

EWG-RPT-028, Rev. 0.0 PNNL-30141, Rev. 0.0

# Vitrification of High-Cr Glass in Research-Scale Melter

August 2020

JS Hardy T Jin MA Hall GJ Sevigny **BP McCarthy** JJ Venarsky ML Kimura DR Dixon JS Tixier JJ Neeway CD Lukins **PP** Schonewill WC Buchmiller **DE Rinehart RP** Pires CM Fischer **BD** Williams JB Lang MJ Schweiger



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Pacific Northwest National Laboratory Richland, Washington 99354

# **Executive Summary**

This test report describes the experimental results from a small-scale test using the research-scale melter (RSM) at Pacific Northwest National Laboratory to demonstrate processing of a high-Cr simulated feed stream, designated HLW-HCr-16.

The RSM is a small, joule-heated melter capable of processing melter feed continuously. The melter is equipped with Inconel<sup>®</sup> 693 electrodes, Monofrax<sup>®</sup> K-3 refractory, and an Inconel 690 pour spout. An electric kiln surrounds the melter body and minimizes heat loss from the melter body during operation. The RSM is equipped with an offgas treatment system that employs quenching, wet scrubbing, and high-efficiency mist elimination. The glass-discharge section is heated to facilitate pouring of the glass. The melter is fitted with a melt cavity that is ~25 cm (10 in.) in diameter with a nominal glass depth of 8.9 cm (3.5 in.). The melter was operated with a target glass temperature of 1150°C and target plenum temperature between 550°C and 700°C for this test. The air bubbling rate was 4.2 L/min. Overall, during the continuous operation of the melter for ~ 103 hours, ~ 141 kg of glass was produced.

At the conclusion of the test, the melter and exhaust lines were visually inspected for particulate deposition and corrosion. Entrained material had adhered to the underside of the melter lid and to the exhaust piping. Enrichments in elements such as Cl, F, B, K, Li, P, Na, and S were measured in these deposits through inductively coupled plasma–optical emission spectroscopy and X-ray fluorescence analysis. When the melter electrodes and air bubbler tube were removed from the glass in the RSM, the electrodes appeared discolored, but no significant loss of metal was observed.

The processing of a high-Cr simulant, HLW-HCr-16, in the RSM produced glass at an average rate of 1.36 kg/h, equaling a melter-surface-area normalized glass generation rate of  $654 \text{ kg/day/m}^2$ . The resulting glass met the toxicity characteristic leaching procedure requirement. Test results of crystallinity, electrical conductivity, and viscosity showed good processing properties of this high-Cr high-level waste glass. RSM offgas was also sampled and analyzed at periodic intervals during steady-state operating conditions. The total decontamination factor averaged by four sampling periods was 134. The concentrations of CO and NO<sub>x</sub> in emissions were 237 to 422 and 69 to 94 parts per million by volume, respectively.

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# Acronyms and Abbreviations

ASTM	American Society for Testing and Materials			
BNI	Bechtel National, Inc.			
CCC	canister centerline cooling			
DAC	data acquisition and control			
DF	decontamination factor			
DOE	U.S. Department of Energy			
dscf	dry standard cubic feet			
dscfm	dry standard cubic feet per minute			
DWPF	Defense Waste Processing Facility			
EC	electrical conductivity			
EDS	energy dispersive X-ray spectroscopy			
EPA	U.S. Environmental Protection Agency			
EVS	ejector venturi scrubber			
EWG	enhanced waste glass			
HASQARD	Hanford Analytical Services Quality Requirements Document			
HDI	PNNL's standards-based management system — "How Do I?"			
HEME	high-efficiency mist eliminator			
HLW	high-level waste			
HX	heat exchanger			
IC	ion chromatography			
ICP-OES	inductively coupled plasma-optical emission spectroscopy			
JHM	joule-heated melter			
LSM	laboratory-scale melter			
NA	not applicable			
ND	not detected			
NDIR	nondispersive infrared			
NIST	National Institute of Standards and Technology			
NM	not measured			
NQAP	Nuclear Quality Assurance Program			
OD	outer diameter			
ORP	Office of River Protection			
PM	particulate matter			
PNNL	Pacific Northwest National Laboratory			
ppm	parts per million			
ppmvd	dry parts per million by volume			
QA	quality assurance			
RSM	research-scale melter			
sccm	standard cubic centimeters per minute			
SEM	scanning electron microscopy			
SRM	Standard Reference Material			
SwRI	Southwest Research Institute			
TAP	Test/Analytical Procedure			

TCLP	toxicity characteristic leaching procedure
VSL	Vitreous State Laboratory
WTP	Waste Treatment and Immobilization Plant
wscfm	wet standard cubic feet per minute
XRD	X-ray diffraction
XRF	X-ray fluorescence

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# 1.0 Introduction

The U.S. Department of Energy (DOE), Office of River Protection (ORP), has contracted with Bechtel National, Inc. (BNI) to design, construct, and commission the Waste Treatment and Immobilization Plant (WTP) at the Hanford Site (DOE 2000). This plant is designed to operate for ~40 years and treat millions of gallons of radioactive waste stored in 177 underground tanks at the Hanford Site. Vitrification technology was chosen to treat a portion of the tank waste at the DOE's Hanford and Savannah River sites. Joule-heated melters (JHMs) are being used at the Defense Waste Processing Facility (DWPF) at Savannah River Site and will be used at the WTP to vitrify tank waste fractions.

A JHM developed by Pacific Northwest National Laboratory (PNNL), referred to as the research-scale melter (RSM), is used for small-scale tests, which are intended to provide programmatic guidance to the WTP mission for efficient vitrification of a variety of Hanford waste streams. The RSM configured for the test includes Inconel<sup>®</sup> 693 plate electrodes and Monofrax<sup>®</sup> K-3 as the glass contact refractory. The melt cavity is 25.4 cm (10 in.) in diameter and 8.9 cm (3.5 in.) deep. The results described here are from a test performed in fiscal years 2013-2014 as part of ORP validation tests for melters used by PNNL and the Vitreous State Laboratory (VSL) at The Catholic University of America (Matlack et al. 2014).

This test report describes the experimental results from processing simulated high-Cr high-level waste (HLW) feed using the RSM. A high-Cr glass composition developed by VSL (Matlack et al. 2014) was prepared in a single batch by NOAH Technologies Inc. and shipped to testing facilities at PNNL and VSL to be tested concurrently. The tests measured process rates, crystal formation, and retention of constituents in the glass.

Requirements for this assessment included, but were not limited to, successful production of a durable, compliant glass with an acceptable waste loading at design production rates without causing excessive corrosion of JHM components or operating instability. The specific objectives of the study were to (1) obtain steady-state operations to determine processing rate and melter operating characteristics; (2) collect and analyze samples from feed, glass, and offgas; (3) verify composition and quality of glass produced through inductively coupled plasma–optical emission spectroscopy (ICP-OES), X-ray diffraction (XRD), and microscopic analyses; (4) measure processing properties, viscosity, and electric conductivity of the produced glasses; and (5) conduct sampling of the melter exhaust to characterize particulate and major gas emissions.

