		3	3	3	4	2	0	several	0
Cold Cap Thickness	inch		1/2	1/2	1/2	3/4	1"	>1"	1/2"	?
Phase Separation (Note in LRE	y or n		N	N	N	N	N	—	-	Y
Glass Pouring	y or n		N	N	N	N	N	N	N	N
Electrical Data										
Electrode Potential	Volts		41.4	46.3	49.7	41.7	38.6	30.7	33.5	32.6
Electrode Current	Amps		68.7	70.3	75.3	70.4	60.3	55.4	61.0	61.5
Electrode Power	kW		2.8	3.3	3.7	3.0	2.3	1.7	2.1	2.0
Melt Resistance	Ω		.587	.653	.626	0.602	.668	.561	.553	.553
Melt (Electrode) Setpoint Temp	° C		1150	1150	1150	1150	1150	1150	1150	1150
(Electrode Power) Output	%		35	36	38	36	31	28	31	31
(Electrode) Control Mode	A or M		A	A	A	A	A	A	A	A
Kiln Power	kW		0	0	0	0	0	0.2	0.0	0.2
Kiln Temp Setpoint	° C		850	850	850	850	850	850	850	850
Kiln Actual (Middle) Temp	° C		856	853	869	867	858	851	851	848
Kiln Power Output	%		0	0	0	0	0	30	13	26
Kiln Control Mode	A or M		A	A	A	A	A	A	A	A
Discharge Can Power	kW		0.2	0.3	0.2	0.2	0.1	0.2	0.3	0.2
Discharge Can Temp Setpoint	° C		750	750	750	750	750	750	750	750
Discharge Can Power Output	%		51	55	47	49	34	50	53	50
Overflow Heater Power	kW		2.0	1.8	2.1	1.9	1.7	2.3	2.1	2.0
Overflow Heater Setpoint	° C		1150	1150	1150	1150	1150	1150	1150	1150
Overflow Heater Power Output	%		70	67	72	67	65	75	72	69
Time	hh:mm		2005	2102	2209	2300	0000	0107	0213	0308

* Glass is foaming
* May be starting a feed bridge

Date: 8-1-02 sheet 10 of ___

Reviewed and Approved: [signature]

Data Sheet #1: Priority & Electrical Data

RSM-F1	By:		[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	TOP	TOP	TOP
	Date:		8/01/02	8/01/02	8/01/02	08/01/02	08/01/02	8/1/02	8/1/02	8/1/02
	Time		0412	0507	0602	0702	0818	1007	1139	1200
DESCRIPTION	UNITS	Range								
Priority Data										
Melt Temperature (T1, control)	° C	1125 - 1175	1152	1149	1151	1150	1169	1150	1150	1152
Melt Temperature (T2)	° C	1125 - 1175	1143	1137	1152	1150	1163	1140	1147	1142
Plenum Temperature	° C	400 - 600	426	598	559	512	484	606	605	630
Cold Cap Coverage	%	> 75	100	50	95	95-100	95	~~[illegible]~~ 20	30	25
Slurry Pool Coverage of Cap	%		0	0	40	?	0	20	10	20
Cold Cap Flexibility Visible	y or n		n	n	n	n	n	n	y	y
Number of Vents	#		0	0	0	0	1	2	5	4
Cold Cap Thickness	inch		several?	?	?	?	2?	1	–	1
Phase Separation (Note in LRB	y or n		–	–	–	–	–	–	–	–
Glass Pouring	y or n		no	no	N	Y	N	N	N	N
Electrical Data										
Electrode Potential	Volts		29.8	28.3	30.6	29.0	~~48.5~~ 27.5	38	39	36
Electrode Current	Amps		58.7	59.7	61.7	60.4	~~47.0~~ 27.0	69	69	64
Electrode Power	kW		1.8	1.7	1.9	1.8	~~1.0~~ 21.6	2.7	2.9	2.4
Melt Resistance	Ω		0.523	0.501	0.495	0.486	0.475	0.553	0.574	0.552
Melt (Electrode) Setpoint Temp	° C		1150	1150	1150	1150	1150	1150	1150	1150
(Electrode Power) Output	%		30	30	31	31	29	35	35	33
(Electrode) Control Mode	A or M		A	A	A	A	A	A	A	A
Kiln Power	kW		0.1	0.0	0.0	0.0	0.1	0	0	0.1
Kiln Temp Setpoint	° C		850	850	850	850	850	850	850	850
Kiln Actual (Middle) Temp	° C		851	855	853	848	846	855	848	850
Kiln Power Output	%		18	0	0	15	22	0	0	17
Kiln Control Mode	A or M		A	A	A	A	A	A	A	A
Discharge Can Power	kW		0.3	0.2	0.3	0.2	0.2	0.3	0.3	0.3
Discharge Can Temp Setpoint	° C		750	750	750	750	750	750	750	750
Discharge Can Power Output	%		57	~~[illegible]~~	60	49	48	50	57	55
Overflow Heater Power	kW		1.9	1.9	2.0	2.1	1.9	2.3	2.3	2.2
Overflow Heater Setpoint	° C		1150	1150	1150	1150	1150	1150	1150	1150
Overflow Heater Power Output	%		68	68	70	71	68	75	75	74
Time	hh:mm		0415	0509	0604	0706	0821	1009	1139	1201

Reviewed and Approved: [illegible] Date: 8/1/02 sheet 11 of 12

Data Sheet #1: Priority & Electrical Data

RSM-F1	By:		TDP	TDP						
	Date:		8/1/02	8/1/02						
	Time		1410	1500						
DESCRIPTION	UNITS	Range								
Priority Data										
Melt Temperature (T1, control)	° C	1125 - 1175	1151	1148						
Melt Temperature (T2)	° C	1125 - 1175	1148	1160						
Plenum Temperature	° C	400 - 600	623	608						
Cold Cap Coverage	%	> 75	40	50						
Slurry Pool Coverage of Cap	%		20	20						
Cold Cap Flexibility Visible	y or n		Y	Y						
Number of Vents	#		4	3						
Cold Cap Thickness	inch		1	1-2						
Phase Separation (Note in LRE	y or n		—	—						
Glass Pouring	y or n		N	Y						
Electrical Data										
Electrode Potential	Volts		35.8	36.8						
Electrode Current	Amps		65.5	71.5						
Electrode Power	kW		2.3	2.6						
Melt Resistance	Ω		0.541	0.526						
Melt (Electrode) Setpoint Temp	° C		1150	1150						
(Electrode Power) Output	%		37	37						
(Electrode) Control Mode	A or M		A	A						
Kiln Power	kW		0.1	0.2						
Kiln Temp Setpoint	° C		850	850						
Kiln Actual (Middle) Temp	° C		850	850						
Kiln Power Output	%		17	10						
Kiln Control Mode	A or M		A	A						
Discharge Can Power	kW		0.1	0.2						
Discharge Can Temp Setpoint	° C		750	750						
Discharge Can Power Output	%		58	51						
Overflow Heater Power	kW		2.2	1.9						
Overflow Heater Setpoint	° C		1150	1150						
Overflow Heater Power Output	%		74	69						
Time	hh:mm		1410	1507						

Reviewed and Approved: [signature] Date: 8/1/02 sheet 12 of 12

RSM Manual Data Sheets: Routine Status Sheet

Data Sheet #2: Routine Status Sheet

RSM-F1		By:	WCB	WCB	WCB	WCB	[illegible]	[illegible]	[illegible]	[illegible]
		Date:	7/28/02	7/28/02	7/28/02	7/28/02	7/28/02	7/28/02	7/28/02	7/28/02
		Time:	1300	1400	1501	1554	1702	1757	1859	2004
DESCRIPTION	**units**	**range**								
Feed Nozzle Cooling Flow	gpm	.5 - 1(reg FNT)	.73	.72	.7	.7	.7	.7	.7	.7
Film Cooler Air Supply	scfm	1-10 (reg OGT)	6.7	6.5	6.5	6.5	6.5	6.5	6.5	6.6
Film Cooler Pressure	psig	20-50	8	8	8	8	8	8	8	9
Melter Vacuum-Magnehelic	in. H_2O	0.1 - 2.0	2.5	1.5	1.8	2	3	2	2	2.2
EVS ΔP	in. H_2O		0	0	0	0	0	0	-2.8	-2.9
Film Cooler ΔP	in. H_2O		0	0	0	0	0	0	-0.1	-0.1
HEME ΔP	in. H_2O		1.7	0	0.3	.4	1.2	0.1	1.0	1.0
System ΔP	in. H_2O		0.0	0	0	0	0	0	-1.5	-1.6
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	3	2.9	2.9	3.0	3	3	3	2.8
EVS Scrub Tank Volume	gallon	~~35 - 50~~	97L	97L	99L	99L	99L	99	100	100
EVS Nozzle Pressure	psi	50 - 55	50	50	50	49	50	50	50	50
EVS Nozzle Flow	gpm		2.7	2.7	2.7	2.7	2.7	2.7	2.7	2.7
EVS Scrub Solution pH	pH	> 9	NA	NA	NA	NA	—	—	—	—
OG Control Valve position	%		NA	NA	NA	NA	—	—	—	—
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	%		OK	OK	OK	OK	OK	OK	OK	OK
Blower Cooling Flow	gpm	1 - 1.5	1.8	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Feed Nozzle Temp (FNT)	° C	< 40	30	30	29	29	30	30	31	29
Off-Gas Temp (OGT)	° C	< 250	144	140	125	124	98	123	102	98
Post EVS Off-Gas Temp	° C	< 50	34	35	35	34	33	34	34	33
Post HEME Off-Gas Temp	° C	< 50	32	34	35	34	33	33	34	32
Scrub Liquid Temp (SLT)	° C	<40	32	35	34	34	32	32	32	33
EVS HX Cooling Temp	° C	< 30	33	34	34	32	32	34	34	32
Feed Pump Setting	%		15	14	12	0	10	13	12	11
Feed Pump Control Mode	A or M		A	A	A	A	A	A	A	A
Feed Pressure	psi	<1.0	—	—	—	—	0	—	—	—
Feed Tank Wt	Kg	decreasing	224.1	221.4	219.3	218.2	217.0	215	213	211
Overflow Temp	° C	1125 - 1100	1147	1149	1150	1152	1147	1151	1152	1149
Discharge Can Temp	° C	750 - 850	738	749	750	748	747	750	750	750
Glass Scale	Kg	< 10	0	1.4	1.85	1.90	2.92	2.93	3.52	4.11
Alarm Condition	On/Off	off	off	off	off	off	off	off	off	OFF
time	hh:mm		1307	1407	1512	1558	1710	1803	1904	2009

Reviewed and Approved: [illegible] Date: 8/1/02

sheet 1 of 12

Data Sheet #2: Routine Status Sheet

RSM-F1		By:	[illegible]	[illegible]	[illegible]	SCL	[illegible]	[illegible]	[illegible]	[illegible]
		Date:	7/28/02	7/28/02	7/28/02	7/29/02	7/29/02	7/29/02	7/29/02	7/29/02
		Time:	2106	2202	2306	0007	0102	0200	0312	0403
DESCRIPTION	**units**	**range**								
Feed Nozzle Cooling Flow	gpm	.5 - 1(reg FNT)	~~6.0~~ 0.7	0.7	0.7	0.7	0.68	0.69	0.69	.68
Film Cooler Air Supply	scfm	1-10 (reg OGT)	6.5	6.6	6	6	6	6	6	6
Film Cooler Pressure	psig	20-50	9	9	7	7	7	7	7	7
Melter Vacuum-Magnehelic	in. H2O	0.1 - 2.0	3	2.5	2	2	1.8	1.7	2.1	2.5
EVS ΔP	in. H2O		-3.0	-3.0	-3.1	-3.1	-3.0	-3.0	-3.1	-3.1
Film Cooler ΔP	in. H2O		-0.1	-0.1	-.1	-0.1	-0.11	-0.13	-.18	-0.13
HEME ΔP	in. H2O		.9	0.9	0.9	0.9	0.91	0.89	0.79	0.80
System ΔP	in. H2O		-1.7	-1.6	-1.9	-1.9	-1.75	-1.75	-2.05	-2.0
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	3	2.9	3.0	3.0	3.0	3.0	2.95	2.9
EVS Scrub Tank Volume	gallon	35 - 50	101	102	102	102	103	103	104	104
EVS Nozzle Pressure	psi	50 - 55	50	50	50	50	50	50	50	50
EVS Nozzle Flow	gpm		2.7	2.7	2.7	2.7	2.8	2.8	2.8	2.8
EVS Scrub Solution pH	pH	> 9	—	—	—	—	—	—	—	—
OG Control Valve position	%		—	—	—	—	—	—	—	—
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	%		OK	OK	OK	OK	3	3	3	
Blower Cooling Flow	gpm	1 - 1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Feed Nozzle Temp (FNT)	° C	< 40	29	29	29	29	29	29	30	29
Off-Gas Temp (OGT)	° C	< 250	105	90	102	96	114	105	83	84
Post EVS Off-Gas Temp	° C	< 50	32	32	31	30	30	31	30	30
Post HEME Off-Gas Temp	° C	< 50	32	31	30	31	29	30	30	29
Scrub Liquid Temp (SLT)	° C	<40	32	30	30	30	30	31	30	29
EVS HX Cooling Temp	° C	< 30	32	32	30	30	30	31	30	29
Feed Pump Setting	%		10	11	11	11	11	11	9.0	10.0
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
Feed Tank Wt	Kg	decreasing	209.0	207.0	205	203	201.5	200.0	197.5	196.0
Overflow Temp	° C	1125 - 1100	1150	1150	1150	1159	1156	1149	1149	1150
Discharge Can Temp	° C	750 - 850	750	750	750	749	750	750	751	750
Glass Scale	Kg	< 10	5.03	5.15	5.76	6.80	7.15	7.76	9.64	0.17
Alarm Condition	On/Off	off	off	off	off	OFF	off	off	off	off
time	hh:mm		2112	2219	2312	0016	0111	0205	0316	0409

Reviewed and Approved: [illegible] Date: 8/1/02

sheet 2 of 12

* re-tared
*2 -3.0

Data Sheet #2: Routine Status Sheet

RSM-F1		By:	CRG	TJJ	TRP	WCB	WCB	WCB	WCB	WCB
		Date:	7/29/02	7/29/02	7/29/02	7/29/02	7/29/02	7/29/02	7/29/02	7/29/02
		Time:	0515	0603	0808	0950	1101	1219	1308	1400
DESCRIPTION	**units**	**range**								
Feed Nozzle Cooling Flow	gpm	.5 - 1(reg FNT)	0.7	0.63	0.7	0.68	.7	.72	.72	.7
Film Cooler Air Supply	scfm	1-10 (reg OGT)	6	6	6	6	5.8	5.9	6.2	5.8
Film Cooler Pressure	psig	20-50	7	7	7	7	7	7	7	7
Melter Vacuum-Magnehelic	in. H2O	0.1 - 2.0	1.9	1.9	2	2	2.5	2.5	3.5	3.5
EVS ΔP	in. H2O		-3	-3.2	-2.9	-2.9	~~-2.8~~	-3	-2.88	-2.99
Film Cooler ΔP	in. H2O		-.097	-0.13	-0.9	-.09	-0.4	-.1	-.1	-.13
HEME ΔP	in. H2O		1.046	0.81	1.02	.98	.88	.87	.88	.89
System ΔP	in. H2O		-1.486	-2.0	-1.59	-1.54	-1.65	-1.78	-1.73	-1.71
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	2.7	2.8	2.8	3	3	3.1	3.2	3.2
EVS Scrub Tank Volume	gallon	35 - 50	105	105	105L	105L	110L	110L	112L	112L
EVS Nozzle Pressure	psi	50 - 55	50	50	50	50	50	50	50	50
EVS Nozzle Flow	gpm		2.8	2.8	2.75	2.7	2.75	2.7	2.7	2.7
EVS Scrub Solution pH	pH	> 9	✓	–	–	NA	NA	NA	NA	NA
OG Control Valve position	%		–	–	–	NA	NA	NA	NA	NA
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	%		23 ok	3 ok		OK	OK	OK	OK	OK
Blower Cooling Flow	gpm	1 - 1.5	1.4	1.4	1.5	1.5	1.5	1.5	1.6	1.5
Feed Nozzle Temp (FNT)	° C	< 40	29	30	29	29	28	29	29	29
Off-Gas Temp (OGT)	° C	< 250	97	81	156	171	122	112	121	107
Post EVS Off-Gas Temp	° C	< 50	29	29	29	30	30	31	32	32
Post HEME Off-Gas Temp	° C	< 50	29	29	29	29	30	29	30	32
Scrub Liquid Temp (SLT)	° C	<40	30	30	29	30	31	31	31	32
EVS HX Cooling Temp	° C	< 30	30	30	29	31	30	31	31	31
Feed Pump Setting	%		10.0	10.0	0	10	10.5	10.5	10.5	10.7
Feed Pump Control Mode	A or M		A	A	A	A	A	A	A	A
Feed Pressure	psi	<1.0	0	0	0	0.2	0.2	0.2	0.1	0.2
Feed Tank Wt	Kg	decreasing	144.4	143.0	225.7	223.0	221.2	218.7	217.7	216.2
Overflow Temp	° C	1125 - 1100	1150	1150	1150	1150	1151	1155	1149	1150
Discharge Can Temp	° C	750 - 850	749	754	752	750	750	750	750	752
Glass Scale	Kg	< 10	0.80	1.09	2.0	0 *	7	7.15	1.96	
Alarm Condition	On/Off	off	off	off	off	off	off	off	off	off
time	hh:mm		0515	0608	807	1000	1108	1227	1314	