Test objectives and goals were satisfied; however, operational issues related to plugging of melter feed lines limited the collection data over time periods typical of vitrification tests. This report discusses the challenges presented to the operations staff during the run and considerations for future melter tests.

### 1.1 Quality Assurance

Activities supporting the research contained in this report has been ongoing since 2012. From 2012 through 2016, this work was performed in accordance with PNNL's laboratory-level Quality Management Program, which is based upon the requirements as defined in the United States Department of Energy (DOE) Order 414.1D, Quality Assurance, and 10 CFR 830, Nuclear Safety Management, Subpart A, Quality Assurance Requirements. PNNL implements these requirements with a graded approach using the consensus standard ASME NQA-1-2000, Quality Assurance Requirements for Nuclear Facility Applications, graded on the approach presented in NQA-1-2000, Subpart 4.2, Guidance on Graded Application of Quality Assurance (QA) Standard for Nuclear-Related Research and Development.

Since 2017, work has been performed in accordance with the PNNL Nuclear Quality Assurance Program (NQAP) Quality Assurance Manual (NQAP-2012) and associated QA procedures. The NQAP is based on the requirements of NQA-1-2012, Quality Assurance Requirements for Nuclear Facility Application, graded on the approach presented in NQA-1-2012, Subpart 4.2.1, Guidance on Graded Application of Nuclear Quality Assurance (NQA) Standard for Research and Development. The NQAP works in conjunction with PNNL's laboratory-level Quality Management Program.

All analytical project work was performed following the Hanford Analytical Services Quality Assurance Requirements Document (HASQARD; DOE 2014).

# 2.0 RSM System

This section describes the RSM processing system used to support this study. The Process Development Laboratory East building, located at PNNL in Richland, Washington, housed the RSM system, for which a schematic is shown in Figure 2.1. The liquid lines are shown in blue and the gas lines are shown in black.



Figure 2.1. Research-Scale Melter Configuration

The RSM is a small JHM capable of processing melter feed continuously, which is a key capability that is needed for the melter to be representative of a full-scale melter system at the WTP. Testing in the RSM allows parametric studies to be conducted in a relatively short time.

The RSM processing system provides unit offgas treatment operations of quenching, wet scrubbing, and high-efficiency mist elimination. The offgas port contains a simple film cooler fabricated from a sintered metal filter that allows injected air to pass from the outside of the filter through the sintered metal into the offgas line while melter exhaust gas passes through the middle of the filter, combining with the injected air. The aqueous quench scrubber is an ejector venturi scrubber (EVS), previously shown to be functionally equivalent to the WTP submerged bed scrubber technology (Goles and Schmidt 1992). The exhaust of the RSM EVS is treated by a high-efficiency mist eliminator (HEME) which efficiently removes sub-micron aerosols and particulate matter penetrating the EVS. Table 2.1 provides RSM dimensions and other operational features.

Parameter	RSM
Melter cavity diameter	25.4 cm
Melt surface area	$500 \text{ cm}^2$
Melter cavity height	17 cm
Melter internal volume	8.6 L
Nominal glass melt depth	8.9 cm
Nominal glass melt volume	4.5 L
Maximum operating temperature	1200°C
Nominal operating temperature for borosilicate glass	1150°C
Bubbler dimensions	1/4-in. OD <sup>(a)</sup> tubing
Bubbler material	Inconel 690
Electrode dimensions ( $W \times H \times T$ )	$11 \times 10 \times 0.9$ cm
Electrode material	Inconel 693
Electrode distance from bottom	0 cm <sup>(b)</sup>
Electrode current (average)	90 A
Electrode voltage (average)	60 V
Electrode current density (average/maximum)	0.5/2.0 A/cm <sup>2</sup>
<ul><li>(a) OD = outer diameter.</li><li>(b) Electrodes rest on the bottom of the RSM cavity.</li></ul>	

Table 2.1. RSM Dimensions and Operational Features

The body of the RSM is an Inconel 625 closed-ended cylinder lined with Alfrax<sup>®</sup> refractory that contains a Monofrax K3 refractory melt cavity. An Inconel pour spout tube discharges molten glass into a stainless-steel canister. Two Inconel 693 electrodes enter the melter through ports in the lid and are suspended in the glass to supply joule-heating power to the RSM. An electric kiln surrounds the melter body to heat the melter during startup and to minimize heat loss from the melter body during operation by decreasing the temperature gradient across the melt chamber walls. The discharge section is heated to facilitate pouring of the glass. The stainless-steel canister, for receiving poured glass, sits inside a smaller kiln maintained between 700°C and 900°C to promote uniform canister filling. Accounting for the areas displaced by the electrodes and air bubblers (used to promote mixing), the melt surface area of the RSM is 500 cm<sup>2</sup>, with a nominal glass depth of 8.9 cm. This results in a corresponding glass inventory of 11 kg, assuming a glass density of 2.5 g/cm<sup>3</sup> at 1150°C. The melter is controlled using a data acquisition and control (DAC) system (hardware and software from Allen-Bradley/Rockwell Automation, Milwaukee, WI), which allows temperature and power control. Temperature control was typically employed during

these tests, with alarms set to alert the operators if temperature or power strayed outside of its pre-defined range.

### 2.1 RSM Feed System

During the test, feed was delivered from the conical bottom of the 55-gallon feed tank to the RSM melter with two peristaltic pumps (Figure 2.2). One pump recirculated the slurry in a 1/2-in. line to a point near the melter to keep solids in the feed line suspended, and the second pump provided slurry in a 1/4-in. line to the water cooled melter feed nozzle. An agitator in the feed tank kept the slurry well mixed. The feed tank was mounted on a scale that was monitored by the computer DAC system. The speed of the second pump was used to control the feed rate to the melter. While the feed system performed well in prior tests, during this test, frequent plugging by undissolved NaOH pellets was observed in the 1/4-in. line.



Figure 2.2. RSM Feed System Diagram

### 2.2 EVS

The EVS sprays scrubber solution through a nozzle for direct contact with the melter exhaust. At the beginning of a melter run, the scrubber solution consists only of water. As the melter operates, the EVS condenses water from the melter exhaust and removes particulates and some acid gases. The resulting two-phase stream travels through a separator chamber and the scrubber solution returns to the scrub tank under the force of gravity. The scrubber solution is recirculated from a tank with a pump located adjacent to the RSM platform and through a heat exchanger (HX) to remove the heat transferred from the melter

exhaust. From the scrubber, the exhaust passes through a HEME to remove condensed-phase aerosols. Quench-scrubber samples were collected periodically during the test for chemical analysis.

### 2.3 Process Conditions

The process conditions maintained during the feeding/pouring period of the test are described in this section. The process conditions targeted during testing are described below and are given in Table 2.2.

Parameter	RSM			
Glass inventory <sup>(a)</sup>	11 kg			
Glass melt temperature	1150°C			
Plenum temperature range	550–700°C			
Plenum vacuum	~0.5 to ~ 3.0 in. water			
Post-film-cooler temperature range	150–350°C			
Melt bubbling rate	4.2 L/min			
Initial scrub solution volume 50 L				
Melt condensate pH	> 3			
(a) Assuming a glass density of 2.5 g/cm <sup>3</sup> at $1150^{\circ}$ C.				

Table 2.2. Target RSM Operating Conditions

The major process conditions that were controlled were glass pool temperature, melter vacuum, melt pool bubbling rate, processing rate, plenum temperature, offgas temperature, and quench-scrubber condensate temperature. Strategies for maintaining baseline conditions are discussed in the following subsections.

#### 2.3.1 Glass Pool Temperature

The 1150°C target temperature was automatically controlled by the RSM DAC system. The electrode current density was constrained to  $\leq 2 \text{ A/cm}^2$  (~190 A for the RSM) to prevent excessive corrosion of the electrodes. If the electrode current density was to become a constraint in maintaining the target glass temperature, the kiln temperature could be adjusted to mitigate heat loss from the melt. The thermocouples monitoring the melt temperature were located within the melter electrodes.

#### 2.3.2 Melter Vacuum

The RSM blower was able to provide up to 28-in. water gauge vacuum (at 200 cubic feet per minute). The RSM vacuum was automatically controlled at a set point, nominally between 0.5- and 2-in. water gauge below ambient conditions. The vacuum pressure was adjusted to instigate and postpone glass pours.

#### 2.3.3 Melt Pool Bubbling Rate

Glass pool agitation using subsurface air injection was employed to enhance melter feed processing rates. To accomplish this, a flow meter delivered air into the RSM at 4.2 L/min divided between two tubes that entered the melt from the top of the melter. The accuracy of the flow meter was such that the bubbling rates in the RSM were within  $\pm 0.05$  L/min of the rates indicated by the flow meter.

#### 2.3.4 Processing Rate

Steady-state feed processing rates for the melter were controlled based on cold-cap conditions, which were visually observed through a view port in the lid of the melter at least once every hour and obliquely

monitored by tracking the plenum temperature. Changes in feed rate were required to maintain target cold-cap coverage of 80% to 90%. During the test, to achieve the target glass processing rate of  $\sim$ 1.0 MT/day/m<sup>2</sup>, a feed rate of  $\sim$ 4.1 L/h was necessary.

#### 2.3.5 Plenum Temperature

The targeted plenum temperature range was 550°C to 700°C during periods when maximum feeding rates were sustained. While plenum temperature was not directly controlled, in-leakage, melter kiln temperature, and bubbling rate all influenced it under steady-state processing conditions (80% to 90% cold-cap coverage).

#### 2.3.6 Offgas Temperature

The post-film-cooler offgas temperature was constrained to  $<350^{\circ}$ C to prevent the offgas lines from becoming plugged by particulate deposition. The temperature was controlled by a valve adjusting the film-cooler air injection rate.

#### 2.3.7 Quench-Scrubber Solution Temperature

The expected EVS solution temperature was ~30°C to 40°C. If there was a need to increase or decrease this temperature, the cooling flow rate of the condensate HX was adjusted appropriately.

After changes in operating parameters, some operating time was needed to allow the glass melt to approach a new equilibrium. After changes to the bubbling rate, a significant amount of time was required to find the appropriate feed rate for reaching steady-state conditions. Stability can be difficult to assess because plenum temperatures have normal fluctuations and cold-cap observations are subjective. For the present test, conditions were required to be stable for >5 h to declare the system at steady state. Many feed rates were tested to determine the maximum sustainable rate.

### 2.4 Data Collection and Process Controls

The collection of process, operational, and control data was performed primarily by the RSM DAC system, which monitors, controls, and electronically logs key system variables at 1-s intervals. Process data not electronically logged by this system and selected parameters of most interest were recorded manually on operator datasheets every hour (shown in Appendix A).

Table 2.3 identifies the process information that was electronically logged by the RSM DAC system and/or manually logged on RSM operation datasheets. The data documented important operational conditions associated with the melter, offgas system, feed, glass, and secondary waste streams.

Parameter	Units	Electronic Log	Manual Log
Melt temperature (T1, T2)	°C	Х	Х
Melt (electrode) set-point temperature	°C		Х
Plenum temperature	°C	Х	Х
Feed pump setting (power output %)	%	Х	
Feed tank weight	kg	Х	
Glass poured	g		Х
Electrode potential	volt	Х	Х
Electrode current	amp	Х	Х
Electrode power	kW & %	Х	Х
Electrode power control mode	auto or manual		Х
Melt resistance	Ω		Х
Kiln power	kW & %		Х
Kiln set-point temperature	°C		Х
Kiln actual (middle) temperature	°C		Х
Kiln top/bottom temperatures	°C/°C		Х
Pour spout heater set-point temperature	°C		Х
Pour spout heater temperature	°C		Х
Pour spout heater power output	kW & %		Х
Feed nozzle temperature	°C		Х
Offgas temperature	°C		Х
Post-EVS offgas temperature	°C		Х
Scrub liquid EVS inlet temperature	°C		Х
HX temperature	°C		Х
Post HEME temperature	°C		Х
Plenum vacuum	in. H <sub>2</sub> O	Х	Х
Bubbling rate	sccm	Х	Х

Table 2.3. RSM Process Data Logged Electronically (1-s intervals) or Manually (~1-h intervals)

## 3.0 Simulated Waste and Melter Feed

This section describes the preparation and analysis of the melter feed. The feed was originally formulated based on a high-Cr glass, HLW-HCr-16 (Matlack et al. 2014). The high-Cr glass composition is summarized in Table 3.1, which also shows the relative proportions of the glass formers used and the resulting target glass composition. The feed composition was prepared in a batch by the supplier (NOAH Technologies Inc., San Antonio, TX) and split into two to be tested concurrently in this run and in VSL melters. The high-Cr melter feed was shipped from the supplier to PNNL in 55-gallon drums. However, due to an errant formula in the spreadsheet that was used to calculate the batch, an insufficient amount of sodium was present in the feed received from the supplier. Therefore, both VSL and PNNL had to mix additional Na into the feed. After receiving the prepared feed at PNNL, NaOH pellets were added directly to the 55-gallons drums that the feed was shipped in to restore the Na concentration to target levels [20.43 g of reagent grade (97%) NaOH per kg of as-received feed]. The adjusted feed was then mixed within the drums for several hours and transferred to the melter feed tank. During the test, samples of the melter feed stream were collected for post-test analytical validation of feed composition. The feed samples were taken from the feed tank during the RSM operation. The sampling log is shown in Appendix B and summarized in Table 5.1 of Section 5.0.