Reviewed and Approved: R.W. Gale Date: 8/1/02 sheet 3 of 12

Data Sheet #2: Routine Status Sheet

RSM-F1		By:	WCB	WCB	RW	RW	RW	RW	RW	RW
		Date:	7/29/02	7/29/02	7/29/02	7/29/02	7/29/02	7/29/02	7/29/02	7/29/02
		Time:	1500	1554	1704	1803	1904	1958	2101	2201
DESCRIPTION	**units**	**range**								
Feed Nozzle Cooling Flow	gpm	.5 - 1(reg FNT)	.75	.73	.7	.7	.7	.7	.7	.7
Film Cooler Air Supply	scfm	1-10 (reg OGT)	x6.9	x6.9	5.9	5.8	5.8	5.8	5.8	6
Film Cooler Pressure	psig	20-50	6.5	7	7	7	7	7	7	7
Melter Vacuum-Magnehelic	in. H_2O	0.1 - 2.0	3.0	2.2	2	2	2	2	2	2
EVS ΔP	in. H_2O		-2.96	-2.99	-3.1	-3.1	-2.99	-3.1	-3.1	-3.1
Film Cooler ΔP	in. H_2O		-.13	-.10	-.25	-.12	-.1	-.12	-.13	-.12
HEME ΔP	in. H_2O		.86	.91	.75	.78	.78	.81	.8	.8
System ΔP	in. H_2O		-1.78	-1.67	-1.8	-2.0	-1.9	-1.7	-1.9	-2.0
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0
EVS Scrub Tank Volume	gallon	35 - 50	112L	114L	113	113	113	114	114	114
EVS Nozzle Pressure	psi	50 - 55	50	48	50	50	50	50	50	50
EVS Nozzle Flow	gpm		2.7	2.7	2.7	2.7	2.7	2.7	2.7	2.7
EVS Scrub Solution pH	pH	> 9	NA	NA	NA	NA	NA	NA	NA	NA
OG Control Valve position	%		NA	NA	NA	NA	NA	NA	NA	NA
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	%		OK	OK	OK	OK	OK	OK	OK	OK
Blower Cooling Flow	gpm	1 - 1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.6
Feed Nozzle Temp (FNT)	° C	< 40	27	29	30	29	30	29	29	29
Off-Gas Temp (OGT)	° C	< 250	107	126	100	106	114	102	91	84
Post EVS Off-Gas Temp	° C	< 50	32	32	33	33	34	33	32	32
Post HEME Off-Gas Temp	° C	< 50	32	33	34	34	34	34	34	32
Scrub Liquid Temp (SLT)	° C	<40	31	31	31	32	32	30	30	30
EVS HX Cooling Temp	° C	< 30	31	31	31	32	32	32	31	30
Feed Pump Setting	%		10.5	10.5	10.5	9.0	9.5	9.5	10.0	9.5
Feed Pump Control Mode	A or M		A	A	A	A	A	A	A	A
Feed Pressure	psi	<1.0	0.2	0.0	0.1	0.1	0.2	0.1	0.3	0.9
Feed Tank Wt	Kg	decreasing	214.6	213.7	212.0	210.0	208.5	207.0	205.5	203.5
Overflow Temp	° C	1125 - 1100	1149	1149	1147	1155	1152	1149	1149	1153
Discharge Can Temp	° C	750 - 850	750	749	752	750	749	752	750	750
Glass Scale	Kg	< 10	2.74	3.13	3.84	4.32	4.91	5.41	5.62	6.34
Alarm Condition	On/Off	off	off	off	off	off	off	off	off	off
time	hh:mm		1504	1601	1709	1809	1908	2002	2105	2205

Reviewed and Approved: R W Nelson Date: 8/1/02

sheet 4 of 12

missed 0000 Data during feed prep MJ

Data Sheet #2: Routine Status Sheet

RSM-F1		By:	SL	MJ	CRH	MJ	CRH	CRH	MJ	MJ
		Date:	7/29/02	7/30/02	7/30/02	7/30/02	7/30/02	7/30/02	7/30/02	7/30/02
		Time:	2305	0107	0206	0258	0404	0500	0602	0702
DESCRIPTION	**units**	**range**								
Feed Nozzle Cooling Flow	gpm	.5 - 1(reg FNT)	0.7	~~0.65~~	0.7	0.70	0.70	0.70	0.69	0.65
Film Cooler Air Supply	scfm	1-10 (reg OGT)	6	6	6	6	6	6	6	6
Film Cooler Pressure	psig	20-50	7	7	7	7	7	7	7	7
Melter Vacuum-Magnehelic	in. H2O	0.1 - 2.0	1.8	1.7	2.0	1.8	1.9	2.0	1.7	1.6
EVS ΔP	in. H2O		-3.0	-3.1	-3.09	-3.0	-3.0	-3.0	2.96	-2.9
Film Cooler ΔP	in. H2O		-0.1	-0.11	-.138	-0.12	-0.12	-0.125	-0.12	-.11
HEME ΔP	in. H2O		1.0	0.93	0.789	0.82	0.81	0.80	0.87	0.90
System ΔP	in. H2O		-1.9	-1.8	-1.96	-1.9	-1.9	-1.92	-1.7	-1.68
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	2.9	2.95	3.0	2.95	2.95	2.85	2.8	2.7
EVS Scrub Tank Volume	gallon	35 - 50	108	108	108	108	108	108	109	109
EVS Nozzle Pressure	psi	50 - 55	50	50	50	50	50	50	50	50
EVS Nozzle Flow	gpm		2.7	2.8	2.8	2.8	2.8	2.8	2.8	2.8
EVS Scrub Solution pH	pH	> 9	—	—	—	—	—	—	—	—
OG Control Valve position	%		—	—	—	—	—	—	—	—
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	%		OK		ok	3.8	ok	ok	3.8	ok
Blower Cooling Flow	gpm	1 - 1.5	1.5	1.55	1.5	1.5	1.5	1.5	1.5	1.4
Feed Nozzle Temp (FNT)	° C	< 40	29	30	30	30	29	29	28	29
Off-Gas Temp (OGT)	° C	< 250	116	118	77	95	83	97	114	108
Post EVS Off-Gas Temp	° C	< 50	32	29	30	30	29	29	30	30
Post HEME Off-Gas Temp	° C	< 50	32	30	29	30	29	29	29	30
Scrub Liquid Temp (SLT)	° C	<40	31	29	30	31	30	30	30	30
EVS HX Cooling Temp	° C	< 30	30	29	29	31	30	30	30	30
Feed Pump Setting	%		10	0	10.0	10.6	10.4	10.4	10.5	10.5
Feed Pump Control Mode	A or M		A	A	A	A	A	A	A	A
Feed Pressure	psi	<1.0	0.1	0.1		0	0.1	0.1	0	0
Feed Tank Wt	Kg	decreasing	201.5	233.5	231.5	230.0	228.0	226.5	225.0	222.7
Overflow Temp	° C	1125 - 1100	1150	1149	1153	1146	1150	1148	1149	1150
Discharge Can Temp	° C	750 - 850	749	750	730	745	752	745	750	750
Glass Scale	Kg	< 10	6.94	7.14	0.42	0.98	1.20	2.00	2.64	3.26
Alarm Condition	On/Off	off	off	off	off	off	off	off	off	off
time	hh:mm		2311	0107	0215	0304	0411	0505	0607	0707

Reviewed and Approved: [signature] Date: 7/30/02 sheet 5 of 12

Changed Peristaltic Tubing

Data Sheet #2: Routine Status Sheet

RSM-F1		By:	TBP	TBP	TBP	WCB	TBP	WCB	WCB	TBP
		Date:	7-30-02	7-30-02	7-30-02	7/30/02	7/30/02	7/30/02	7/30/02	7/30/02
		Time:	804	900	957	1055	1206	1303	1409	1517
DESCRIPTION	**units**	**range**								
Feed Nozzle Cooling Flow	gpm	.5 - 1(reg FNT)	0.7	0.7	0.7	0.72	0.7	0.72	.76	0.75
Film Cooler Air Supply	scfm	1-10 (reg OGT)	6	6	6	5.9	6	5.9	5.9	5.9
Film Cooler Pressure	psig	20-50	15	7.5	7.5	7	7	7	7	7.5
Melter Vacuum-Magnehelic	in. H_2O	0.1 - 2.0	1.5	1.5	1.5	1.5	1.5	1.8	1.8	2
EVS ΔP	in. H_2O		-3	-3	-3	-3	-3	-3	-3	-2.8
Film Cooler ΔP	in. H_2O		-1.25	-.113	-.129	-.117	-.125	-.125	-.10	-0.105
HEME ΔP	in. H_2O		0.8	-0.8	-.9	.85	0.8	0.8	0.78	-1.6
System ΔP	in. H_2O		-1.8	-1.8	-1.9	-1.9	-1.9	-1.93	-1.85	-1.6
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	3	3	3	3	3	3	3	3
EVS Scrub Tank Volume	gallon	35 - 50	110L	110L	110L	112L	112L	114L	116L	115L
EVS Nozzle Pressure	psi	50 - 55	50	50	50	50	50	50	50	50
EVS Nozzle Flow	gpm		2.75	2.75	2.75	2.7	2.7	2.7	2.7	2.7
EVS Scrub Solution pH	pH	> 9	—	—	—	NA	—	NA	NA	—
OG Control Valve position	%		—	—	—	NA	—	NA	NA	—
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	%		—	—	—	OK	OK	OK	OK	OK
Blower Cooling Flow	gpm	1 - 1.5	1.5	1.5	1.5	1.5	1.6	1.5	1.35	1.5
Feed Nozzle Temp (FNT)	° C	< 40	28	28	27	28	27	28	29	29
Off-Gas Temp (OGT)	° C	< 250	98	106	93	98	98	86	93	126
Post EVS Off-Gas Temp	° C	< 50	30	29	29	30	30	31	32	33
Post HEME Off-Gas Temp	° C	< 50	28	29	29	30	30	30	32	32
Scrub Liquid Temp (SLT)	° C	<40	30	29	29	30	30	29	31	33
EVS HX Cooling Temp	° C	< 30	30	29	30	30	30	30	31	32
Feed Pump Setting	%		0	10.5	10.5	10.5	10.5	10.5	10.5	10.5
Feed Pump Control Mode	A or M		A	A	A	A	A	A	A	A
Feed Pressure	psi	<1.0	0	0.1	0.5	0.1	0	0.1	0.80	0
Feed Tank Wt	Kg	decreasing	0	220	218.7	216.6	215	213	211.5	209.4
Overflow Temp	° C	1125 - 1100	1144	1150	1150	1149	1149	1150	1149	1150
Discharge Can Temp	° C	750 - 850	749	749	749	751	751	750	750	749
Glass Scale	Kg	< 10	3.81	4.28	4.97	5.21	5.92	6.43	7.24	7.82
Alarm Condition	On/Off	off	OFF	OFF	OFF	OFF	OFF	OFF	OFF	OFF
time	hh:mm		804	900	1000	1100	1205	1308	1415	1523

Reviewed and Approved: [signature] Date: 8/1/02 sheet 6 of 12

Data Sheet #2: Routine Status Sheet

RSM-F1		By:	WCB	JW	SC	JW	SC	JW	JW	SC
		Date:	7/30/02	7/30/02	7/30/02	7/30/02	7/30/02	7/30/02	7/30/02	7/30/02
		Time:	1555	1701	1802	1900	2008	2059	2200	2305
DESCRIPTION	**units**	**range**								
Feed Nozzle Cooling Flow	gpm	.5 - 1(reg FNT)	.73	.75	0.73	0.73	0.7	.7	.7	0.7
Film Cooler Air Supply	scfm	1-10 (reg OGT)	6.8	5.8	5.8	5.8	5.8	5.8	5.9	6.0
Film Cooler Pressure	psig	20-50	6.5	7	6.5	7	7	7	7	7
Melter Vacuum-Magnehelic	in. H_2O	0.1 - 2.0	2.5	2.5	2.8	2.2	2.7	2.2	2.8	3.0
EVS ΔP	in. H_2O		-2.95	-3.1	-3.00	-2.6	-2.9	-2.9	-3.0	-3.3
Film Cooler ΔP	in. H_2O		-.113	-.11	-0.11	-.09	-0.1	-.11	-.13	-0.11
HEME ΔP	in. H_2O		.848	.79	0.78	1.2	0.8	.9	.80	.79
System ΔP	in. H_2O		-1.71	-1.9	-1.96	-1.1	-1.9	-1.6	-1.9	-2.28
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	3	3 JW	3	3	2.9	3	3	3
EVS Scrub Tank Volume	gallon	35 - 50	114L	~~110~~ 115	115	115	115	115	115	115
EVS Nozzle Pressure	psi	50 - 55	50	50	50	50	50	50	50	50
EVS Nozzle Flow	gpm		2.7	2.7	2.7	2.7	2.8	2.7	2.7	2.7
EVS Scrub Solution pH	pH	> 9	NA	NA	N/A	NA	N/A	NA	NA	NA
OG Control Valve position	%		NA	NA	N/A	NA	N/A	NA	NA	NA
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	%		OK	OK	OK	OK	OK	OK	OK	OK
Blower Cooling Flow	gpm	1 - 1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Feed Nozzle Temp (FNT)	° C	< 40	28	29	29	29	28	29	29	29
Off-Gas Temp (OGT)	° C	< 250	121	100	107	126	93	114	91	84
Post EVS Off-Gas Temp	° C	< 50	33	32	32	33	32	30	30	29
Post HEME Off-Gas Temp	° C	< 50	33	34	33	34	32	31	30	29
Scrub Liquid Temp (SLT)	° C	<40	32	30	31	31	30	30	30	29
EVS HX Cooling Temp	° C	< 30	32	31	32	32	31	31	30	28
Feed Pump Setting	%		10.5	10.5	10.5	10.5	10.6	11.0	10.5	0
Feed Pump Control Mode	A or M		A	A	A	A	A	A	A	~~off~~ off
Feed Pressure	psi	<1.0	0.1	0.0	0.0	0.0	0.0	0.1	0.1	off
Feed Tank Wt	Kg	decreasing	208.5	206.5	205.0	203.0	201.0	199.0	197.5	213.0
Overflow Temp	° C	1125 - 1100	1146	1150	1143	1150	1155	1150	1149	1150
Discharge Can Temp	° C	750 - 850	749	750	747	749	752	751	745	751
Glass Scale	Kg	< 10	8.03	9.00	0.35	0.78	1.71	2.30	2.56	3.40
Alarm Condition	On/Off	off	off	off	off	off	off	off	off	off
time	hh:mm		1602	1710	1806	—	2012	2104	2206	2309

Reviewed and Approved: RW Geeb Date: 8/1/02 sheet 7 of 12

Data Sheet #2: Routine/Status Sheet

RSM-F1		By:	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
		Date:	7/30/02	7/31/02	7/31/02	7/31/02	7/31/02	7/31/02	7/31/02	7/31/02
		Time:	2357	0101	0207	0303	0402	0504	0604	0707
DESCRIPTION	**units**	**range**								
Feed Nozzle Cooling Flow	gpm	.5 - 1(reg FNT)	.7	0.68	0.68	0.66	0.65	0.64	0.63	0.61
Film Cooler Air Supply	scfm	1-10 (reg OGT)	6	6	6	6	6	6	6	6
Film Cooler Pressure	psig	20-50	7	7.5	7	7	7	7	7	7
Melter Vacuum-Magnehelic	in. H2O	0.1 - 2.0	2.6	2.6	2.5	2.7	2.1	2.4	2.0	2.7
EVS ΔP	in. H2O		-3.0	-3.0	-3.0	-3.1	-2.9	-3.1	-2.9	-3.1
Film Cooler ΔP	in. H2O		-.16	-.13	-.12	-.125	-.11	-.15	-.10	-.125
HEME ΔP	in. H2O		.87	.83	.85	.79	0.94	.82	.96	.82
System ΔP	in. H2O		-1.9	-1.89	-1.82	-2.0	-1.65	1.87	1.60	1.98
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	3	3	2.95	2.91	2.8	2.8	2.8	2.7
EVS Scrub Tank Volume	gallon	35 - 50	115	116	116	116	117	117	117	117
EVS Nozzle Pressure	psi	50 - 55	50	50.5	50.5	50.6	50.6	50.5	50.5	50.5
EVS Nozzle Flow	gpm		2.7	2.8	2.8	2.8	2.8	2.8	2.8	2.8
EVS Scrub Solution pH	pH	> 9	NA	–	–	–	–	–	–	–
OG Control Valve position	%		NA	–	–	–	–	–	–	–
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	%		OK	ok 3.6	ok	ok	ok	ok	ok	ok
Blower Cooling Flow	gpm	1 - 1.5	1.5	1.5	1.4	1.4	1.4	1.4	1.4	1.35
Feed Nozzle Temp (FNT)	° C	< 40	29	29	28	28	27	27	27	29
Off-Gas Temp (OGT)	° C	< 250	116	98	109	98	126	97	124	95
Post EVS Off-Gas Temp	° C	< 50	29	30	29	28	29	29	29	30
Post HEME Off-Gas Temp	° C	< 50	29	29	27	26	27	27	27	28
Scrub Liquid Temp (SLT)	° C	<40	29	29	29	28	29	29	29	29
EVS HX Cooling Temp	° C	< 30	29	29	30	29	30	30	30	30
Feed Pump Setting	%		11	10.6	10.5	10.4	10.4	10.4	10.4	10.4
Feed Pump Control Mode	A or M		A	A	A	A	A	A	A	A
Feed Pressure	psi	<1.0	0.1	0.1	0.0	0.0	0.0	0.0	0.0	0.0
Feed Tank Wt	Kg	decreasing	211.5	209.5	206.9	205.0	203.9	202.0	200.5	198.9
Overflow Temp	° C	1125 - 1100	1154	1149	1151	1142*	1150	1152	1150	1156
Discharge Can Temp	° C	750 - 850	755	750	748	749	750	747	747	754
Glass Scale	Kg	< 10	3.6	4.25	4.77	5.32	6.13	6.62	7.15	7.77
Alarm Condition	On/Off	off	off	off	off	off	off	off	off	off
time	hh:mm		0000	0108	0213	0317	0407	0511	0612	0712

*had scale down longer than normal - may have cooled overflow port

Reviewed and Approved: [signature] Date: 7/31/02

sheet 8 of 12

Data Sheet #2: Routine Status Sheet

RSM-F1		By:	WCB	WCB	TRP	WCB	WCB	[illegible]	[illegible]	[illegible]
		Date:	7/31/02	7/31/02	7/31/02	7/31/02	7/31/02	7/31/02	7/31/02	7/31/02
		Time:	0807	0905	1300	1447	1551	1654	1800	1901
DESCRIPTION	**units**	**range**								
Feed Nozzle Cooling Flow	gpm	.5 - 1(reg FNT)	0.63	0.6	0.65	.68	.63	.75	.75	.75
Film Cooler Air Supply	scfm	1-10 (reg OGT)	6	5.9	5.9	5.9	5.4	5.8	5.8	5.8
Film Cooler Pressure	psig	20-50	7	7	7.5	7	9.5	7	7	7
Melter Vacuum-Magnehelic	in. H_2O	0.1 - 2.0	2.2	2.1	2	2.2	1	2.5	2.6	3.0
EVS ΔP	in. H_2O		-2.97	-2.91	-2.6	-2.6	-1.92	-2.6	-2.6	-3.0
Film Cooler ΔP	in. H_2O		-.11	-.11	-0.16	-0.11	-0.09	-.10	-.09	-.13
HEME ΔP	in. H_2O		.91	.94	-0.832	.79	1.18	1.0	.77	.78
System ΔP	in. H_2O		-1.75	-1.63	-1.5	-1.47	-.273	-1.1	-1.5	-1.9
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	3	3.1	3.5	3.9	3.1	2.9	3.0	2.9
EVS Scrub Tank Volume	gallon	35 - 50	117L	117L	118L	117L	117L	117	118	118
EVS Nozzle Pressure	psi	50 - 55	50	50	50	50	50	50	51	/
EVS Nozzle Flow	gpm		2.7	2.7	2.7	2.75	2.7	2.8	2.8	2.8
EVS Scrub Solution pH	pH	> 9	NA	NA	–	NA	NA	NA	NA	N/A
OG Control Valve position	%		NA	NA	–	NA	NA	NA	NA	N/A
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	Failed
Agitator Setting	%		OK	OK	ok	OK	OK	OK	OK	OK
Blower Cooling Flow	gpm	1 - 1.5	1.4	1.5	1.5	1.6	1.5	1.5	1.4	1.4
Feed Nozzle Temp (FNT)	° C	< 40	29	27	29	29	29	29	28	29
Off-Gas Temp (OGT)	° C	< 250	121	127	140	127	112	107	109	93
Post EVS Off-Gas Temp	° C	< 50	30	29	30	32	32	30	31	30
Post HEME Off-Gas Temp	° C	< 50	29	29	29	30	30	30	30	30
Scrub Liquid Temp (SLT)	° C	<40	31	30	29	30	31	30	29	29
EVS HX Cooling Temp	° C	< 30	30	30	30	31	32	32	32	29
Feed Pump Setting	%		10.4	10.4	11	11.5	11.7	12.0	12.0	12.0
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	0.0	0.0	0.0
Feed Tank Wt	Kg	decreasing	197.5	195.5	226.6	224.6	222.5	220.0	217.5	216.5
Overflow Temp	° C	1125 - 1100	1152	1153	1150	1149	1139	1146	1154	1164
Discharge Can Temp	° C	750 - 850	752	752	753	749	749	751	747	749
Glass Scale	Kg	< 10	8.13	8.81	0	0.85	1.69	2.74	3.15	3.49
Alarm Condition	On/Off	off	OFF	OFF	OFF	off	off	OFF	OFF	OFF
time	hh:mm		0815	0914	1305	1453	1559	1701		1911

Tubing being replaced

Reviewed and Approved: R W Johnson Date: 8/1/02 sheet 9 of 12

Data Sheet #2: Routine Status Sheet

RSM-F1		By:	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
		Date:	7/31/02	7/31/02	7/31/02	7/31/02	8/1/02	8/1/02	8/1/02	8/1/02
		Time:	2002	2102	2202	2258	0001	0100	0206	0331
DESCRIPTION	**units**	**range**								
Feed Nozzle Cooling Flow	gpm	.5 - 1(reg FNT)	0.7	0.7	0.7	0.7	0.7	0.71	0.71	.69
Film Cooler Air Supply	scfm	1-10 (reg OGT)	5.8	5.8	5.8	5.8	5.9	5.95	5.9	5.9
Film Cooler Pressure	psig	20-50	7	7	7	7	7	7	7	7
Melter Vacuum-Magnehelic	in. H_2O	0.1 - 2.0	2.5	2.6	2.4	3.5	2.2	2.2	2.4	2.3
EVS ΔP	in. H_2O		-2.6	-2.6	-2.4	-2.7	-2.62	-2.8	-2.7	-2.7
Film Cooler ΔP	in. H_2O		-0.1	-.13	-.09	-.13	-.12	-.11	-.14	-.13
HEME ΔP	in. H_2O		1.0	.80	.98	.78	.85	.86	.91	.81
System ΔP	in. H_2O		-1.2	-1.5	-1.06	-1.5	-1.4	-1.3	-1.6	-1.6
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	2.8	2.8	2.8	2.6	2.8	2.85	3	3
EVS Scrub Tank Volume	gallon	35 - 50	119 ~~124~~	119	120	120	120	120	120	121
EVS Nozzle Pressure	psi	50 - 55	52	51	51	51	51	51	51	51
EVS Nozzle Flow	gpm		2.8	2.8	2.8	2.8	2.8	2.8	2.8	2.8
EVS Scrub Solution pH	pH	> 9	N/A	N/A	NA	NA	NA	—	—	—
OG Control Valve position	%		N/A	N/A	NA	NA	NA	—	—	—
OG Control Valve mode	A or M		M	NA	M	M	M	M	M	M
Feed Pump (tubing) Condition			OK	OK	OK	OK	OK	OK	OK	OK
Agitator Setting	%		OK	OK	OK	OK	OK	OK	OK	OK
Blower Cooling Flow	gpm	1 - 1.5	1.4	1.4	1.4	1.4	1.4	1.4	1.4	1.4
Feed Nozzle Temp (FNT)	° C	< 40	29	29	28	29	27	27	27	27
Off-Gas Temp (OGT)	° C	< 250	109	111	151	113	117	107	88	76
Post EVS Off-Gas Temp	° C	< 50	30	32	32	32	31	29	29	29
Post HEME Off-Gas Temp	° C	< 50	30	31	30	30	30	29	29	28
Scrub Liquid Temp (SLT)	° C	<40	29	30	30	30	30	29	28	29
EVS HX Cooling Temp	° C	< 30	30	31	32	31	31	30	29	29
Feed Pump Setting	%		12.0	12.5	12.7	12.7	12.7	11.0	5.0	9
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
Feed Tank Wt	Kg	decreasing	215.5	213.5	211.0	209.0	206.0	204.6	203.9	201.4
Overflow Temp	° C	1125 - 1100	1154	1150	1150	1149	1143	1146	1150	1150
Discharge Can Temp	° C	750 - 850	749	749	755	754	743	751	749	749
Glass Scale	Kg	< 10	4.12	4.70	5.15	5.54	7.12	7.17	7.18	7.30
Alarm Condition	On/Off	off	off	off	off	off	off	off	off	off
time	hh:mm		2008	2106	2206	2302	0009	0105	0210	0336

Reviewed and Approved: [signature] CG ? Date: 8-1-02 sheet 10 of 12

Data Sheet #2: Routing Status Sheet

RSM-F1		By:	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	TDP	[illegible]	TDP
		Date:	8/1/02	8/1/02	8/1/02	8/1/02	8/1/02	8/1/02	8/1/02	8/1/02
		Time:	0403	0504	0601	0705	0820	1010	1131	1200
DESCRIPTION	**units**	**range**								
Feed Nozzle Cooling Flow	gpm	.5 - 1(reg FNT)	0.67	.65	0.65	0.66	.68	0.72	0.72	0.75
Film Cooler Air Supply	scfm	1-10 (reg OGT)	5.9	6.0	6.0	6.0	6	6	5.8	6
Film Cooler Pressure	psig	20-50	7	7	7	7	7	7	7	7
Melter Vacuum-Magnehelic	in. H2O	0.1 - 2.0	2.5	2.9	2.6	2.2	2.0	1.8	2.0	2
EVS ΔP	in. H2O		-2.9	-3.1	-2.8	-2.8	-2.9	-2.5	-2.5	-2.5
Film Cooler ΔP	in. H2O		-.125	-.13	-.13	-.13	-.13	-0.1	-0.11	-0.1
HEME ΔP	in. H2O		.84	.77	.77	.83	.76	0.97	0.88	.88
System ΔP	in. H2O		-1.8	-2.0	-1.65	-1.6	-1.3	-1.1	-1.26	-1.34
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	2.95	2.85	2.9	2.9	3.0	3	3.2	3
EVS Scrub Tank Volume	L ~~gallon~~	~~35 - 50~~	121	121	121	121	121	121L	121L	121L
EVS Nozzle Pressure	psi	50 - 55	51.1	51.1	51.1	51.1	51.1	51	51	51
EVS Nozzle Flow	gpm		2.8	2.8	2.8	2.80	2.8	2.7	2.7	2.65
EVS Scrub Solution pH	pH	> 9	—	—	—	—	—	—	NA	—
OG Control Valve position	%		—	—	—	—	—	—	NA	—
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	%		OK	OK	OK	OK	OK	OK	OK	OK
Blower Cooling Flow	gpm	1 - 1.5	1.35	1.3	1.35	1.3	1.4	1.4	1.5	1.5
Feed Nozzle Temp (FNT)	° C	< 40	27	27	28	27	27	27	29	25
Off-Gas Temp (OGT)	° C	< 250	74	53	77	74	93	140	137	125
Post EVS Off-Gas Temp	° C	< 50	27	27	27	27	28	29	30	30
Post HEME Off-Gas Temp	° C	< 50	27	27	27	25	27	27	29	29
Scrub Liquid Temp (SLT)	° C	<40	28	27	27	27	29	29	30	31
EVS HX Cooling Temp	° C	< 30	29	27	27	28	29	30	30	30
Feed Pump Setting	%		0	0	8.0	8.0	0	10	11.2	11.5
Feed Pump Control Mode	A or M		M	M	A	M	A	A	A	A
Feed Pressure	psi	<1.0	0.0	0	0	0	0	0	0	0
Feed Tank Wt	Kg	decreasing	201.0	201.0	199.8	199.0	197.8	196.9	195.0	193.9
Overflow Temp	° C	1125 - 1100	1149	1150	1150	1160	1135 *	1150	1149	1150
Discharge Can Temp	° C	750 - 850	747	754	756	757	597 *	750	749	749
Glass Scale	Kg	< 10	7.41	7.60	8.01	8.26	9.05	0.5	1.67	1.69
Alarm Condition	On/Off	off	off	off	off	off	off	off	off	off
time	hh:mm		0413	0509	0605	0715	0829	1007	1136	1201

* just changed out can so temps are low

Reviewed and Approved: [signature] Date: 8/01/02

sheet 11 of 12

1500

Data Sheet #2: Routine Status Sheet

RSM-F1		By:	TDP	WCB						
		Date:	8/1/02	8/1/02						
		Time:	1400	~~WCB~~						
DESCRIPTION	**units**	**range**								
Feed Nozzle Cooling Flow	gpm	.5 - 1(reg FNT)	0.8	0.68						
Film Cooler Air Supply	scfm	1-10 (reg OGT)	6	5.8						
Film Cooler Pressure	psig	20-50	7	7						
Melter Vacuum-Magnehelic	in. H_2O	0.1 - 2.0	2	2.8						
EVS ΔP	in. H_2O		-2.6	-2.6						
Film Cooler ΔP	in. H_2O		-0.1	-.09						
HEME ΔP	in. H_2O		0.86	0.79						
System ΔP	in. H_2O		-1.4	-1.48						
EVS HX Cooling Flow	gpm	1-5 (reg SLT)	3	3						
EVS Scrub Tank Volume	gallon	35 - 50	121L	121L						
EVS Nozzle Pressure	psi	50 - 55	51	51						
EVS Nozzle Flow	gpm		2.7	2.7						
EVS Scrub Solution pH	pH	> 9	—	NA						
OG Control Valve position	%		—	NA						
OG Control Valve mode	A or M		M	M						
Feed Pump (tubing) Condition			OK	OK						
Agitator Setting	%		OK	OK						
Blower Cooling Flow	gpm	1 - 1.5	1.5	1.35						
Feed Nozzle Temp (FNT)	° C	< 40	29	29						
Off-Gas Temp (OGT)	° C	< 250	130	119						
Post EVS Off-Gas Temp	° C	< 50	31	31						
Post HEME Off-Gas Temp	° C	< 50	30	30						
Scrub Liquid Temp (SLT)	° C	<40	31	30						
EVS HX Cooling Temp	° C	< 30	31	30						
Feed Pump Setting	%		10.2	10.2						
Feed Pump Control Mode	A or M			A						
Feed Pressure	psi	<1.0	0	0						
Feed Tank Wt	Kg	decreasing	205	204.5						
Overflow Temp	° C	1125 - 1100	1150	1152						
Discharge Can Temp	° C	750 - 850	749	752						
Glass Scale	Kg	< 10	2.69	3.0						
Alarm Condition	On/Off	off	off	off						
time	hh:mm		1409	1504						

Reviewed and Approved: [signature] Date: 8/1/02 sheet 12 of 12

RSM Manual Data Sheets: Off-Gas Monitoring Data

Data Sheet #3: Off-Gas Monitoring Data

RSM-F1		By:		TDP	TDP	TDP	[illegible]	[illegible]	[illegible]	[illegible]
		Date:		7/28/02	7/25/02	7/28/02	7/28/02	7/28/02	7/28/02	7/28/02
		Time:		1352	1511	1556	1704	1758	1900	2001
DESCRIPTION	**units**	Range								
Gas Chromatograph										
He	ppm		5491	5462	5312	5802	7046	6474	7105	6569
H_2	ppm	<1e4	—							
O_2	ppm		187785	193409	185566	196894	199248	194184	191572	195011
N_2	ppm		730138	743369	735203	765503	776342	770806	767973	777118
NO	ppm		—							
CO	ppm	<3E4	—							
CO_2	ppm		1769	2101	2075	960	2221	2298	2635	3008
N_2O	ppm		—							
~~NOx~~	~~ppm~~									
~~H_2 + CO~~	~~ppm~~	<2E4								
NOx Analyzer Computer										
Time	hh:mm		12:51	1356	1511	1557	1705	1801	1903	2005
NOx	ppm		13.866	13.180	6.5428	8.5295	0.9376	0.7562	0.1308	0.6842
NO	ppm		12.449	12.561	6.444	8.581	0.02131	0.05365	-0.0274	-0.0952
~~NOx Voltage~~	~~Volts~~									
NOx Instrument										
Sample Pressure	psig		4.2	4.2	4.2	4.2	4.15	4.15	4.15	4.15
Sample bypass flow	sccm		17	17	17	17	17	17	17	17
Ozone Pressure	psig		5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
MKS Mass Flow										
Ar Carrier, Chan-~~1~~ 2	lpm		3.98	3.99	3.98	3.98	3.98	3.97	3.98	3.98
He Tracer, Chan-~~2~~ 1	lpm		2.00	2.01	2.01	2.00	~~3.99~~ 2.00	2.00	2.00	2.00
Total Sample , Chan-3	lpm		5.01	5.00	5.00	5.00	5.0	5.00	5.00	5.00
Sample Pump*										
Vac Gauge	"Hg		1	1	1	1	1	1	1	1
Pres Gauge	psig	<15	7	5	8.5	8.5	9.0	8.5	8.2	8.4
Gas Cylinders Tank Pres#										
O_2	psig	>200	400	400	400	400	350	310	300	300
Ar GC	psig	>201	700	700	700	700	750	750	750	750
Ar Sweep #	psig	>202	1000/2000	900/2000	800/2000	800/2000	700/2000	600/2000	500/2000	420/2000
He GC	psig	>203	2300	2300	2300	2300	2400	2400	2400	2400
He Tracer #	psig	>204	450	440	360	320	1700	1700	1650	1600
time	hh:mm		13:15	14:00	1517	1601	1708	1802	1905	2007

~5500 None 209,000 780300 ~20ppm none ~1500ppm None ~~~2ppm~~ ~~~~ Is 6.2 Fixed >12 Fixed As goes < 75 42 NOx 2 tanks

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

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

sheet 1 of 11

Reviewed and Approved: [signature] Date: 8/1/02

Data Sheet #3: Off-Gas Monitoring Data

RSM-F1		By:	SH	SH	SH	SH	CRH	CRH	CRH	CRH
		Date:	7/28/02	7/28/02	7/28/02	7/28/02	7/29/02	7/29/02	7/29/02	7/29/02
		Time:	2107	2200	2303	2358	0101	0200	0259	0402
DESCRIPTION	**units**	Range								
Gas Chromatograph										
He	ppm		7199	6770	8682	8493	7863	7458	7985	7573
H_2	ppm	<1e4	—	—	—	—	23.44	21	27	15
O_2	ppm		198915	200513	202807	201700	199281	199111	198389	200869
N_2	ppm		783712	785211	788215	784456	780672	781840	783421	789389
NO	ppm		—	—	—	—	—	—	—	—
CO	ppm	<3E4	—	—	—	—	—	—	—	—
CO_2	ppm		3370	2651	2558	2718	3363	3131	1423	2368
N_2O	ppm		—	—	—	—	—	—	—	—
NOx	ppm		—	—	—	—	—	—	—	—
H_2 + CO	ppm	<2E4	—	—	—	—	—	—	—	—
NOx Analyzer Computer										
Time	hh:mm		2110	2203	2304	0002	0102	0203	0302	0406
NOx	ppm		0.6117	0.4392	1.2609	2.0505	1.774	2.117	0.54327	0.40902
NO	ppm		0.2099	-0.1025	0.8388	1.9442	0.68048	1.2521	0.18088	-0.087
NOx Voltage	Volts		—	—	—	—	—	—	—	—
NOx Instrument										
Sample Pressure	psig		4.15	4.15	4.15	4.15	4.15	4.15	4.15	4.15
Sample bypass flow	sccm		17	17	17	17	17	17	17	17
Ozone Pressure	psig		5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
MKS Mass Flow										
Ar Carrier, Chan-~~1~~2	lpm		3.98	3.98	3.98	3.98	3.98	3.98	3.99	3.99
He Tracer, Chan-~~2~~1	lpm		2.00	2.00	2.00	2.00	2.00	2.00	2.01	2.01
Total Sample , Chan-3	lpm		5.00	5.00	5.00	5.00	5.01	5.01	5.01	5.01
Sample Pump*										
Vac Gauge	"Hg		1	1	1	1	4	4.5	4.6	5.1
Pres Gauge	psig	<15	8	8.4	8.2	7.5	5.5	5.0	5.0	4.6
Gas Cylinders Tank Pres#										
O_2	psig	>200	260	250	220	200	1200	1200	1200	1150
Ar GC	psig	>201	750	750	750	750	750	750	750	750
2) Ar Sweep	psig	>202	310/2000	280/2000	180/2100	0/1800	0/1900	0/1850	0/1800	0/1720
He GC	psig	>203	2400	2400	2400	2400	2400	2400	2400	2400
He Tracer	psig	>204	1500/100	1500/100	1450/0	1400/0	1350/0	1300/0	1250/0	1200/0
time	hh:mm		2112	2205	2307	0005	0109	0205	3:06	4:07

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

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

sheet 2 of 11

Reviewed and Approved: R. W. Gale

Date: 8/1/02

Data Sheet #3: Off-Gas Monitoring Data

RSM-F1		By:	[illegible]	[illegible]	TPP	TPP	TPP	TPP	TPP	TPP
		Date:	7/29/02	7/29/02	7/29/02	7-29-02	7/29/02	7/29/02	7/29/02	7/29/02
		Time:	05:00	06:00	759	1100	1215	1363	1352	1455
DESCRIPTION	**units**	Range								
Gas Chromatograph										
He	ppm		8336	8180	6725	7823.79	7398.56	7435	0	0
H_2	ppm	<1e4	0	0	–				19.226	0
O_2	ppm		200525	0	0	197595	199941	195057	196575	192762
N_2	ppm		789161	0	0	850552	810448	801897	810460	803388
NO	ppm		–	–	–					
CO	ppm	<3E4	–	–	–					
CO_2	ppm		2221	2059	273.1	1868	3541	2898	2572	1972
N_2O	ppm		–	–	–	–				
NOx	ppm		–	–	–	–				
H_2 + CO	ppm	<2E4	–	–	–	–				
NOx Analyzer Computer										
Time	hh:mm		05:04	6:04	800	1058	1219	1317	1359	1443
NOx	ppm		1.4339	0.22478	-1.1	2.74	1.6461	2.5079	1.3833	1.1263
NO	ppm		0.78788	-0.29027	-1.2	0.6129	0.43861	0.65602	-0.23873	0.2320
NOx Voltage	Volts		–		–	–		–	–	
NOx Instrument										
Sample Pressure	psig		4.15	4.15	4.2	4.2	4.2	4.2	4.2	
Sample bypass flow	sccm		17	17	17	17	17	17	17	
Ozone Pressure	psig		5.0	5.0	5.0	5.0	5.0	5.0	5.0	
MKS Mass Flow										
Ar Carrier, Chan-~~1~~2	lpm		3.99	3.99	3.99	3.98	3.98	3.98	3.98	3.98
He Tracer, Chan-~~2~~1	lpm		2.00	2.01	2.01	2.01	2.01	2.01	2.00	2.01
Total Sample , Chan-3	lpm		4.99	4.96	5.01	5.01	5.01	5.01	5.01	5.00
Sample Pump*										
Vac Gauge	"Hg		5.1	5.4	~~7.5~~ 1	1	1	1	1	1
Pres Gauge	psig	<15	4.5	4.3	7.5	8.0	6.0	8.0	9.0	7.0
Gas Cylinders Tank Pres#										
O_2	psig	>200	1140	1140	1140	1140	1500	1500	1500	1500
Ar GC	psig	>201	750	750	720	720	720	710	710	710
Ar Sweep	psig	>202	0/1650	0/1550	0/1400	0/1400	0/1100	0/1000	0/1000	0/900
He GC	psig	>203	2350	2350	2350	2350	2350	2350	2350	2350
He Tracer	psig	>204	1170/0	1120/0	1000/0	950/0	900/0	850/0	800/0	750/0
time	hh:mm		5:05	0606	806	1105	1220	1319	1359	1457