During the test, it was observed that the NaOH pellets had not fully dissolved. The recirculation line was able to pass the pellets but the feed injection line to the melter frequently plugged. The plugs were cleared by water flushes, air blowdowns, and "rodding" out the melter feed nozzle with a wire. The line was also replaced occasionally during the test.

			Glass-Forming and	
	Waste	Waste in	Modifying	Target Glass
Component	Composition	Glass	Additives in Glass	HWL-HCr-16
Al <sub>2</sub> O <sub>3</sub>	44.07	19.83	_	19.83
$B_2O_3$	0.13	0.06	15.00	15.06
Bi <sub>2</sub> O <sub>3</sub>	2.03	0.91	_	0.91
CaO	2.18	0.98	-	0.98
$Cr_2O_3$	4.04	1.82	_	1.82
F	0.14	0.06	-	0.06
$Fe_2O_3$	11.20	5.04	_	5.04
K <sub>2</sub> O	0.86	0.39	6.00	6.39
Li <sub>2</sub> O	0.00	0.00	4.00	4.00
MnO	2.87	1.29	-	1.29
Na <sub>2</sub> O	23.76	10.69	_	10.69
NiO	0.56	0.25	_	0.25
$P_2O_5$	0.84	0.38	_	0.38
PbO	0.70	0.31	-	0.31
$SiO_2$	6.42	2.89	30.00	32.89
WO <sub>3</sub>	0.19	0.09	-	0.09
Sum	100.0	45.00	55.00	99.99 <sup>(a)</sup>
(a) The sum do	es not equal 100.0	00 because of	rounded decimals.	

Table 3.1. HLW-HCr-16 Glass Composition in wt% (Matlack et al. 2014)

One feed sample was analyzed at Southwest Research Institute (SwRI) by ICP-OES, while another sample was analyzed at PNNL using only ion chromatography (IC). At SwRI, samples were digested using hydrochloric and nitric acids in an open vessel. The resulting digestates of the feed samples

contained residue. The remaining residues were separated, dried, and fused with a lithium metaborate/tetraborate mixture. The feed samples were also digested using concentrated nitric, perchloric, hydrofluoric, and hydrochloric acids in an open vessel. All digestions were analyzed. To ensure against false positives, blanks were analyzed intermittently. Calibrations were performed using NIST SRM<sup>1</sup> 278 Obsidian Rock and NIST SRM 688 Basalt Rock laboratory control samples. At PNNL, a subsample of each slurry feed supernate was analyzed directly by IC after filtering the slurry feed through a 0.2-micron filter.

As shown in

<sup>&</sup>lt;sup>1</sup> NIST SRM = National Institute of Standards and Technology Standard Reference Material.

Table 3.2, the SwRI analysis agreed very well with the target composition, as expected. The IC analysis by PNNL was applied to several anion components,  $F^-$ ,  $NO^{2-}$ , and  $NO_3^-$  in the supernate after filtering the slurry feed. The PNNL IC results were consistent among samples and with targets. As shown in Table 3.3, the densities of feed samples were consistent among samples, varying only between 1.41 and 1.43 g/mL.

Measured by:		PN	SwRI	
Sample #:	Target	RSM-EWG8-001 <sup>(a)</sup>	RSM-EWG8-059 <sup>(a)</sup>	RSM-EWG8-041
$Al_2O_3$	7.11	NM	NM	7.26
$B_2O_3$	5.40	NM	NM	5.83
Bi <sub>2</sub> O <sub>3</sub>	0.33	NM	NM	0.340
CaO	0.35	NM	NM	0.371
$Cr_2O_3$	0.65	NM	NM	0.421
F <sup>(b)</sup>	0.02	0.0201	0.0179	0.0157
Fe <sub>2</sub> O <sub>3</sub>	1.81	NM	NM	2.02
K <sub>2</sub> O	2.28	NM	NM	1.90
Li <sub>2</sub> O	1.43	NM	NM	1.43
MgO	-	NM	NM	0.0282
MnO	0.46	NM	NM	0.489
Na <sub>2</sub> O	3.83	NM	NM	3.50
NiO	0.09	NM	NM	0.0895
$P_2O_5$	0.14	NM	NM	0.150
PbO	0.11	NM	NM	0.108
$SO_3$	-	NM	NM	0.0592
SiO <sub>2</sub>	11.79	NM	NM	9.33
TiO <sub>2</sub>	-	NM	NM	0.0115
WO <sub>3</sub>	0.03	NM	NM	0.0267
CO <sub>3</sub> <sup>(b)</sup>	4.26	NM	NM	3.18
NO <sub>2</sub> <sup>(b)</sup>	0.09	0.0936	0.0943	0.0736
NO <sub>3</sub> <sup>(b)</sup>	0.25	0.364	0.357	0.242
$C_2O_4^{(b)}$	3.68	NM	NM	3.06
Total, no water	44.11	NM	NM	39.93
Formulated water	55.89	NA	NA	NA

Table 3.2. Feed Sample Compositions (wt%) Measured by ICP-OES and IC

(a) Sample was only analyzed with IC.(b) Measured by IC.

NM = not measured; NA = not applicable.

MgO, SO<sub>3</sub>, and TiO<sub>2</sub>, are not target components.

Table 3.3. Feed Densities

Sample #:	RSM-EWG8-001	RSM-EWG8-004	RSM-EWG8-013	RSM-EWG8-053	RSM-EWG8-059
Density (g/mL)	1.41	1.43	1.42	1.41	1.41

# 4.0 Run Description

This RSM test was run over a period of 103.4 h,  $9/9/2013\ 0548 - 9/13/2013\ 1312$ , processing ~360 kg of slurry feed and producing ~140.8 kg of glass. The melter operated with continuous feeding, and the glass was sampled periodically. This section describes the melter operation as well as the inspection of the melter after operation was completed.

Prior to melting in the RSM, a preliminary melt of the HLW-HCr-16 feed was performed in a laboratoryscale melter (LSM) at 1150°C to evaluate the melting characteristics and resulting glass product. The LSM consists of a 10-cm-diameter quartz melting chamber that is top-loaded into a laboratory furnace (Kim et al. 2011). Unlike the RSM, there is no bubbler to agitate the melt in the LSM. Before placing the melt chamber into the furnace, the bottom of the chamber was covered with a layer of HLW-HCr-16 glass. After the chamber was lowered into the furnace and the starter glass had melted, the high-Cr feed was introduced into the quartz chamber at rates between 3.0 and 6.5 mL/min using a peristaltic pump. The feed rates of the peristaltic pump were calibrated by operating the pump over a wide range of rotational speed settings and measuring the average feed rate at each setting. The melt was completed with no difficulties, and examination of the resulting glass under an optical microscope found little to no crystallization.