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

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

sheet 3 of 11

Reviewed and Approved: [signature] Date: 8/1/02

NOx. Computer locked up @ 1443

Data Sheet #3: Off-Gas Monitoring Data

RSM-F1		By:	TDP	SD	SD	SD	SD	SD	SD	SD
		Date:	7/29/02	7/29/02	7/29/02	7/29/02	7/29/02	7/29/02	7/29/02	7/29/02
		Time:	1543	1701	1800	1910	1957	2101	2208	2311
DESCRIPTION	**units**	Range								
Gas Chromatograph										
He	ppm		0	0		9211	8414	9542	8835	0
H_2	ppm	<1e4	0	0		0	0	0	0	0
O_2	ppm		209553	186056		199941	197309	199843	200485	202441
N_2	ppm		850720	790304	Calibrating	775391	771166	779784	783820	790065
NO	ppm			0		0	0	0	0	0
CO	ppm	<3E4		0		0	0	0	0	0
CO_2	ppm		1678	0		2492	4578	1905	2849	2716
N_2O	ppm			—		0	0	0	0	0
NOx	ppm			—		—	—	—	—	—
H_2 + CO	ppm	<2E4		—		—	—	—	—	—
NOx Analyzer Computer										
Time	hh:mm		1443	—		1914	2001	2103	2203	2316
NOx	ppm		1.1763	—	Calibrating	1.9206	1.4572	1.1528	1.6035	2.7902
NO	ppm		0.2320	—		0.0403	0.2312	0.3574	0.6257	0.5529
NOx Voltage	Volts									
NOx Instrument										
Sample Pressure	psig			4.15	4.15	4.15	4.15	4.15	4.15	4.2
Sample bypass flow	sccm			17	18	17	17	17	17	17
Ozone Pressure	psig			5	5	5	5	5	5	5
MKS Mass Flow										
Ar Carrier, Chan-1	lpm		2.0	2.00	2.00	2.00	2.00	2.00	2.01	2.00
He Tracer, Chan-2	lpm		3.98	3.98	3.98	3.97	3.97	3.98	3.99	3.99
Total Sample , Chan-3	lpm		5.0	5.00	5.00	5.00	5.00	5.00	5.01	5.01
Sample Pump*										
Vac Gauge	"Hg		1	1	1	1	1	1	1	1
Pres Gauge	psig	<15	7	7.5	6	6	6.5	7	7.3	7.8
Gas Cylinders Tank Pres#										
O_2	psig	>200	1500	1450	1420	1380	1400	1350	1320	1300
Ar GC	psig	>201	720	750	750	750	750	750	750	750
Ar Sweep	psig	>202	690	760	700	620	550	500	450	300
He GC	psig	>203	2350	2350	2350	2400	2400	2400	2400	2400
He Tracer	psig	>204	700/0	700	650	620	600	560	590	500
time	hh:mm		1553	1706	1804	1912	2003	2104	2201	2314

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

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

sheet 4 of 11

Reviewed and Approved: RW Gates Date: 8/1/02

Data Sheet #3: Off-Gas Monitoring Data

RSM-F1		By:	CRH	CRH	CRH	CRH	CRH	CRH	CRH	CRH
		Date:	7/30/02	7/30/02	7/30/02	7/30/02	7/30/02	7/30/02	7/30/02	7/30/02
		Time:	1253	0200	0258	0358	0456	0558	0702	0755
DESCRIPTION	**units**	Range								
Gas Chromatograph										
He	ppm		0	0	0	0	0	0	40.2	0
H_2	ppm	<1e4	0	0	0	0	0	0	0	0
O_2	ppm		232580	204833	416113	214486	209621	211505	205191	198273
N_2	ppm		864199	801378	1166928	827467	814909	830131	0	0
NO	ppm		0	0	0	0	0	0	0	0
CO	ppm	<3E4	0	0	0	0	0	0	0	0
CO_2	ppm		600	2327	4931	0	0	0	0	0
N_2O	ppm		0	0	0	0	0	0	0	0
NOx	ppm		–	–	–	–	–	–	–	–
H_2 + CO	ppm	<2E4	–	–	–	–	–	–	–	–
NOx Analyzer Computer										
Time	hh:mm		1258	0204	0302	0402	0500	0602	0707	0800
NOx	ppm		-1.3078	0.73334	5.9088	1.0831	1.4092	1.7903	2.1183	0.62754
NO	ppm		-1.3145	0.022046	~~1.7331~~ CO 1.46331 CRH	0.45876	0.94	0.77339	1.3129	0.45593
NOx Voltage	Volts		–	–		–	–	–	–	–
NOx Instrument										
Sample Pressure	psig		4.15	4.15	4.15	4.15	4.15	4.15	4.15	4.15
Sample bypass flow	sccm		17	17	14	17	17	17	17	17
Ozone Pressure	psig		5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
MKS Mass Flow										
Ar Carrier, Chan-1	lpm		3.99	3.99	1.16*	3.99	3.99	3.99	3.99	3.99
He Tracer, Chan-2	lpm		2.01	2.01	2.00	2.01	2.01	2.00	2.00	2.00
Total Sample , Chan-3	lpm		5.01	5.01	5.00	5.01	5.00	5.00	5.00	5.00
Sample Pump*										
Vac Gauge	"Hg		1 CRH	1.5	1.5	2.0	2.0	2.0	2.0	2.0
Pres Gauge	psig	<15	~~5~~ 7.5	8.1	4.5	5.2	5.0	5.0	4.9	5.0
Gas Cylinders Tank Pres#										
O_2	psig	>200	1250	1240	1200	1180	1170	1130	1110	1100
Ar GC	psig	>201	750	750	750	750	750	750	750	750
Ar Sweep	psig	>202	CRH ~~2400~~ 2300	~~1900~~ 2300	2000	1970	1900	1800	1710	1680
He GC	psig	>203	2400	2400	2400	2400	2400	2400	2400	2400
He Tracer	psig	>204	420	390	310	300	270	210	200	2000
time	hh:mm		0101	02:05	3:10	04:03	05:02	0602	0706	0759

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

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

* Ar bottle switch flow back to 3.99 @ 0306 CRH

Reviewed and Approved: RW Zola Date: 8/1/02

sheet 5 of 11

Data Sheet #3: Off-Gas Monitoring Data

RSM-F1		By:	TDP	TDP	TDP	TDP	TDP	TDP	TDP	[illegible]
		Date:	~~7/30~~	7/30/02	7/30/02	7/30/02	7/30/02	7/30/02	7/30/02	7/30/02
		Time:	857	1003	1058	1208	1306	1410	1515	1556
DESCRIPTION	**units**	**Range**								
Gas Chromatograph										
He	ppm		0	0	0				8840	8219
H_2	ppm	<1e4								—
O_2	ppm		197861	193230	204873	200678	194637	190831	193177	186463
N_2	ppm			788950	830131	821111	805355	793819	805632	778494
NO	ppm									—
CO	ppm	<3E4	0							—
CO_2	ppm		0	~~788950~~ TDP	~~830131~~ TDP 2357	3370	3095	3388	2040	2798
N_2O	ppm									—
NOx	ppm									
H_2 + CO	ppm	<2E4								
NOx Analyzer Computer										
Time	hh:mm		856	1007	1059	1209	1307	1410		1600
NOx	ppm		2.1725	0.31278	1.8166	1.2585	2.3801	1.3449	0.05712	1.5379
NO	ppm		0.50349	1.0136	1.0797	0.76680	2.1251	1.2903	-0.15403	1.1243
NOx Voltage	Volts									
NOx Instrument										
Sample Pressure	psig		4.2	4.2	4.2	4.2	4.2	4.2	~~4.1554~~	4.15
Sample bypass flow	sccm		17	17	17	17	17	17	~~19 81~~	17
Ozone Pressure	psig		5	5	5	5	5	5	~~5 84~~	5
MKS Mass Flow										
Ar Carrier, Chan-1	lpm		2.01	2.01	2.01	2.01	2.01	2.01		2.00
He Tracer, Chan-2	lpm		3.99	3.99	3.98	3.99	3.95	3.98		3.98
Total Sample , Chan-3	lpm		5.0	5.01	5.01	5.01	5.00	5.01		5.00
Sample Pump*										
Vac Gauge	"Hg		1	1	1	1	1	1	1	1
Pres Gauge	psig	<15	7	7	7	9	8.5	9	7.5	7.5
Gas Cylinders Tank Pres#										
O_2	psig	>200	1100	1100	1050	1000	1000	1000	900	900
Ar GC	psig	>201	710	710	710	710	710	710	710	750
Ar Sweep	psig	>202	1600/0	1500/0	1430/0	1400/0	1300/0	1250/0	1150/2200	1100/2200
He GC	psig	>203	2350	2350	2350	2350	2350	2350	2350	2350
He Tracer	psig	>204	0/1950	0/1900	0/1900	1840/0	1800/0	0/1750	2100/1730	400/1700
time	hh:mm		900	1005	1100	1208	1308	1412	1517	1601

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

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

sheet 6 of 11

Reviewed and Approved: [illegible] Date: 8/1/02

Data Sheet #3: Off-Gas Monitoring Data

RSM-F1		By:	SH	SH	SH	SH	SH	SH	SH	CASH
		Date:	7/30/02	7/30/02	7/30/02	7/30/02	7/30/02	7/30/02	7/30/02	7/31/02
		Time:	1658	1807	1858	2004	2100	2159	2302	1259
DESCRIPTION	**units**	Range								
Gas Chromatograph										
He	ppm		8802		7438	8595	8155	7902	8267	7703
H_2	ppm	<1e4	21		0	0	0	0	0	0
O_2	ppm		181834		148594	201180	198814	205474	225062	207868
N_2	ppm		760232		765469	781205	777942	798642	850600	812916
NO	ppm		0		0	0	0	0	0	0
CO	ppm	<3E4	0		18	40	0	0	0	0
CO_2	ppm		3716		2325	1606	2059	2625	421	2536
N_2O	ppm		0		0	0	0	0	0	0
NOx	ppm		—		—	—	—	—	—	—
H_2 + CO	ppm	<2E4	—		—		—	—	—	—
NOx Analyzer Computer										
Time	hh:mm		1703		1902	2009	2103	2202	2307	0104
NOx	ppm		1.555		2.0036	1.8087	0.4763	1.4491	-1.2957	1.2304
NO	ppm		1.2086		1.3702	1.5396	0.1413	1.1868	-1.3096	0.58632
NOx Voltage	Volts									
NOx Instrument										
Sample Pressure	psig		4.1		4.3	4.3	4.3	4.3	4.3	4.3
Sample bypass flow	sccm		17		17	17	17	17	17	16.5
Ozone Pressure	psig		5		5	5	5	5	5	5
MKS Mass Flow										
Ar Carrier, Chan-1	lpm		2.01		2.00	2.00	2.01	2.01	2.01	2.00
He Tracer, Chan-2	lpm		3.98		3.98	3.98	3.99	3.99	3.99	3.99
Total Sample , Chan-3	lpm		5.00		5.00	5.00	5.01	5.01	5.02	5.01
Sample Pump*										
Vac Gauge	"Hg		1		1	1	1	1	1	2.5
Pres Gauge	psig	<15	8		6	6.2	6.4	6.4	6	6
Gas Cylinders Tank Pres#										
O_2	psig	>200	900	880	860	830	800	800	780	720
Ar GC	psig	>201	750	750	750	750	750	740	740	740
Ar Sweep	psig	>202	1050/2200	950/2200	880/2200	800/2200	720/2200	640/2200	550/2200	400
He GC	psig	>203	~~2400~~ 2380	2380	2380	2380	2380	2380	2380	2350
He Tracer	psig	>204	2100/1680	2100/1610	2100/1580	2100/1530	2100/1500	2100/1460	2100/1390	1300
time	hh:mm		1702	1809	1900	2007	2102	2201	2304	0108

CALIBRATING

Calibrating

Calibrating

Calibrating

Calibrating

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

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

sheet 7 of ~~12~~ 11

Reviewed and Approved: [signature] Date: 8/1/02

Data Sheet #3: Off-Gas Monitoring Data

RSM-F1		By:	CRH	CRH	CRH	CRH	CRH	CRH	CRH	TDP
		Date:	7/31/02	7/31/02	7/31/02	7/31/02	7/31/02	7/31/02	7/31/02	7/31/02
		Time:	0209	3:00	4:04	05:01	0600	0702	0804	0917
DESCRIPTION	**units**	Range								
Gas Chromatograph										
He	ppm		0	0	30.5	0	0	0	0	7244
H_2	ppm	<1e4	0	0	0	0	0	0	0	
O_2	ppm		0	0	0	0	0	0	0	0
N_2	ppm		809565	0	0	0	0	0	839804	808179
NO	ppm		0	0	0	0	0	0	0	
CO	ppm	<3E4	0	0	0	0	0	0	0	
CO_2	ppm		2297	1803	2187	1961	2727	1809	2376	1863.9
N_2O	ppm		0	0	0	0	0	0	0	
NOx	ppm		—	—	—	—	—	—	—	
H_2 + CO	ppm	<2E4	—	—	—	—	—	—	—	
NOx Analyzer Computer										
Time	hh:mm		0214	0304	0409	0505	0604	0706	0809	0916
NOx	ppm		2.5688	0.16161	1.5777	1.4112	0.19457	0.78772	-0.008898	0.88088
NO	ppm		1.8143	-0.10527	0.75754	0.57865	-0.2953	0.44113	-0.14183	0.24497
NOx Voltage	Volts		—	—	—	—	—	—		
NOx Instrument										
Sample Pressure	psig		4.3	4.3	4.3	4.3	4.3	4.3	4.3	4.3
Sample bypass flow	sccm		16.5	16.0	16.5	16.5	16.5	16.5	16.5	17
Ozone Pressure	psig		5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
MKS Mass Flow										
Ar Carrier, Chan-1	lpm		3.99	3.99	3.99	3.99	3.99	3.99	3.99	3.99
He Tracer, Chan-2	lpm		2.00	2.00	2.00	2.01	2.01	2.00	2.00	2.00
Total Sample , Chan-3	lpm		5.00	5.01	5.00	5.01	5.01	5.01	5.01	5.00
Sample Pump*										
Vac Gauge	"Hg		2.5	2.5	2.5	2.0	2.5	2.5	2.5	2.5
Pres Gauge	psig	<15	4.3	4.5	5.0	5.5	8.3	9.0	10.0	7
Gas Cylinders Tank Pres#										
O_2	psig	>200	700	700	680	650	630	600	580	400
Ar GC	psig	>201	730	720	720	710	710	710	710	700
Ar Sweep	psig	>202	300	220	2130	2080	2000	1920	1820	0/1800
He GC	psig	>203	2350	2350	2350	2350	2350	2350	2350	2326
He Tracer	psig	>204	1270	1230	1190	1150	1120	1080	1030	1600
time	hh:mm		0216	0307	0413	0507	0607	0709	0811	0920

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

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

sheet 8 of 11

Reviewed and Approved: R W Gale Date: 8/1/02

Changed gas bottle

Data Sheet #3: Off-Gas Monitoring Data

RSM-F1		By:	TDP	TDP	TDP	BP	BP	BP	DW	BP
		Date:	7-31-02	7/31/02	7/31/02	7/31/02	7/31/02	7/31/02	7/31/02	7/31/02
		Time:	1255	1453	1559	1659	1804	1946	2009	2059
DESCRIPTION	**units**	Range								
Gas Chromatograph										
He	ppm		82360	7332	0			5059	5852	4938
H_2	ppm	<1e4						0	0	0
O_2	ppm		199556	177121	172367			145965	170191	148548
N_2	ppm		816584	726349	480937			738782	634480	553093
NO	ppm							0	0	0
CO	ppm	<3E4						0	0	0
CO_2	ppm		1716	1917	1376			437	2078	2796
N_2O	ppm							0	0	0
NOx	ppm							—	—	—
H_2 + CO	ppm	<2E4						—	—	—
NOx Analyzer Computer										
Time	hh:mm		1254	1454	1559			1951	2011	2104
NOx	ppm		5.0804	9.4568	9.2417			12.549	17.779	16.828
NO	ppm		2.8067	9.5978	9.3229			12.679	15.069	15.981
NOx Voltage	Volts									
NOx Instrument										
Sample Pressure	psig		4.3	4.2	4.2			4.3	4.3	4.3
Sample bypass flow	sccm		17	17	17			17	17	17
Ozone Pressure	psig		5.0	5.0	5.0			5.0	5.5	5.0
MKS Mass Flow										
Ar Carrier, Chan-1	lpm		3.99	3.99	3.98			2.00	2.00	2.00
He Tracer, Chan-2	lpm		2.01	2.01	1.34			3.98	3.98	3.98
Total Sample , Chan-3	lpm		5.01	5.01	5.01			5.00	5.00	5.00
Sample Pump*										
Vac Gauge	"Hg		1	2	2			1	2	2
Pres Gauge	psig	<15	8	8	8			5.5	5.5	6.0
Gas Cylinders Tank Pres#										
O_2	psig	>200	400	400	350	300	300	250	2050	2040
Ar GC	psig	>201	710	710	710	710	710	710	710	710
Ar Sweep	psig	>202	0/1500	0/1350	0/1350	0/1150	1180/1100	2100/950	2100/950	2100/820
He GC	psig	>203	2350	2350	2350	2350	2350	2350	2350	2350
He Tracer	psig	>204	2000/850	2000/790	2000/2050	1200/200	1120/2050	1080/2050	1080/2050	1040/2050
time	hh:mm		1256		1600	1703	1806	1950	2016	2102

Calibrating

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

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

sheet 9 of 11

Reviewed and Approved: R W Gale Date: 8/1/02

Data Sheet #3: Off-Gas Monitoring Data

RSM-F1		By:	SP	SP	SP						
		Date:	7/31/02	7/31/02	7/31/02			8/01/02	8/01/02	8/01/02	8/01/02
		Time:	2203	2300	2356			0056	0205	0307	0402
DESCRIPTION	**units**	Range									
Gas Chromatograph											
He	ppm		4518	4634	4516			4340	4553	4747	5141
H_2	ppm	<1e4	0	0	0			0	0	0	0
O_2	ppm		130763	134895	120156			134571	143106	243355	33197
N_2	ppm		496009	512830	484376			510867	552055	391299	443882
NO	ppm		0	0	0			0	0	0	0
CO	ppm	<3E4	0	0	0			0	0	0	0
CO_2	ppm		2672	2353	1469			1924	1225	916.68	2352.
N_2O	ppm		0	0	0			0	0	0	0
NOx	ppm		—	—	—			—	—		—
H_2 + CO	ppm	<2E4	—	—	—			—	—		—
NOx Analyzer Computer											
Time	hh:mm		2208	2308	2359			0058	0210	0313	0407
NOx	ppm		8.6384	3.3049	1.5745			1.0321	1.6021	0.318	1.859
NO	ppm		7.5195	~~3.0374~~	1.5239			0.74001	0.6281	0.23	1.939
NOx Voltage	Volts			3.0377							
NOx Instrument											
Sample Pressure	psig		4.3	4.3	4.3			4.3	4.3	4.3	4.2
Sample bypass flow	sccm		17	17	17			16.5	17	17	17
Ozone Pressure	psig		5.0	5.0	5.0			5.0	5.0	5.0	5.0
MKS Mass Flow											
Ar Carrier, Chan-1	lpm		2.00	2.00	2.00			2.01	2.01	2.00	2.01
He Tracer, Chan-2	lpm		3.98	3.99	3.99			3.99	3.99	3.99	3.99
Total Sample , Chan-3	lpm		5.00	5.00	5.00			5.00	5.01	5.01	5.01
Sample Pump*											
Vac Gauge	"Hg		2	2	2			2	2	2	2
Pres Gauge	psig	<15	6	6	6			6	6	6	6
Gas Cylinders Tank Pres#											
O_2	psig	>200	2010	2000	1980			1980	1950	1900	1890
Ar GC	psig	>201	710	710	710			710	710	710	710
Ar Sweep	psig	>202	2100/750	2100/680	2100/560			2100/500	2100/480	2100/~~750~~	2100/150
He GC	psig	>203	2350	2350	2330			2370	2300	2300	2300
He Tracer	psig	>204	1000/2050	960/2050	940/2050			900/2050	850/2050	800/2000	790/2000
time	hh:mm		2206	2303	0001			~~0002~~ 0106	0210	0316	0407

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

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

Switched over to Ar for GC

Reviewed and Approved: [signature] Date: 8/1/02

sheet 10 of 11

Data Sheet #3: Off-Gas Monitoring Data

RSM-F1		By:							
		Date:	8/01/02	8/01/02	8/01/02	8/01/02			
		Time:	0501	0557	0658	0811			
DESCRIPTION	**units**	Range							
Gas Chromatograph									
He	ppm		5500	5511.80	5151.	2569			
H_2	ppm	<1e4	0	0	0				
O_2	ppm		32447	31369	30070	30668			
N_2	ppm		436988	445299	426675	383831			
NO	ppm		0	0	0	0			
CO	ppm	<3E4	0	0	0	0			
CO_2	ppm		815.5	852.7	987.86	876.9			
N_2O	ppm		0		0	0			
NOx	ppm		—	—	—	—			
H_2 + CO	ppm	<2E4	—	—	—	—			
NOx Analyzer Computer									
Time	hh:mm		0505	0601	0701	0816			
NOx	ppm		-1.16	-0.56	+1.255	0.637			
NO	ppm		-1.18	-0.63	+1.23	0.7166			
NOx Voltage	Volts								
NOx Instrument									
Sample Pressure	psig		4.3	4.3	4.3	4.3			
Sample bypass flow	sccm		17	17	17	17			
Ozone Pressure	psig		5	5	5	5			
MKS Mass Flow									
Ar Carrier, Chan-1	lpm		2.00	2.01	2.00	2.01			
He Tracer, Chan-2	lpm		3.99	4.00	3.99	3.99			
Total Sample , Chan-3	lpm		5.01	5.01	5.01	5.01			
Sample Pump*									
Vac Gauge	"Hg		2	2	1.5	1.5			
Pres Gauge	psig	<15	6	6.5	~~6~~ 7.0	7.0			
Gas Cylinders Tank Pres#									
O_2	psig	>200	1820	1800	1780	1710			
Ar GC	psig	>201	710	710	710	710			
Ar Sweep	psig	>202	2000	1900	1800	1730			
He GC	psig	>203	2300	2300	2290	2290			
He Tracer	psig	>204	730/2000	700/2000	660/2000	600/2000			
time	hh:mm		0505	0601	~~0~~ 0702	0817			

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

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

sheet 11 of 11

Reviewed and Approved: [signature] Date: 8/1/02

Appendix D

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

Appendix D: Colorimetric Procedure for Determining Fe(II) to Total Iron Ratio

Fe^{+2} and Fe_{tot} Ratio

1.0 APPLICABILITY

This procedure is applicable for the determination of Fe^{+2} by completing the ferrous ion with 1, 10-phenanthroline. The concentration of the orange-red complex id determined spectrophotometrically at 510 nm.

2.0 DEFINITIONS

2.1 1,10-phenanthroline (phenanthroline) - The organic chelating agent that forms an orange-red complex with ferrous ion (Fe^{+2}).

2.2 Hydroquinone - An organic reducing agent that will reduce ferric ion (Fe^{+3}) to ferrous ion Fe^{+2} in an aqueous solution at room temperature.

2.3 Standard blank - The Fe_{tot} (Fe^{+2} and Fe^{+3}) concentration in all the reagents and the water used in this method.

2.4 Reagent blank - The Fe^{+2} concentration in all the reagents and the water used in this method.

3.0 RESPONSIBLE STAFF

3.1 Responsible Scientist.

3.2 Cognizant Staff.

4.0 PROCEDURE

4.1 Summary of the method

Samples are dissolved in a non-oxidizing condition using a mixture of sulfuric and hydrofluoric acid. Boric acid is added to complex the excess fluoride ion. The ferrous ion in the solution is-chelated by three molecules of phenanthroline forming an orange-red complex. The colored solution obeys Beer's law; its intensity is independent of pH from 3 to 9. A pH between 2.9 to 3.5 insures the rapid color development in the presence of an excess of phenanthroline. The total iron in the sample is determined by reducing the ferric ion to a ferrous ion with Hydroquinone at room temperature. The orange-red color of 'the complex is stable for up to six -months.

Technical Reviewer Date	Approval Authority Date (Line Manager)		Project Mgr Date
Author Date	Project Quality Engineer Date		Other Date
Procedure No:	REVISION NO.	EFFECTIVE DATE:	Page of

4.2 *Reagent*

4.2.1 Iron Standard Solution - A l00 ppm certified iron solution is used to prepare the calibration curve.

4.2.2 0.25% Phenanthroline Solution – Weigh 0.25 ± 0.05 gram of phenanthroline and dissolve in 100 mL of iron-free water.

4.2.3 4% Boric, Acid Solution - Weigh 40 ± 4 grams of orthoboric acid and transfer the orthoboric acid into a 1 liter plastic bottle. Fill the bottle with iron-free water.

4.2.4 Potassium Hydrogen Phthalate (KHP) - Weigh 100 + 10 grams of KHP and transfer the KHP into a 1 liter plastic bottle. Fill the solution with iron-free water and shake the solution to form a saturate the solution (Note: Since the solution is a saturated, some undissolved KHP is present in the bottom of the bottle).

4.3 *Equipment*

4.3.1 pH Meter - The pH meter used in this procedure is the-Corning Model 240-pH meter. Refer to APSL-08, CALIBRATION of PH METER, technical procedure to calibrate the pH meter

4.3.2 Spectrophotometer - The spectrophotometer used in this procedure is the Milton Roy, "Spectronic", model 601 spectrophotometer. Refer to APSL-09, USING THE "SPECTRONIC," MODEL 601 SPECTROPHOTOMETER, technical procedure for the set-up of the spectrophotometer.

4.4 *Safety*

4.4.1 Eye protection required in the laboratory.

4.4.2 Rubber or plastic cloves must be worn when working with concentrated acids.

4.5 *Calibration*

The spectrophotometer is calibrated with a certified iron solution at seven different calibration points. A linear regression is performed on the seven calibration points and the estimate of the slope (m), the y intercept (b), and the correlation coefficient (r) are calculated. If the calculated correlation coefficient is greater than 0.999, the calibration curve is closely approximated by a linear function. The closer the correlation coefficient approaches unity, the closer all the points used to establish the calibration curve falls on a straight line.

4.5.1 Dilute the 1000-ppm iron standard solution to a 100-ppm (0.1 mg/mL) solution by pipetting 10 mL of the 1000-ppm solution into a l00-mL volumetric flask. Add about 5 mL of concentrated HCL and dilute to volume with iron-free water. Thoroughly mix the solution in the flask.

4.5.2 Prepare a 100-mL disposable plastic beaker for the standard blank and each of the seven points used to calibrate the Fe^{+2} CALIBRATION CURVE (Figure 1). Mark the 8 plastic beakers as Std Blk, 0.005 mg Fe, 0.01 mg Fe, 0.05 mg Fe, 0.10 mg Fe, 0.20 mg Fe, 0.30 mg Fe, and 0.40 mg Fe.

4.5.3 Pipet the required volume of the 0.10 mg/mL Fe standard solution prepared in step 4.5.1 into the 7 beakers from step 4.5.2. Since no Fe is added to the Std Blk, this beaker will be empty.

4.5.4 Prepare a second set of disposable plastic beakers for the Std Blk and each of the 7 Fe calibration points. Each of the beakers with a capacity greater than 50 mL shall contain;

a) a glass covered magnetic stir-bar,

b) 25 mL of boric acid solution,

c) 7 mL of KHP solution,

d) 6 mL of phenanthroline solution.

4.5.5 Transfer the beakers from 4.5.4 to a fume hood and add 2 mL of concentrated ammonium hydroxide to each of the beakers. Swirl each of the beakers to mix the solution.

4.5.6 Slowly dispense 0.5 mL of concentrated sulfuric acid dropwi8e to each of the beakers containing the Std Blk and the 7 Fe standards prepared in step 4.5.2 using a 500 microliter pipet. After the sulfuric acid has been added to each of the beakers, add 1.5 mL of concentrated hydrofluoric acid to each of the beakers.

4.5.7 Pour one beaker of the buffer solution prepared in step 4.5.5 into each of the beakers from step 4.5.6.

4.5.8 After mixing the two solutions in step 4.5.7, place each of the beakers on a magnetic stirrer. Using a calibrated pH meter, adjust the pH of each of the solutions to a pH between 3.3 and 3.5 with either dilute sulfuric acid or dilute ammonium hydroxide.

4.5.9 Transfer the solution to a 100-mL volumetric flask. Add 20 ± 10 mg of Hydroquinone to each of the flasks. Using a small amount of iron-free water, wash the adhering Hydroquinone from the neck of the volumetric flask. Swirl the solution to dissolve the Hydroquinone and let the solution stand for at least 30 minutes.

4.5.10 After letting the solution stand for at least 30 minutes, dilute the solution to volume with iron-free water. Thoroughly mix the solution in the flask.

4.5.11 Set the absorbance of the spectrophotometer to 510 nm and zero the instrument with iron-free water in the sample cell.

4.5.12 Read the absorbance of the standard blank and each the seven iron standards. Record the absorbance reading from each of the solutions onto a Xerox copy of the Fe^{+2} CALIBRATION CURVE sheet (Figure 1).

4.5.13 Once all the absorbance readings have been taken, using a linear regression calculation, calculate the slope (m), the γ intercept (b), and the correlation coefficient(r) for the calibration data. If the calculated correlation coefficient is less than 0.999, notify the Responsible Scientist. If the correlation coefficient is greater than 0.999, enter the slope, the intercept, and all the absorbance readings onto the Fe^{+2} CALIBRATION CURVE using the Excel program called "FERCAL.XSL". The Excel program will calculate the values for the 7 calibration points using a linear curve with the estimated slope and intercept values determined above. The 0.005 mg Fe/100 mL of solution must have a calculated value between 0.003 and 0.007 mg Fe/100 mL. The mid-range values of the calibration curve must be within ± 5% of the true values. If the calculated values are not within these limits, notify the Responsible Scientist.

4.5.14 This completes the calibration of the spectrophotometer for the Fe^{+2} analyses. A copy of the signed and approved Fe^{+2} CALIBRATION CURVE shall be pasted into the notebook assigned to the Milton Roy, "Spectronic" model 601 spectrophotometer.

4.6 Sample Log-In Procedure

Refer to APSL-01.

4.7 Sample Preparation

The sample taken in the laboratory for the final grinding will depend upon the appearance and the size of the sample submitted by the customer. If a large sample (greater than 100 grams total) is submitted by the customer and the sample is not homogeneous, the sample will be ground to a particle size of less than approximately ¼ inch. This ground sample will then be reduced to a manageable sample size using the quartering method. If the sample is a homogeneous glass sample, between 5 and 10 grams of sample will be broken from the sample for final sample preparation.

4.7.1 Large non-homogeneous sample.

4.7.1.1 If the total sample weight is greater than 100 grams, grind the sample to less than ¼ inch particle size.

4.7.1.2 Place the sample in a pile on a clean sheet of paper. Using a flat spatula, quarter the sample into four equal parts.

4.7.1.3 Using alternate quarters of the sample in step 4.7.1.2, separate sample into two equal parts.

4.7.1.4 If one of the separated samples from step 4.7.1.3 is still greater than 40 grams, repeat steps 4.7.1.2 and 4.7.1.3 until the sample obtained by the quartering method weighs less than 40 grams.

4.7.1.5 When the sample weight has been reduced to less than 40 grams, grind the sample so that the particle size is less than 1/8 inch. Quarter the sample using steps 4.7.1.2 and 4.7.1.3.

4.7.1.6 Grind the sample from step 4.7.1.5 so that the particle size is less than 1/16 inch.

4.7.1.7 Quarter the sample two times using step 4.7.1.2 and 4.7.1.3. At this point, the sample should be reduced to less than 5 grams.

4.7.1.8 Grind the sample from step 4.7.1.7 using an agate or a porcelain mortar and pestle. Sieve the sample through a 140-mesh sieve. Repeat the grinding and sieving until all the sample has been ground and sieved to less than 140 mesh.

4.7.2 Homogeneous sample.

4.7.2.1 Cover the glass sample with a paper towel. Hit an edge of the sample to chip off pieces of glass. Collect the pieces of chipped glass from the larger sample.

4.7.2.2 Continue step 4.7.2.1 until approximately 3-5 grams of glass chips have been collected.

4.7.2.3 Grind the glass chips using an agate or a porcelain mortar and pestle. Sieve the sample through a 140-mesh sieve. Repeat the grinding and sieving until all the sample has been ground and sieved to less than 140 mesh.

4.8 *<u>Fe^{+2} Analyses</u>*

4.8.1 Prepare a disposable plastic beaker (beaker volume of at least 50 mL) for the standard blank and the sample blank and a beaker for each of the samples, the 0.005 mg Fe standard, and the 0.20 mg Fe standard. Add to each of the beakers:

a) a glass stir-bar,

b) 25 mL of boric acid solution,

c) 7 mL of KHP solution,

d) 6 mL of 4% phenanthroline solution and in a fume hood, pipet,

e) 2 mL of concentrated ammonium hydroxide.

4.8.2 To a second set 100-mL disposable plastic beakers, weight 0.025 ± 0.010 gram of ground sample from step 4.7.1.8 or 4.7.2.3 into a disposable plastic beaker. Record the sample weight and sample identification on each of the beakers. Also record the laboratory number, the customer identification, and the sample weights on a Xerox copy of the <u>Fe^{+2} AND Fe_{tot} DATA SHEET</u> (Figure 2).

4.8.3 Prepare two 100-mL disposable plastic beakers for the standard blank and the sample blank as well as the two beakers for the 0.005 mg Fe and the 0.20 mg Fe standards. Pipet

50 microliter of the 0.10 mg/mL Fe standard prepared in step 4.5.1 into the beaker marked 0.005 mg Fe Std and 2.0 mL of the 0.10 mg/mL Fe standard into the beaker marked 0.20 mg of Fe.

4.8.4 Pipet 0.5 mL of concentrated sulfuric acid to each of the beakers from steps 4.8.2 and 4.8.3 in a fume hood. Swirl the beakers with the samples so the samples are mixed with the sulfuric acid.

4.8.5 Tip the beaker so the sulfuric acid/sample mixture moves the slurry to one side of the beaker. Set the beaker on the floor of the fume hood and gently pipet 1.5 mL of concentrated hydrofluoric acid into the beaker on the opposite side of the sulfuric acid/sample mixture. Gently tip the beaker so the two acids are mixed. Once the initial vigorous reaction between the hydrofluoric acid and the silicate in the glass has subsided, swirl the beakers so that the hydrofluoric acid is mixed completely with the glass sample in the beaker. (Note: The total time for hydrofluoric acid to dissolve the glass must be less than about 30 seconds to reduce the amount of air oxidation of the Fe^{+2} in the solution. Once the solution from step 4.8.1 containing the phenanthroline has been added to the solution in 4.8.5, air oxidation of Fe^{+2} is minimized. Each sample is carried through the pH adjustment steps 4.8.6, 4.8.7, and 4.8.8 before adding hydrofluoric acid to the next beaker.)

4.8.6 After the dissolution of the glass by the hydrofluoric acid, immediately transfer the contents of one of the beakers prepared in step 4.8.1 into the beaker containing the dissolved glass in step 4.8.5.

4.8.7 Mix the two solutions together from step 4.8.6 and using a calibrated pH meter, adjust the solution pH to between 3.3 and 3.5 using either dilute sulfuric acid or ammonium hydroxide. (Note: There should be no precipitate formed during the pH adjustment. If a precipitate forms, notify the Responsible Scientist.)

4.8.8 Transfer the pH-adjusted solution into a 100-mL volumetric flask. Use a small volume of iron-free water to rinse all the solution from the beaker into the volumetric flask.

4.8.9 Repeat steps 4.8.5, 4.8.6, 4.8.7, and 4.8.8 with each beaker one at a time until all the blanks, the two standards, and all the samples have been carried through the pH adjustment and solution transfer outlined in steps 4.8.7 and 4.8.8.

4.8.10 Set aside the flasks containing the standard blank, the 0.005 mg Fe and the 0.20 mg Fe standards. These solutions will be completed in the Fe_{tot} analyses portion of this procedure.

4.8.11 Fill the remaining flasks to volume with iron-free water. Cap and thoroughly mix the solution in each of the flasks.

4.8.12 Adjust the spectrophotometer to read zero absorbance with iron-free water in the sample cell.

4.8.13 Transfer the sample blank to the sample cell and record the Fe^{+2} absorbance reading onto the Xerox copy of the Fe^{+2} AND Fe_{tot} DATA SHEET in the column marked Fe^{+2} Abs. Place the next sample from step 4.8.11 into the sample cell. Read the absorbance of the sample and record absorbance reading onto the Xerox copy of the Fe^{+2} AND Fe_{tot} DATA SHEET. Repeat reading and recording of the absorbance until all the samples in step 4.8.11 are completed.

4.9 Fe_{tot} Analyses

Soluble iron in an acidic aqueous solution is present either as ferrous or ferric ion. Hydroquinone will reduce all ferric ion (Fe^{+3}) in the solution to a ferrous ion (Fe^{+2}). With the reduction of all the Fe^{+3} to Fe^{+2}, the analyses of Fe^{+2} in the solution with the phenanthroline are the Fe_{tot} analyses.