### 4.1 Melter Operation

The glass was melted under operational targets that are discussed in Section 2.3. Several charts are provided in Appendix C to graphically present actual operating conditions that were achieved during testing. The manual log of temperature and electrical data is provided in Appendix A as a reference and supplemental material to the electronically logged data. A summary of the main operating parameters is given in Table 4.1.

Start date	9/9/2013			
Start time	0548			
Total hours of normal operation (h)	103.4			
Total hours of feeding (h)	91.9			
Feeding interruptions (h)	11.5			
Glass Temperature (°C)				
Min. <sup>(a)</sup>	958			
Max. <sup>(a)</sup>	1177			
Avg. glass temperature	1137			
Plenum Temperature (°C)				
Min.	320			
Max.	972			
Avg.	598			
Steady-State <sup>(b)</sup> Plenum Temperature (°C)				
Min.	515			
Max.	622			
Avg.	569			
Melter Vacuum (in. H <sub>2</sub> O)				
Min.	-1.1			
Max.	2.8			
Avg.	0.9			
Average electrode power (kW/m <sup>2</sup> )	109.2			
Average glass resistance (ohms)	8.5			
Bubbling rate (L/min)	4.2			
Target feed rate (L/h) <sup>(c)</sup>	4.1			
Avg. overall feed rate, including feed outages (L/h)	2.65			
Avg. stable processing feed rate(L/h)	2.98			
Avg. glass pour rate (kg/h)	1.36			
Avg. glass pour rate, by melt surface area (kg/day/m <sup>2</sup> )	654			
Steady-state production rate (L/h)	3.33			
Steady-state production rate (kg/day/m <sup>2</sup> ) <sup>(d)</sup>	822			
Max. sustained production (kg/day/m <sup>2</sup> ) <sup>(e)</sup>	964			
(a) Averaged by two thermocouples measuring glass pool temperature simultaneously at				

#### Table 4.1. Summary of RSM Operations

(a) Averaged by two thermocouples measuring glass pool temperature simultaneously at different locations in the melter.

(b) Selected "steady-state" is a 5-h period from 9/10 1505 to 9/10 2004.

(c) Converted by target glass processing rate of  $\sim 1.0 \text{ MT/day/m}^2$ .

(d) Rate calculated from feed rate data.

(e) Rate calculated from feed data based on surface area of the melt and best > 5-h period.

Before the actual feed processing was initiated, the melter was loaded with 6.4 kg of previously melted HLW-HCr-16 glass and heated by gradually increasing the set points for the melter kiln and pour spout heaters. The middle kiln thermocouple measured 862°C and the melter electrode thermocouple measured 747°C before joule heating was started at 0155 hours on Monday, September 9, 2013. The melter reached the targeted glass temperature of 1150°C at 0524 hours on Monday, September 9, 2013. Testing operations were initiated at 0548 hours, although refinements to the processing conditions continued during testing, which produced a total of 140.8 kg of glass. During testing, erroneously fluctuating temperature readings were observed on several of the thermocouples, including the pour spout, kiln, HX, and process water thermocouples. This was corrected by providing a steady ground for all affected thermocouples. Plots of melter power, melter feed rate, plenum temperature, and glass temperature are shown in Figure 4.1 and Figure 4.2.



Figure 4.1. Feed Rate (30-min average) and Electrode Power



Figure 4.2. Melter Glass and Plenum Temperatures

The operation of the RSM was successful and enabled collection of basic operational data, albeit less than ideal due to short steady-state periods. The plenum temperatures ranged between 515°C and 622°C during steady-state operation, in keeping with the 550°C to 700°C target range. The maximum steady-state feed rate was attempted early in the melter run but was not optimized due to feed line plugging and limited durations of steady state cold cap coverage. The temperatures of the bulk glass and electrodes were relatively stable throughout the test, with no electrode temperature data having a standard deviation greater than 17°C over the course of normal operation during the test. Likewise, the standard deviation of the supplied power data was less than 1 kW during the test, meaning good temperature control was maintained without large swings in electrode power.

Feed-line and feed-nozzle plugging problems were the primary cause of disruption during processing and the main obstacle to obtaining a steady equilibrium melt rate. In one case, while clearing a plugged feed nozzle, power to the electrodes was lost due to a loose fuse. Toward the end of the test, cold cap bridging led to a couple of brief pauses in feeding. Interruptions in feeding to resolve these issues totaled around 12 h over the course of the test (~ 103 h) and did not prevent steady-state conditions from being achieved. There was no foaming in the glass discharge or on the melt pool surface. The cold-cap coverage was continuously changing throughout the tests and can roughly be related to plenum temperature trends. However, during most of the testing, it remained within the target range of 80% to 90%. In the feed tank, there was some settling of NaOH pellets that did not completely dissolve. These undissolved pellets contributed to most of the feed line plugging issues that were experienced during the test, which caused

subsequent interruptions to cold cap coverage, plenum temperatures, glass temperatures, glass pouring, and glass chemistry.

The RSM test commenced on September 9, 2013, at 0548 hours with an initial feed rate of 5.0 L/h. The bubbling rate was 4.2 L/min for the duration of the test. Several hours after beginning the test, at 1202 hours, feeding was interrupted for 2 h and 50 min to remove a blockage in the feed nozzle that had to be bored out with a drill. A few hours later, at 1824 hours, another interruption in feeding was initiated to remove another blockage in the feed nozzle. The electrode power was turned off to allow entry into the plenum space and could not be turned back on after the blockage was cleared. While troubleshooting the loss of power, feed remaining in the nozzle solidified and the nozzle had to be removed from the melter to clear the plug. Meanwhile, it was found that a fuse had become loose, causing the loss of power. Normal operation was restored after this second 2-h and 50-min interruption. A little over 2 h later, it was found that the feed recirculation line was plugged and that there was no feed going into the melter. The lines and nozzle were cleared, and feeding was restored after 33 min.

A little less than an hour later, on September 10, 2013 at 0053 hours, feeding was again halted due to a plug in the line from the feed pump. The line and nozzle were cleared and feeding started after an 11-min interruption. After a little more than an hour, feeding was stopped again to clear feed plugs at 0214 hours and reinitiated after 38 min. Slightly over an hour and a half later, the chiller tripped, causing the feed in the nozzle to bake and solidify. Another feed outage began at 0434 hours, during which the feed nozzle was removed and drilled out. The outage ended after 3 h and 9 min. During this outage, the power output was unstable, and the glass temperature dropped below 950°C for several minutes. It should be noted power loss was observed during each of the feed outages (see Appendix C, Figure C.5) due to the low glass level, and the two major interruptions, September 9, 2013 at 1824 to 2115 hours and September 10, 2013 at 0434 to 0744 hours, glass temperature was < 950°C intermittently (Figure 4.2). Temperature data measured from those two periods were not included in the summary in Table 4.1. There was no significant power loss after those two interruptions.