4.9.1 Pipet 10 mL (or a suitable aliquot) of each the sample blank and the samples from step 4.8.13 into separate 100-mL volumetric flasks containing 4 mL of phenanthroline solution in each of the flasks.

4.9.2 Add 20 ± 10 mg of Hydroquinone to each of the flasks from step 4.9.1 and the three flasks containing the standard blank, the 0.005 mg Fe and the 0.20 mg Fe standards from step 4.8.10.

4.9.3 Wash down any Hydroquinone adhering to the neck of the flasks with a small volume of iron-free water. Swirl the solution in the flasks to dissolve the Hydroquinone. Let the flasks stand for at least 30 minutes.

4.9.4 After 30 minutes, dilute the solution in the flasks to volume with iron-free water. Cap the flasks and thoroughly mix the solution in each flask.

4.9.5 Adjust the spectrophotometer to read zero absorbance with iron-free water in the sample cell.

4.9.6 Transfer each of the solutions from Step 4.9.4 into the sample cell and read the Fe_{tot} absorbance of the solution. Record the Fe_{tot} absorbance on the Xerox copy of the Fe^{+2} AND Fe_{tot} DATA SHEET in the column labeled Fe_{tot} Abs. Also record the volume of solution pipetted in Step 4.9.1 (divided by 100) onto the Fe^{+2} AND Fe_{tot} DATA SHEET.

4.10 Calculation

The Fe^{+2} and Fe_{tot} are calculated by entering the values from the Fe^{+2} AND Fe_{tot} DATA SHEET onto the first page of the Excel program labeled "FERATIO4.XLS" and titled Fe^{+2} AND Fe_{tot} DATA ENTRY SHEET (Figure 3). The Excel program will perform the required calculations using a linear equation, which is:

$$y = mx + b$$

where:

m = slope

x = mg Fe/100 mL of solution

b = y intercept of the calibration curve

y = sample absorbance – blank absorbance

The third page of the Excel program will generate the ANALYSIS OF Fe^{+2} AND Fe_{tot} report that is shown in Figure 4 and will be submitted to the customer.

For verification of the excel program labeled "FERATIO4.XLS", a set of sample data will be "hand" calculated and compared with the data generated by the "FERATIO4.XLS." This will be pasted in the notebook for the Milton Roy, "Spectronic", model 601 spectrophotometer.

4.11 Calibration Acceptance Criteria

4.11.1 If there is more than 0.005 differences in the absorbance of the sample blank and the standard blank, notify the Responsible Scientist. A significant difference between the two blanks typically indicates a reagent contamination of ferric ion. This must be corrected before continuing with the analysis.

4.11.2 Check the calculated values for the 0.20 mg Fe standard. This value in the mid-range of the calibration curve should read within 0.20 ± 0.01 mg of Fe_{tot}/100 mL of solution. If the calculated value is not within the acceptable range, notify the Responsible Scientist.

4.11.3 Check the calculated value for the 0.005 mg Fe standard. This standard is at the lower end of the calibration curve. If the calculated value is not within 0.005 ± 0.002 mg of Fe/100 mL of solution, notify the Responsible Scientist.

4.12 *Archiving of Data*

4.12.1 A copy of all the Fe^{+2} CALIBRATION CURVE analyses performed on the Milton Roy, "Spectronic", model 601 spectrophotometer are posted in the notebook for this instrument. The calibration curve used for the Fe^{+2} analyses is identified by the slope (m), the intercept (y), and correlation coefficient (r) recorded on the Fe^{+2} AND Fe_{tot} DATA SHEET, the Fe^{+2} ANAD Fe_{tot} DATA ENTRY SHEET, and the ANALYSIS OF Fe AND Fe_{tot} forms.

4.12.2 The completed Fe^{+2} AND Fe_{tot} DATA SHEET is stored in the sample data file stored under Laboratory Number.

4.12.3 A copy of the completed and approved ANALYSIS OF Fe^{+2} AND Fe_{tot} form sent to the customer shall be stored in the sample data file stored under the Laboratory Number.

Figure 1

Fe^{+2} CALIBRATION CURVE

Analytical and Process Support Laboratory

1.0 Date of Calibration _____

2.0 Spectrophotometer Used _____

3.0 Cell Used _____

4.0 Iron Standard Used _____

5.0 Absorbance Readings:

		Absorbance		Absorbance	Calculated Fe Values
5.1	Standard Blank	_____			
5.2	0.005 mg Fe Std	_____	Minus Blk	_____	_____
5.3	0.01 mg Fe Std	_____	Minus Blk	_____	_____
5.4	0.05 mg Fe Std	_____	Minus Blk	_____	_____
5.5	0.10 mg Fe Std	_____	Minus Blk	_____	_____
5.6	0.20 mg Fe Std	_____	Minus Blk	_____	_____
5.7	0.30 mg Fe Std	_____	Minus Blk	_____	_____
5.8	0.40 mg Fe Std	_____	Minus Blk	_____	_____

6.0 Linear Regression Analysis Calculation:

6.1 Correlation Coefficient (r) _____

6.2 Slope (m) _____

6.3 Intercept (y) _____

7.0 Calibrated by and Date: _____

8.0 Reviewed by and Date: _____

Figure 2

Fe^{+2} AND Fe_{tot} DATA SHEET

Analytical and Process Support Laboratory

Customer _____

Calibration curve used: Slope (m) = _____

Intercept (y) = _____

Correlation coefficient = _____

Low and high standard used to verify calibration curve.

Standard blank Absorbance = _____

0.005 mg Fe Std Absorbance = _____

0.200 mg Fe Std Absorbance = _____

Sample blank (Fe^{+2}) Absorbance = _____

Sample blank (Fe_{tot}) Absorbance = _____

Row	Laboratory Number	Customer's Sample ID	Sample Wt	Fe^{+2} Dil	Fe^{+2} Abs	Fe_{tot} Dil	Fe_{tot} Abs
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							
16							

Balance Used and Date _____

Analyst Signature and Date _____

Figure 3

Fe^{+2} AND Fe_{tot} DATA ENTRY SHEET

Analytical and Process Support Laboratory

Customer _____

Calibration curve used Slope (m) = _____

Intercept (y) = _____

Correlation Coefficient = _____

Low and high standard used to verify calibration curve.

Standard blank Absorbance = _____

0.005 mg Fe Std Absorbance = _____

0.200 mg Fe Std Absorbance = _____

Sample blank (Fell) Absorbance = _____

Sample blank (Fe_{tot}) Absorbance = _____

Row	Laboratory Number	Customer's Sample ID	Sample Wt	Fe^{+2} Dil	Fe^{+2} Abs	Fe_{tot} Dil	Fe_{tot} Abs
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							
16							

Data entered by and date _____

Figure 4

ANALYSIS OF Fe^{+2} AND Fe_{tot}

Analytical and Process Support Laboratory

To:

The Iron II (Fe^{+2}) and Total Iron (Fe_{tot}) have been analyzed in your samples. Two iron standards are analyzed with each set of samples to verify the slope and intercept of the linear calibration curve. The first standard containing 0.005 mg Fe/100 mL is near the detection limit of the colormetric method, while the second standard containing 0.20 mg Fe/100 mL is used to verify the mid-range of the calibration curve. The results of the two iron standards and your samples are tabulated below:

Calibration Curve Used. Slope (m) = _____

Intercept (y) = _____

Correlation Coefficient = _____

Low and high iron standard analysis to verify calibration curve.

0.005 Fe Std = _____ Lower Limit = 0.0025 Upper Limit = 0.0075 mg

0.200 Fe Std = _____ Lower Limit = 0.190 Upper Limit = 0.210 mg

Sample Analyses

Lab No	Customer's Sample ID	Wt % Fe^{+2}	Wt % Fe_{tot}	Fe^{+2}/Fe_{tot}	Precipitate Yes	Precipitate No	If yes, comment

Comments:
1. Dark non-magnetic precipitate.
2. Dark magnetic precipitate.
3. Light colored (whitish) precipitate.
4. Other _____

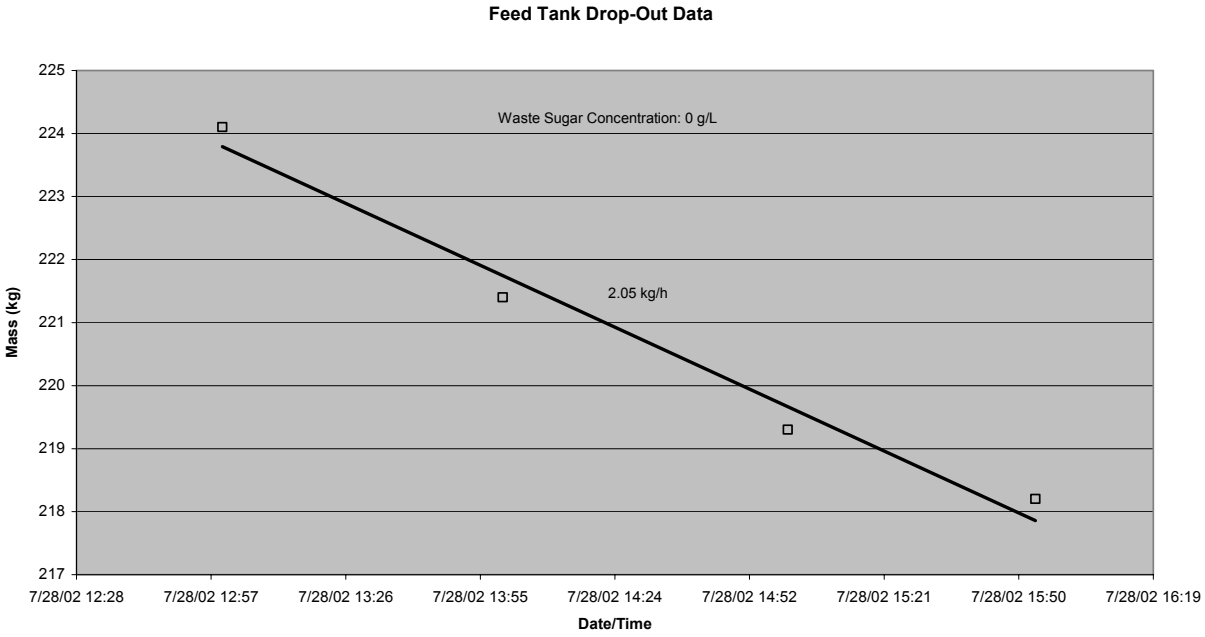
Analyst signature and date _____

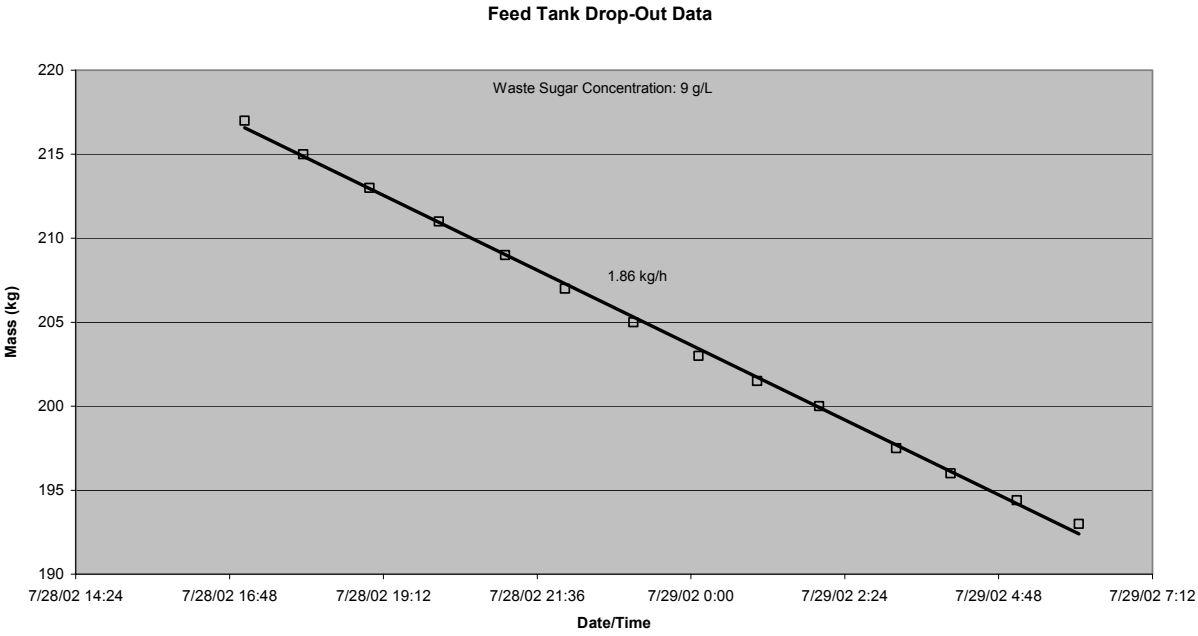
Approved by and date _____

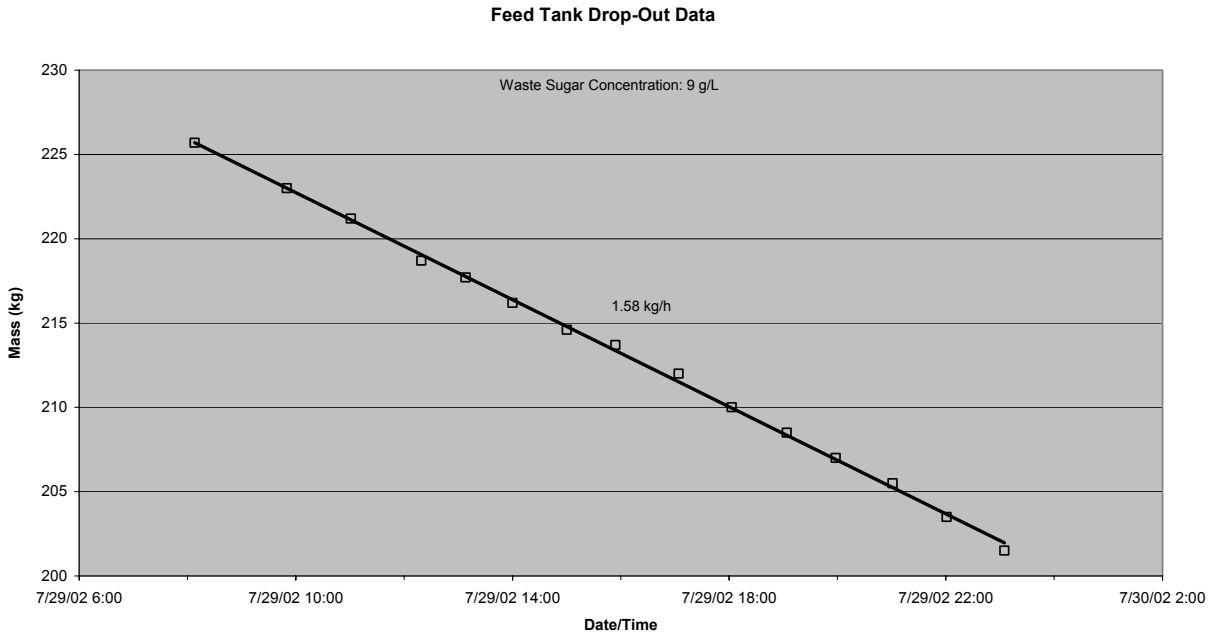
Appendix E

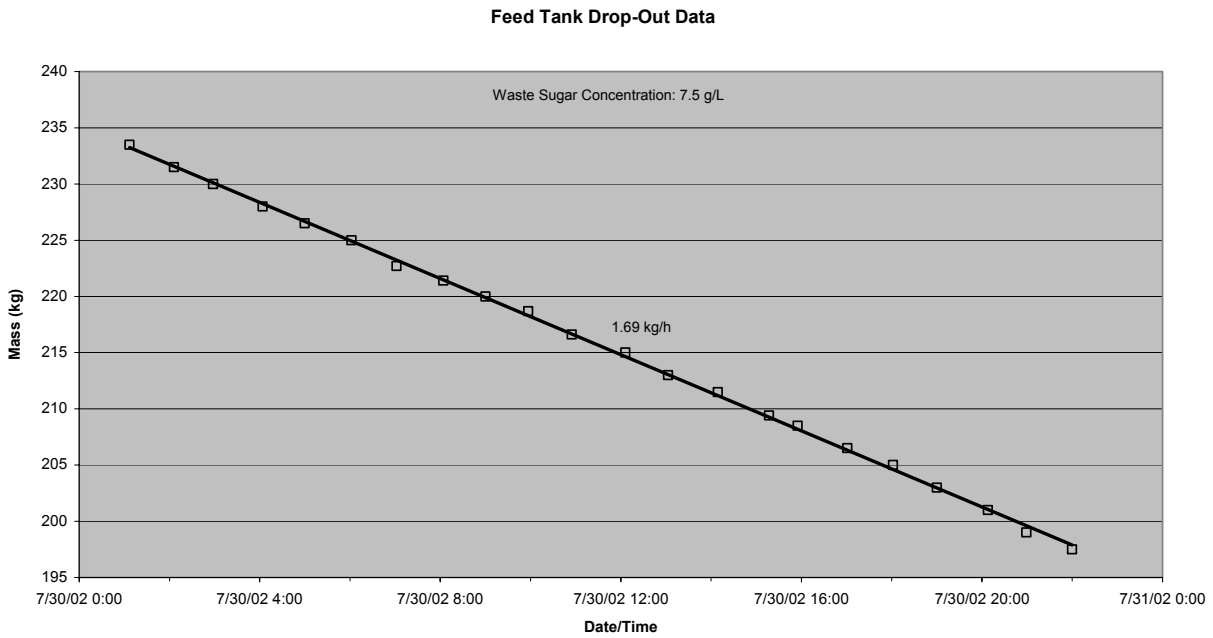
Variable Process Data Collected During the C-106/AY-102 Flowsheet Evaluation