Normal operation was maintained for over 12.5 h before it was found that a hose had to be replaced on September 10, 2013 at 2021 hours. This outage only took 4 min. A 1-min feed interruption was required almost 12 h later, on September 11, 2013 at 0819 hours, to knock down a stalactite that had formed in the melter. Almost 4 h later, at 1214 hours, a 14-min feed outage occurred to remove a feed plug. There was not another feed stoppage for over 33 h, when, on September 12, 2013 at 2133 hours, feeding was interrupted because the cold cap was bridging. After 55 min, the cold cap had dissipated enough to start feeding again. Feeding then continued uninterrupted until the test was concluded on September 13, 2013, at 1312 hours.

As shown in Figure 4.3, with the exception of brief interruptions and two successive data points when the bubbler set points were recorded as 2100 followed by 5000 standard cubic centimeters per minute (sccm), the bubbling rates were steady, oscillating around the 4200 sccm target value without trending higher or lower. The average surface-area-specific bubbling rate, calculated by dividing the bubbling rate of 4200 sccm or 4.2 L/min by the melter surface area of 500 cm<sup>2</sup> or 0.05 m<sup>2</sup>, was 84 L/min/m<sup>2</sup>. There wasn't an entry in the lab record book to suggest that the bubbler set point was intentionally changed or that the change was recognized.



Figure 4.3. Bubbler Flow Rates

### 4.2 Melter System Inspection

At the conclusion of the test, the melter and exhaust lines were inspected. The melter electrodes were removed from the melt while the glass was hot. After the glass cooled, the melter lid and the first section of the exhaust line were disassembled. There was no significant corrosion damage observed. The electrodes were discolored, but it did not appear that a significant amount of metal had been removed. The edges of the electrodes were sharp, as shown in Figure 4.4, although there was evidence of heavy oxidation and very small pits on the surface. The bubbler flow in the RSM was split into two bubbler tubes, which were made from Inconel 690 tubing with a 0.05-in. wall thickness. One of them had been used in previous melter runs, and both were heavily oxidized but not structurally damaged after this melter run. Inspection of the melter lid showed that some entrained material adhered to the underside and to the exhaust piping. The deposits appeared to be entrained feed or glass. The inside of the melt chamber after testing is shown in Figure 4.5. The yellow spots in the melter chamber (Figure 4.5) are likely Cr-containing salts/oxide crystals, which are not observed in output glass samples.



Figure 4.4. Melter Electrode Pulled from the Glass Melt



Figure 4.5. Inside of the Melt Chamber after Testing

Samples of deposits in the offgas line were collected from the RSM after the test. One was taken from the film cooler, one from the 90° bend above the melt chamber, and one from the long tube downstream from the 90° bend. These areas of the offgas line are labeled in Figure 4.6. The collected samples were analyzed using X-ray fluorescence (XRF), ICP-OES, and IC for the given ions and metals provided in Table 4.2. All the elements expected in the target glass composition were detected in the offgas line deposits except Bi, Ni, Pb, and W, each of which was a minor constituent representing less than 1 wt% of the glass recipe on an oxide basis. Some elements found in the chemical analysis were not in the target composition, such as Cl, MgO, SO<sub>3</sub>, and ZrO<sub>2</sub>. Total mass recovery was lower than 100% because some components were not measured (e.g., Bi<sub>2</sub>O<sub>3</sub>). In addition to XRF, Cl and F were measured by IC. F is important for emission analysis, which will be used in mass balance analysis.



Figure 4.6. Areas in Offgas Line from which Samples of Deposit Material Were Collected

Compound	Film Cooler	Long Tube	90° Bend
$Al_2O_3$	13.61	11.27	8.89
$B_2O_3$	18.50	13.20	10.00
CaO	0.71	0.82	0.79
Cl <sup>(a)</sup>	0.22	0.39	0.33
$Cr_2O_3$	1.80	1.49	1.46
Fe <sub>2</sub> O <sub>3</sub>	5.44	4.58	4.33
F <sup>(a)</sup>	0.49	0.45	0.64
K <sub>2</sub> O	7.33	6.48	6.45
MgO	0.06	0.04	0.04
MnO	1.42	0.91	0.88
Na <sub>2</sub> O	11.06	9.14	8.05
$P_2O_5$	0.38	0.93	0.87
SO <sub>3</sub>	0.34	0.00	0.28
SiO <sub>2</sub>	21.35	17.82	14.75
$ZrO_2$	0.05	0.24	0.32
Total <sup>(b)</sup>	83.97	68.33	58.44

Table 4.2. Plenum and Exhaust Line Deposit Composition (wt%) Measured by XRF and IC

(a) Cl and F were analyzed by IC. Others were all analyzed by XRF.

(b) The XRF concentrations sum to less than 100% because some elements could not be analyzed.

# 5.0 Sample Collection and Analysis Methods

Routine sampling of the feed, glass, and offgas streams was conducted throughout the test. The melter feed recirculation loop allowed for direct sampling of the feed stream just before it entered the melter. Glass samples were collected from the melter pour spout stream with rectangular graphite boats. Because the newly formed glass bar could shatter and create a sharp projectile hazard, glass samples were shielded while cooling. These samples were used as the rapidly cooled "quenched" samples for toxicity characteristic leaching procedure (TCLP) tests. The EVS condensate samples were directly extracted from a valve on the condensate recirculation line. The HEME runoff was manually recycled back to the EVS recirculation tank. Accumulated undissolved solids in the quench scrubber's condensate tank were collected; however, the amount was negligible and was not used in mass balance evaluations. The collection and analysis of the RSM offgas is discussed in Section 8.0.

Process samples collected for analysis included the feed slurry, glass product, and EVS scrubbing liquid samples (Appendix B)<sup>2</sup>. In general, process samples were collected at least once per day and for every identified "stable" operating condition except for offgas line deposits and the EVS undissolved solids, which were collected only at the conclusion of testing. Sample analyses were conducted to characterize the quantities, compositions, and properties of these process streams following the protocol called out in the HASQARD (DOE 2014).

Process and offgas samples were analyzed, as applicable, for elemental composition, durability, and density. Some analyses were contemporaneous with test operations. Other analyses required preparations of several hours or days, depending on the analysis performed, the sample preparation required prior to analysis, and the location of the analytical equipment. Table 5.1 briefly describes the different analyses included in this test program. Table 5.2 lists the collected feed, glass, and EVS solution samples for analysis together with the analyses that were performed. For glass samples, the analyses were performed on an as-cooled or quenched glass, while one canister centerline cooling (CCC)-treated sample was prepared by further heat-treating part of the RSM-EWG8-044 quenched sample following the CCC heating profile (Section 6.1). The quenched and CCC samples were compared to investigate the crystallinity under different cooling conditions.

 $<sup>^{2}</sup>$  In the sampling log (Appendix B), a white residue sample in glass canister and two glass samples from melt pool surface were also collected but not used for analysis.

Analysis	Sample Matrix	Analysis Method	Analysis Description
Cations	Solid or liquid	ICP-OES, XRF	Analysis of total amount of element, regardless of speciation
Anions	Liquid	IC	Br <sup>-</sup> , Cl <sup>-</sup> , NO <sub>3</sub> <sup>-</sup> , NO <sub>2</sub> <sup>-</sup> , PO <sub>4</sub> <sup>3-</sup> , SO <sub>4</sub> <sup>2-</sup> , F <sup>-</sup>
Durability	Solid (glass)	TCLP	ASTM and EPA procedures (ASTM C 1285-02(2008); EPA 1992)
Density	Liquid (feed)	Mass/Volume	Mass/Volume

Table 5.1. Sample Analysis Methods for Process (feed slurry, glass, and EVS) and Offgas Samples

Table 5.2. List of Feed, Glass, and EVS Solution Samples Analyzed

Sample Type	ID	Analyses	
Feed	RSM-EWG8-001	IC, Density	
	RSM-EWG8-004	Density	
	RSM-EWG8-013	Density	
	RSM-EWG8-053	Density	
	RSM-EWG8-059	IC, ICP-OES, Density	
Glass	RSM-EWG8-003	XRF	
	RSM-EWG8-014	XRF	
	RSM-EWG8-018	IC, ICP-OES, TCLP	
	RSM-EWG8-032	XRF	
	RSM-EWG8-035	IC, ICP-OES	
	RSM-EWG8-044 <sup>(a)</sup>	$\eta^{(b)}$ , SEM, XRD, Liquidus Temperature	
	RSM-EWG8-048	XRF	
	RSM-EWG8-057	XRF, ICP-OES	
	Mixture of Samples	EC <sup>(b)</sup>	
EVS Solution	RSM-EWG8-002	IC, ICP-OES	
	RSM-EWG8-019	IC, ICP-OES	
	RSM-EWG8-029	IC, ICP-OES	
	RSM-EWG8-045	IC, ICP-OES	
	RSM-EWG8-058	IC, ICP-OES	

(a) A CCC-treated specimen was also prepared and analyzed by scanning electron microscopy (SEM) and XRD.

(b) Viscosity ( $\eta$ ) and electrical conductivity (EC) were measured on glass melts by re-melting of the listed samples.

# 6.0 Glass Characterization

This section describes the test results for the chemical composition, crystallinity, TCLP, electrical conductivity, and viscosity for the RSM-produced glass.

Over the course of the test, 140.8 kg of glass was produced. Quenched glass samples were collected in the manner described in Section 5.0, and were used for most of the characterizations. In addition, selected quenched samples were heat treated for crystallinity analysis.

### 6.1 Canister Centerline Cooling Heat Treatment of Glass

One selected glass sample, RSM-EWG8-044, was heat treated following the HLW CCC profile per CCN 074851.<sup>3</sup> Glass samples were placed in a Pt-10% Rh crucible, covered with a lid, and placed in a high-temperature furnace at 1050°C for 1 h. They were then cooled using a programmable furnace controller following the CCC schedule shown in Table 6.1 and Figure 6.1. Some of the samples from the CCC heat treatment were characterized by SEM, XRD, and product consistency test.

	Time	Start Temp.	Rate
Segment	(min)	(°C)	(°C/min)
1	0–45	1050	-1.556
2	45-107	980	-0.806
3	107-200	930	-0.591
4	200-329	875	-0.388
5	329–527	825	-0.253
6	527-707	775	-0.278
7	707-1776	725	-0.304

Table 6.1. Temperature Schedule for CCC Treatment

<sup>&</sup>lt;sup>3</sup> Memorandum, "Canister Centerline Cooling Data," CCN 074851, Rev. 1, RPP-WTP, Richland, WA, October 29, 2003.



Figure 6.1. Graph of CCC Heat Treatments of HLW Glass

### 6.2 Analyses of Glass for Chemical Composition

To confirm that the fabricated glass composition agreed with the target, quenched glass samples RSM-EWG8-018 and RSM-EWG8-035 were chemically analyzed at SwRI and sample RSM-EWG8-057 was analyzed at PNNL. At SwRI, the samples were prepared for analysis using three techniques. The first technique was a closed vessel digestion using concentrated nitric, hydrochloric, and hydrofluoric acids. Boron and silicon were reported from this digestion. The second technique was a lithium metaborate/tetraborate fusion. Aluminum, calcium, chromium, iron, and sodium were reported from this fusion. The third technique used concentrated nitric, perchloric, hydrofluoric, and hydrochloric acids in an open vessel. The remaining metals were reported from this digestion. Blanks were run to ensure that no analytes were falsely detected above SwRI's reporting limits. Two solid laboratory control samples (NIST SRM<sup>4</sup> 278 Obsidian Rock and NIST SRM 688 Basalt Rock) were used during all sample preparation techniques. Aqueous laboratory control samples were also digested for calibration.

Additionally, SwRI performed IC analysis on the samples. Approximately 0.25 g of the sample was extracted with 50 mL of deionized water. The extract was analyzed for fluoride, chloride, nitrate, nitrite, phosphate, oxalate, and sulfate. Another ~0.20 g of the sample was fused using sodium carbonate and analyzed for total fluorine, chlorine, and sulfur. The fusion was diluted to 50 mL using deionized water. Due to the high sodium carbonate concentration, it was diluted an additional 10× prior to analysis. IC analyses followed SW-846 Method 9056 (EPA 1994) and SwRI procedure TAP 01-0406-042, Rev. 6.<sup>5</sup>

At PNNL, samples were prepared by finely grinding ~0.1 g of glass with an agate mortar and pestle and digesting the resulting powder in 9 mL of concentrated nitric acid, 3 mL of concentrated hydrofluoric acid, and 2 mL of concentrated hydrochloric acid at 210°C for 30 min. After cooling, 30 mL of 5% boric acid solution was added and further digestion took place at 180°C for 25 min. Each sample was poured

<sup>&</sup>lt;sup>4</sup> NIST SRM = National Institute of Standards and Technology Standard Reference Material.

<sup>&</sup>lt;sup>5</sup> SWRI. 2012. *Inorganic Anions and Disinfection Byproducts using Ion Chromatography*. Test/Analytical Procedure (TAP) 01-0406-042, July 2012, Rev. 6. Southwest Research Institute, San Antonio, TX.

into a 50-mL centrifuge tube and the digestion vessel was rinsed three times with a 1-mL aliquot of deionized water to ensure the entire sample had been transferred. The centrifuge tube was then capped and shaken to homogenize the solution. The tube was weighed, and a 10-mL sample was removed and filtered using a 0.45- $\mu$ m polyvinylidene fluoride or polyvinylidene difluoride syringe filter and analyzed by ICP-OES. Three 1-mL samples were taken from the remaining solution and weighed to determine density so that the final weight of the solution could be density corrected.