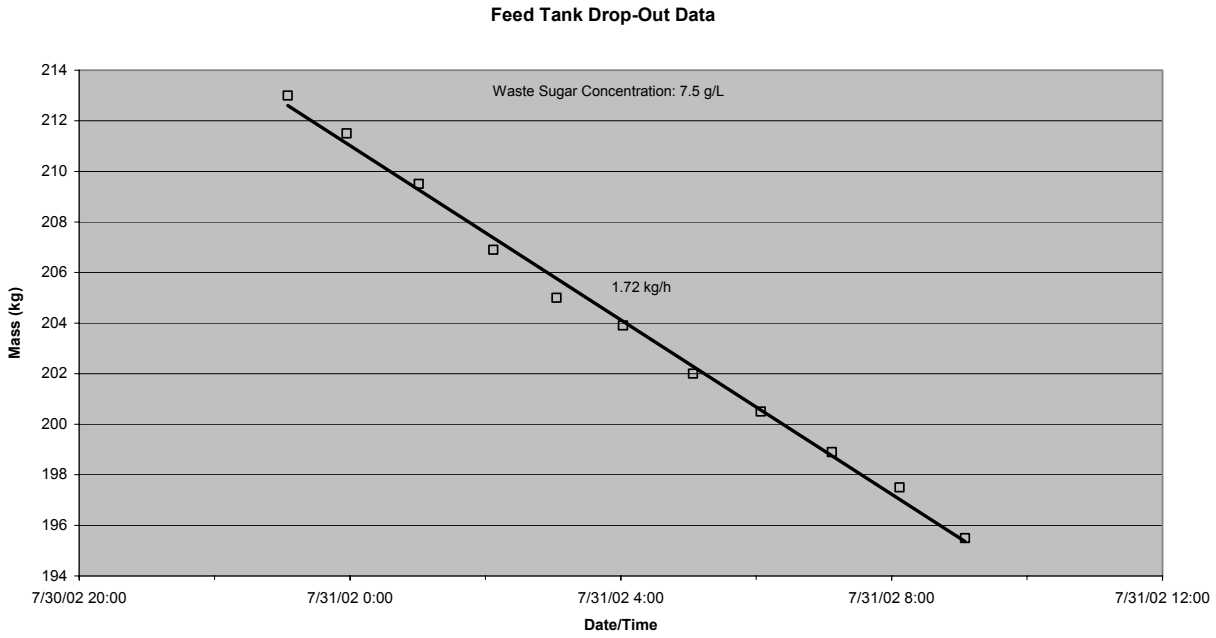
Appendix E.1: Feed Processing Data

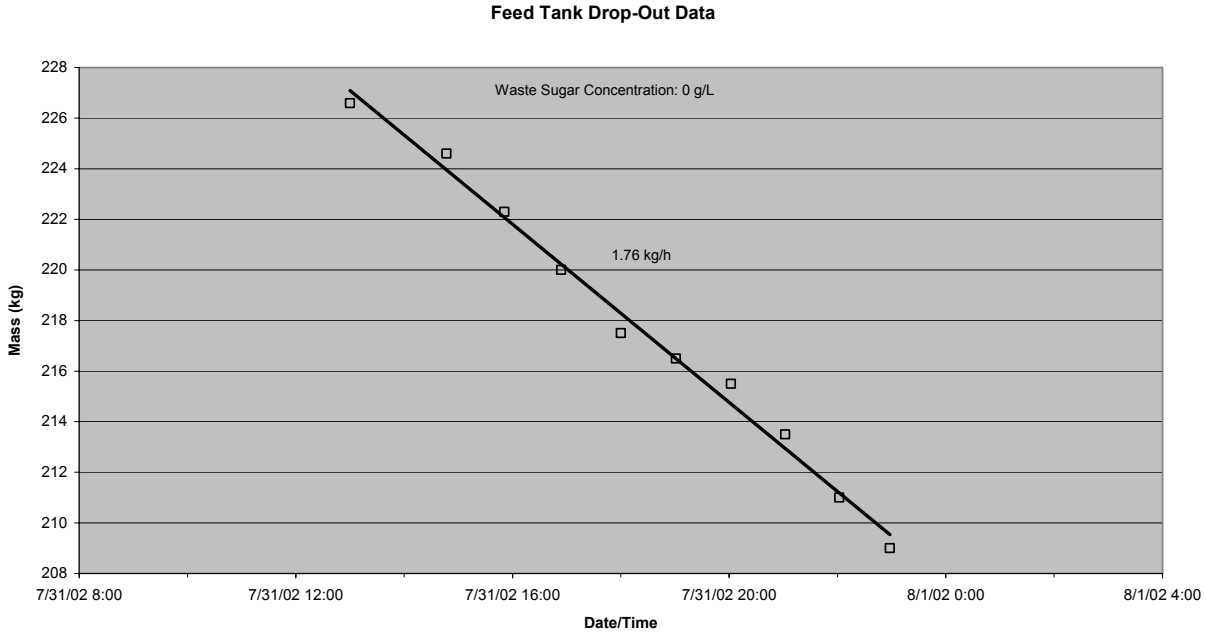


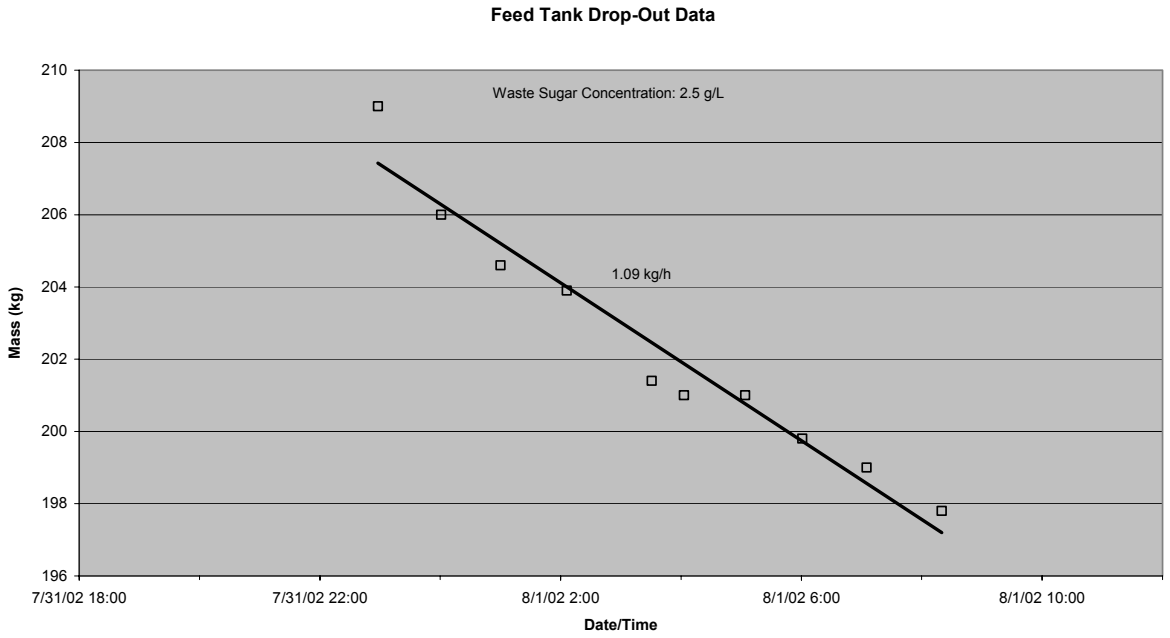


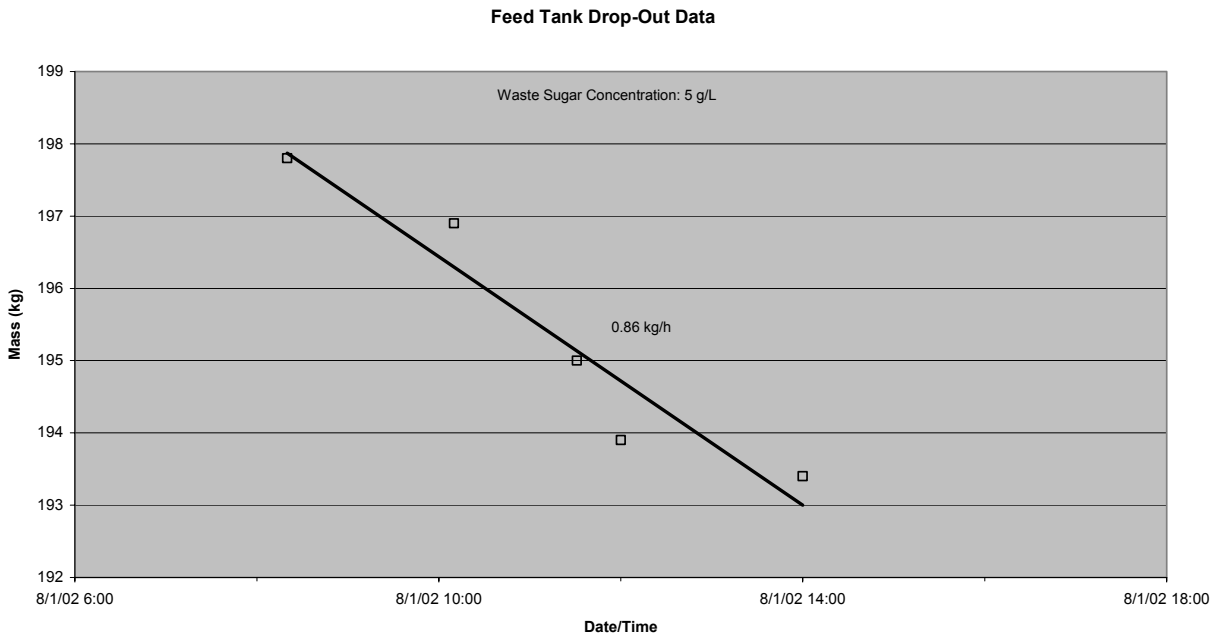

Feed Tank Drop-Out Data
Waste Sugar Concentration: 9 g/L
1.58 kg/h
Mass (kg)
230
225
220
215
210
205
200
7/29/02 6:00
7/29/02 10:00
7/29/02 14:00
7/29/02 18:00
7/29/02 22:00
7/30/02 2:00
Date/Time


Feed Tank Drop-Out Data
Waste Sugar Concentration: 7.5 g/L
1.69 kg/h
Mass (kg)
240
235
230
225
220
215
210
205
200
195
7/30/02 0:00
7/30/02 4:00
7/30/02 8:00
7/30/02 12:00
7/30/02 16:00
7/30/02 20:00
7/31/02 0:00
Date/Time

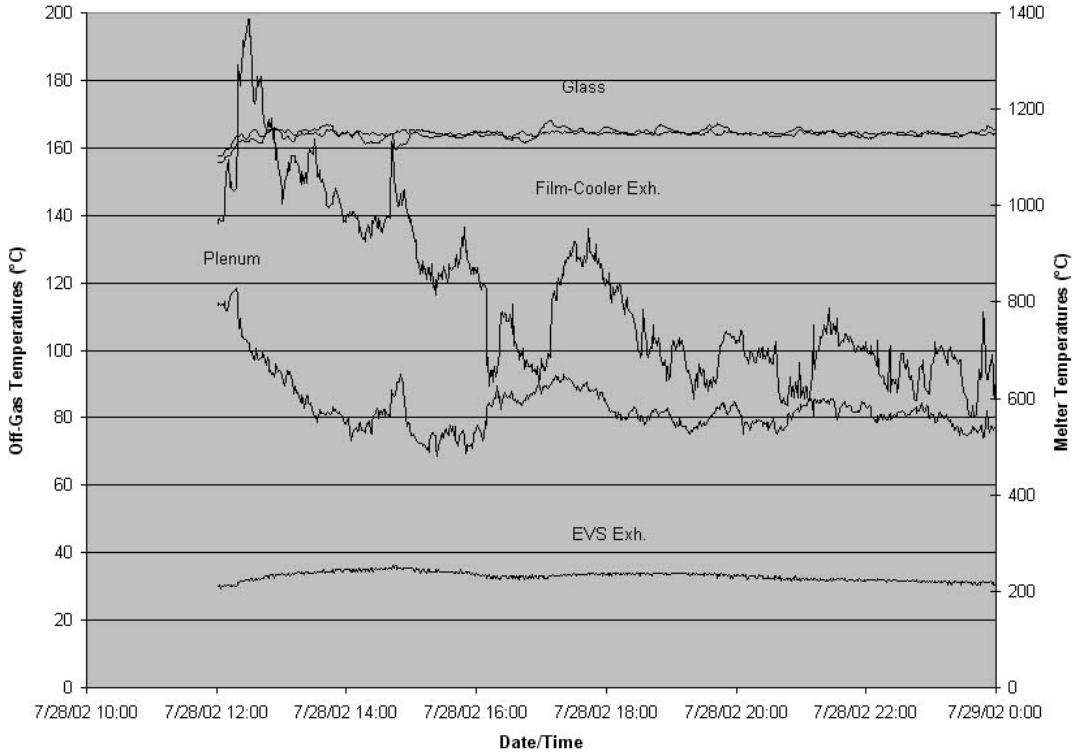

Feed Tank Drop-Out Data
Waste Sugar Concentration: 7.5 g/L
1.72 kg/h
Mass (kg)
214
212
210
208
206
204
202
200
198
196
194
7/30/02 20:00
7/31/02 0:00
7/31/02 4:00
7/31/02 8:00
7/31/02 12:00
Date/Time

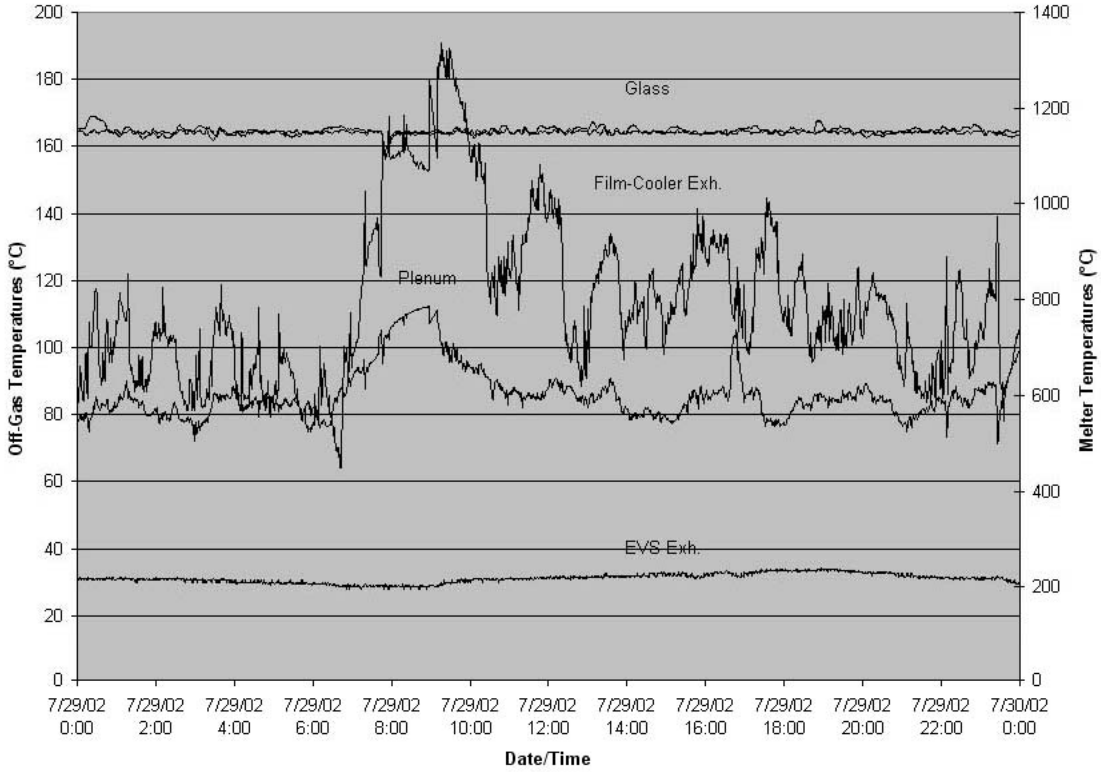

Feed Tank Drop-Out Data
Waste Sugar Concentration: 0 g/L
1.76 kg/h
Mass (kg)
228
226
224
222
220
218
216
214
212
210
208
7/31/02 8:00
7/31/02 12:00
7/31/02 16:00
7/31/02 20:00
8/1/02 0:00
8/1/02 4:00
Date/Time

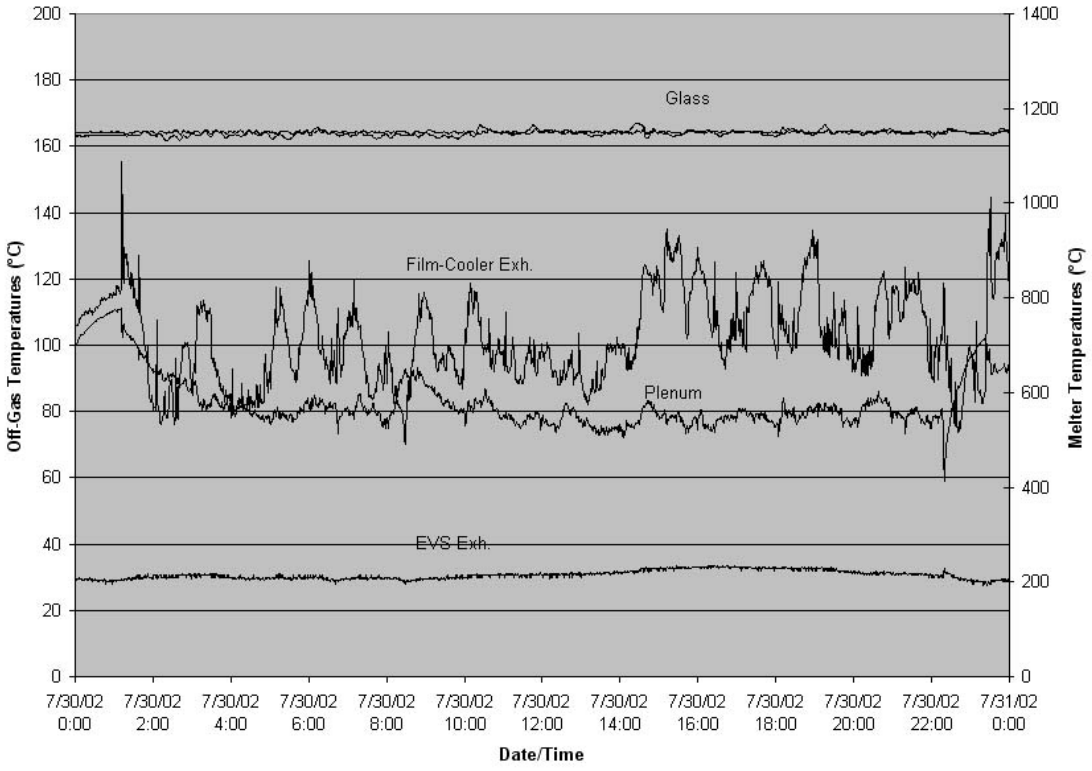

Feed Tank Drop-Out Data
Waste Sugar Concentration: 2.5 g/L
1.09 kg/h
Mass (kg)
210
208
206
204
202
200
198
196
7/31/02 18:00
7/31/02 22:00
8/1/02 2:00
8/1/02 6:00
8/1/02 10:00
Date/Time

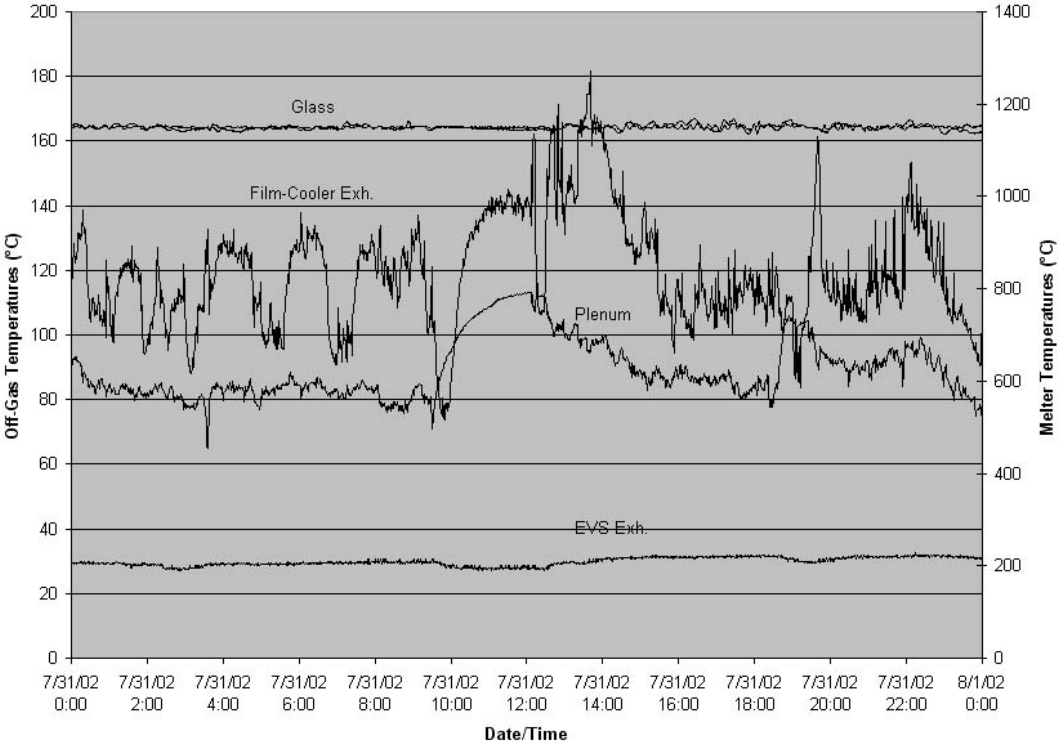

Feed Tank Drop-Out Data
Waste Sugar Concentration: 5 g/L
0.86 kg/h
Mass (kg)
199
198
197
196
195
194
193
192
8/1/02 6:00
8/1/02 10:00
8/1/02 14:00
8/1/02 18:00
Date/Time

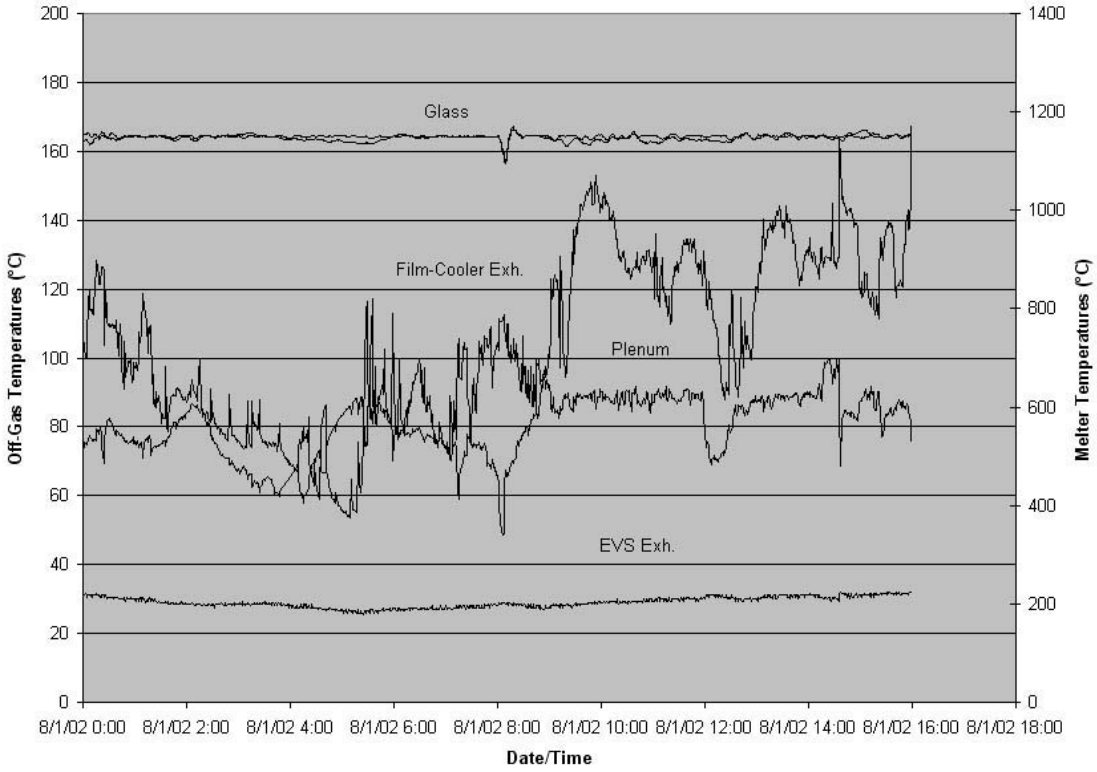
Appendix E.2: Process Temperature Data



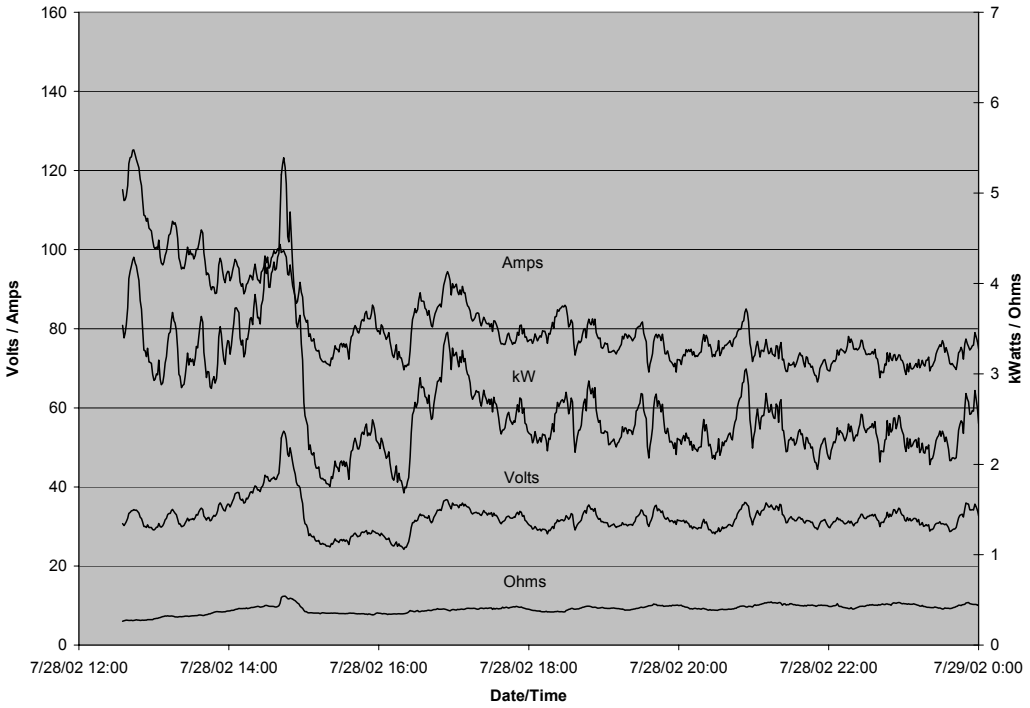


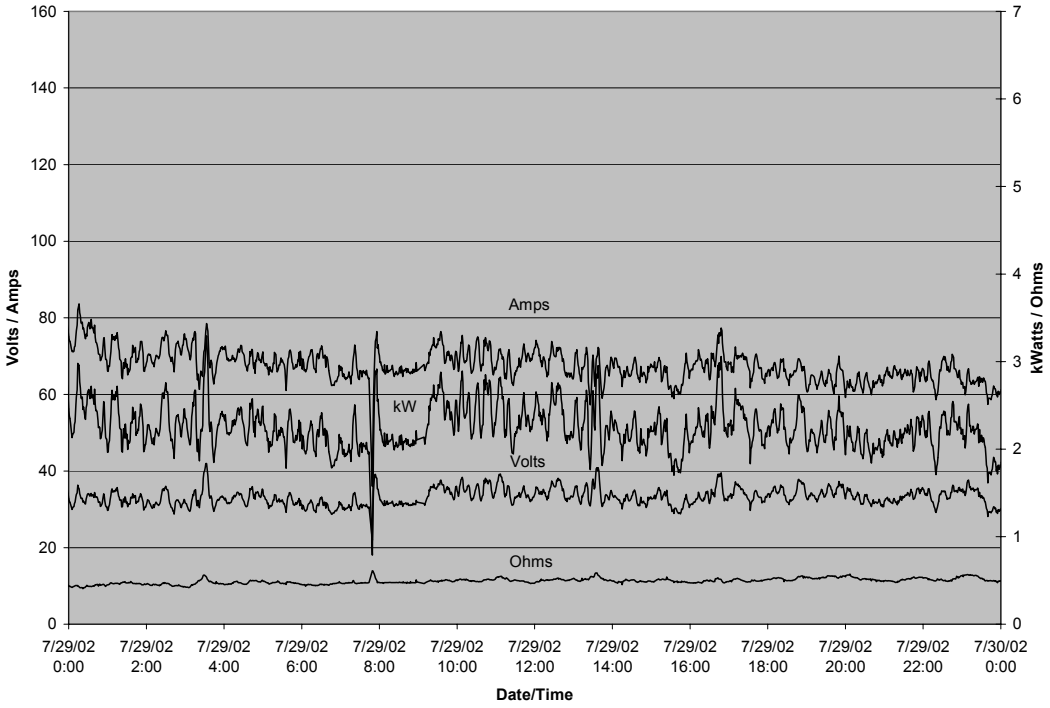

Glass
Film-Cooler Exh.
Plenum
EVS Exh.
Off-Gas Temperatures (°C)
Melter Temperatures (°C)
Date/Time
200
180
160
140
120
100
80
60
40
20
0
1400
1200
1000
800
600
400
200
0
7/30/02 0:00
7/30/02 2:00
7/30/02 4:00
7/30/02 6:00
7/30/02 8:00
7/30/02 10:00
7/30/02 12:00
7/30/02 14:00
7/30/02 16:00
7/30/02 18:00
7/30/02 20:00
7/30/02 22:00
7/31/02 0:00

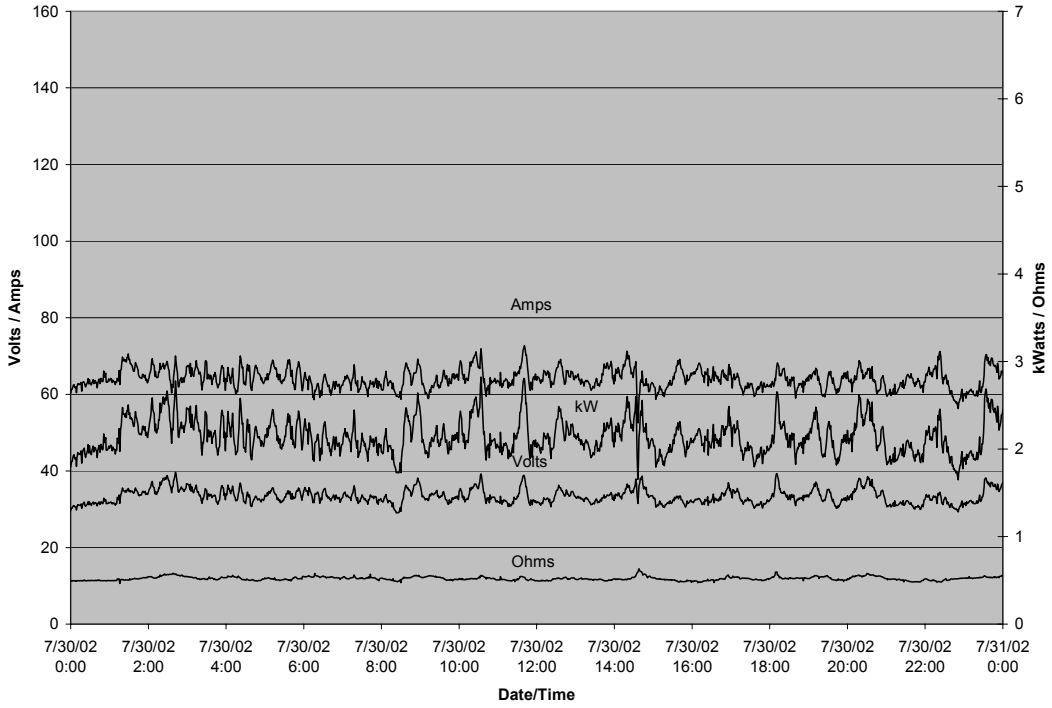

Glass
Film-Cooler Exh.
Plenum
EVS Exh.
Off-Gas Temperatures (°C)
Melter Temperatures (°C)
Date/Time
200
180
160
140
120
100
80
60
40
20
0
1400
1200
1000
800
600
400
200
0
7/31/02 0:00
7/31/02 2:00
7/31/02 4:00
7/31/02 6:00
7/31/02 8:00
7/31/02 10:00
7/31/02 12:00
7/31/02 14:00
7/31/02 16:00
7/31/02 18:00
7/31/02 20:00
7/31/02 22:00
8/1/02 0:00

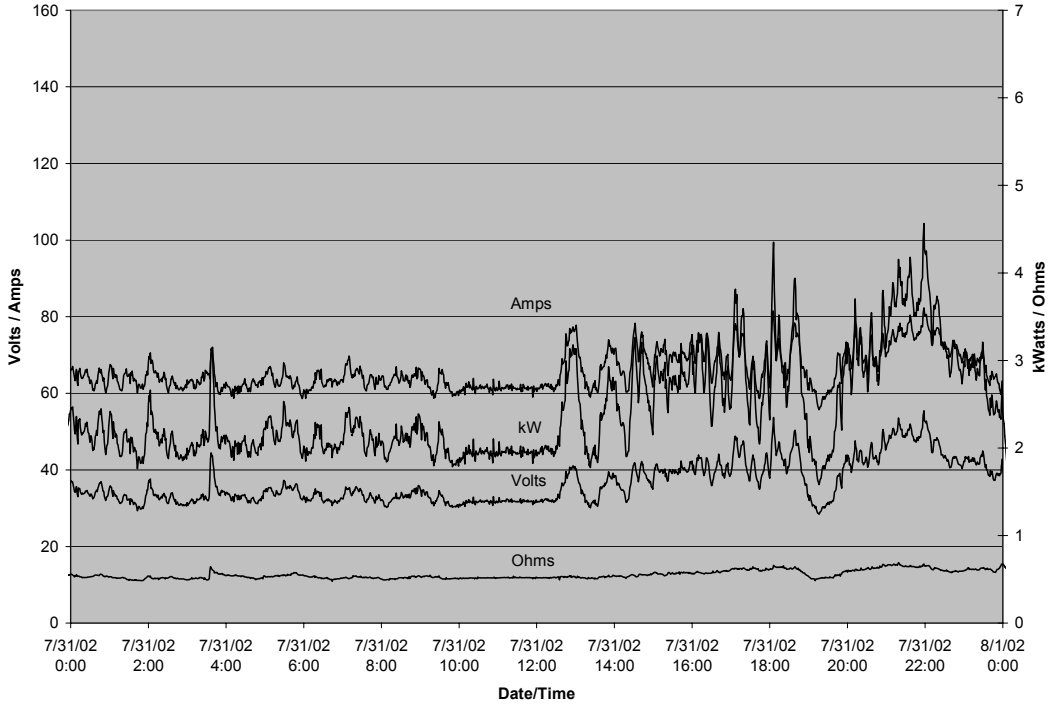

Glass
Film-Cooler Exh.
Plenum
EVS Exh.
Off-Gas Temperatures (°C)
Melter Temperatures (°C)
Date/Time
200
180
160
140
120
100
80
60
40
20
0
1400
1200
1000
800
600
400
200
0
8/1/02 0:00
8/1/02 2:00
8/1/02 4:00
8/1/02 6:00
8/1/02 8:00
8/1/02 10:00
8/1/02 12:00
8/1/02 14:00
8/1/02 16:00
8/1/02 18:00

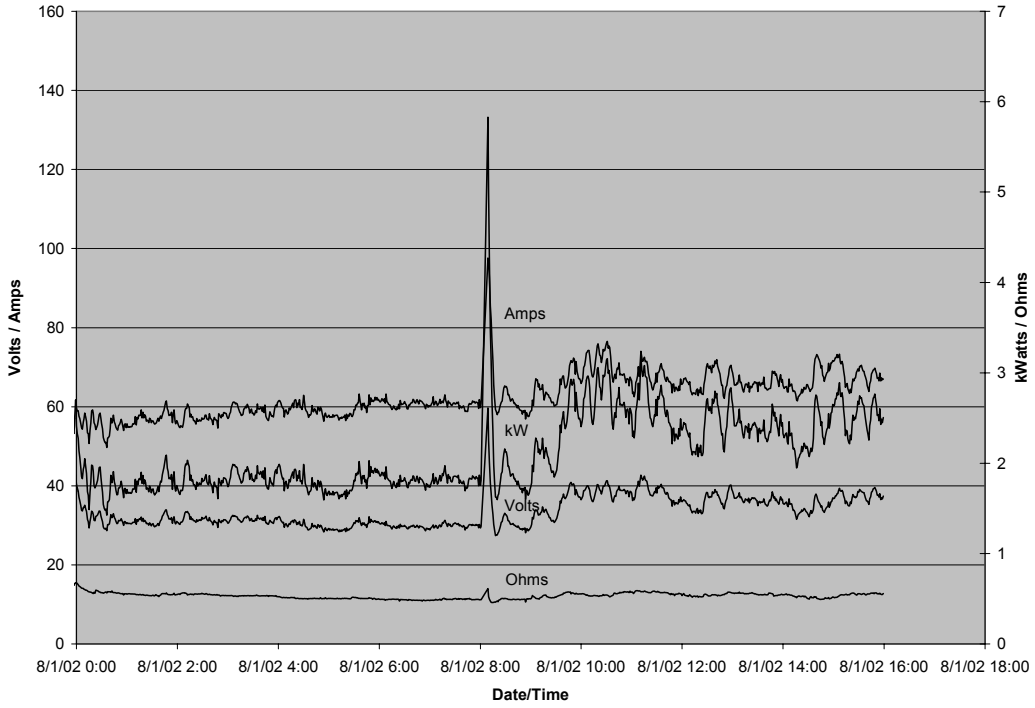
Appendix E.3: Melter Electrical Data






Volts / Amps
kWatts / Ohms
Amps
kW
Volts
Ohms
Date/Time
7/30/02 0:00
7/30/02 2:00
7/30/02 4:00
7/30/02 6:00
7/30/02 8:00
7/30/02 10:00
7/30/02 12:00
7/30/02 14:00
7/30/02 16:00
7/30/02 18:00
7/30/02 20:00
7/30/02 22:00
7/31/02 0:00


Volts / Amps
kWatts / Ohms
Amps
kW
Volts
Ohms
Date/Time
7/31/02 0:00
7/31/02 2:00
7/31/02 4:00
7/31/02 6:00
7/31/02 8:00
7/31/02 10:00
7/31/02 12:00
7/31/02 14:00
7/31/02 16:00
7/31/02 18:00
7/31/02 20:00
7/31/02 22:00
8/1/02 0:00


160
140
120
100
80
60
40
20
0
Volts / Amps
7
6
5
4
3
2
1
0
kWatts / Ohms
Amps
kW
Volts
Ohms
8/1/02 0:00
8/1/02 2:00
8/1/02 4:00
8/1/02 6:00
8/1/02 8:00
8/1/02 10:00
8/1/02 12:00
8/1/02 14:00
8/1/02 16:00
8/1/02 18:00
Date/Time