The ICP-OES results from the two labs are summarized in Table 6.2, which provides the measured composition of the glass together with the targeted composition and the relative differences between the measured and targeted values for major components (those with target concentrations >2 wt%). Although PNNL did not measure as many of the constituent elements as SwRI, the measured concentrations of the major components measured by PNNL were similar to those measured by SwRI and within error of the target composition. The major glass components measured at PNNL were within  $\pm$ 7% of the target concentration, indicating that the glass was very close to the target composition. The two major components not measured by PNNL, SiO<sub>2</sub> and B<sub>2</sub>O<sub>3</sub> (as the use of hydrofluoric acid and boric acid preclude analysis of SiO<sub>2</sub> and B<sub>2</sub>O<sub>3</sub>, respectively), were also within 7% of their target concentrations in the samples measured at SwRI.

Five of the melter glass samples were also analyzed by XRF (Table 6.3). The results of the major components show good agreement with the target composition. Light elements such as boron and lithium cannot be detected by XRF. All samples from early to late stages of the melter run have very similar compositions, which demonstrates consistency of the glass production during the melter operation.
	Measured by:		Sw	PNNL			
	Sample ID:	RSM-EWG	8-018	RSM-EWC	38-035	RSM-EWC	38-057
	Targeted	Measured	%	Measured	%	Measured	%
Oxide	(wt%)	(wt%)	Diff	(wt%)	Diff	(wt%)	Diff
Al <sub>2</sub> O <sub>3</sub>	19.83	19.07	-3.8	18.84	-5.0	20.03	1.0
$B_2O_3$	15.06	14.42	-4.3	14.12	-6.3	NM	NA
$Bi_2O_3$	0.91	0.63	-30.6	0.55	-39.1	0.63	-31.1
CaO	0.98	0.96	-2.1	0.95	-3.4	0.95	-2.6
$Cr_2O_3$	1.82	1.62	-10.8	1.64	-10.2	1.64	-10.1
F	0.06	ND	NA	ND	NA	NM	NA
$Fe_2O_3$	5.04	4.66	-7.5	4.68	-7.1	5.02	-0.4
K <sub>2</sub> O	6.39	5.67	-11.2	5.76	-9.9	5.94	-7.1
Li <sub>2</sub> O	4.00	3.78	-5.6	3.80	-5.0	3.81	-4.8
MgO	-	< 0.01	NA	0.02	NA	0.09	NA
MnO	1.29	1.20	-7.1	1.19	-7.8	NM	NA
Na <sub>2</sub> O	10.69	9.40	-12.1	9.34	-12.6	10.14	-5.2
NiO	0.25	0.28	10.0	0.25	-0.8	0.21	-17.5
$P_2O_5$	0.38	0.28	-25.8	0.26	-30.5	NM	NA
PbO	0.31	0.24	NA	0.22	NA	0.26	NA
SiO <sub>2</sub>	32.89	34.72	5.6	34.94	6.2	NM	NA
$SO_3$	_	0.06	NA	0.06	NA	NM	NA
TiO <sub>2</sub>	-	0.03	NA	0.03	NA	NM	NA
$WO_3$	0.09	0.09	3.0	0.09	4.7	NM	NA
ZnO	_	0.01	NA	< 0.01	NA	NM	NA
ZrO <sub>2</sub>	-	0.01	NA	0.01	NA	NM	NA
Sum	100	97.10	-2.9	96.66	-3.3	NA	NA

Table 6.2. Composition of HLW-HCr-16 Glass Samples Analyzed by ICP-OES at SwRI and PNNL

% Diff = % difference of measured vs. targeted.

NM = not measured; NA = not applicable; ND = not detected.

MgO, SO<sub>3</sub>, TiO<sub>2</sub>, ZnO, and ZrO<sub>2</sub> are not target components.

S	ample ID:	RSM-EWO	38-003	RSM-EWO	<b>G8-014</b>	RSM-EWO	58-032	RSM-EWO	38-048	RSM-EWO	38-057
	Targeted	Measured	%	Measured	%	Measured	%	Measured	%	Measured	%
Oxide	(wt%)	(wt%)	Diff	(wt%)	Diff	(wt%)	Diff	(wt%)	Diff	(wt%)	Diff
$Al_2O_3$	19.83	20.50	3.4	21.70	9.4	20.32	2.5	21.70	9.4	21.47	8.3
$B_2O_3$	15.06	NM	NA	NM	NA	NM	NA	NM	NA	NM	NA
Bi <sub>2</sub> O <sub>3</sub>	0.91	0.74	-18.7	0.79	-13.2	0.73	-19.8	0.80	-12.2	0.69	-24.3
CaO	0.98	1.23	25.5	1.11	13.3	1.06	8.2	1.02	4.1	0.99	1.2%
$Cr_2O_3$	1.82	1.61	-11.5	1.77	-2.7	1.68	-7.7	1.69	-7.4	1.67	-8.5
F	0.06	NM	NA	NM	NA	NM	NA	NM	NA	NM	NA
Fe <sub>2</sub> O <sub>3</sub>	5.04	5.35	6.2	5.54	9.9	5.41	7.3	5.35	6.2	5.26	4.3
K <sub>2</sub> O	6.39	6.21	-2.8	6.63	3.8	6.51	1.9	6.45	1.0	6.53	2.2
Li <sub>2</sub> O	4.00	NM	NA	NM	NA	NM	NA	NM	NA	NM	NA
MgO	_	ND	NA	0.04	NA	0.05	NA	0.09	NA	ND	NA
MnO	1.29	1.40	8.5	1.53	18.6	1.52	17.8	1.49	15.5	1.51	16.8
Na <sub>2</sub> O	10.69	9.80	-8.3	10.00	-6.5	9.70	-9.3	9.79	-8.4	9.99	-6.5
NiO	0.25	0.30	20.0	0.30	20.0	0.28	12.0	0.26	4.4	0.26	3.6
$P_2O_5$	0.38	0.45	18.4	0.41	7.9	0.41	7.9	0.37	-2.6	0.39	2.4
PbO	0.31	0.39	25.8	0.40	29.0	0.40	29.0	0.37	18.7	0.36	17.4
SiO <sub>2</sub>	32.89	34.40	4.6	36.64	11.4	35.65	8.4	36.20	10.1	36.38	10.6
$SO_3$	-	0.06	NA	0.07	NA	0.08	NA	0.08	NA	0.06	NA
$WO_3$	0.09	NM	NA	NM	NA	NM	NA	NM	NA	NM	NA
ZnO	_	0.02	NA	0.01	NA	0.00	NA	0.00	NA	0.01	NA
$ZrO_2$	_	0.04	NA	0.00	NA	0.00	NA	0.00	NA	0.00	NA
Sum	100	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA

Table 6.3. Composition of HLW-HCr-16 Glass Samples Analyzed by XRF at PNNL

% Diff = % difference of measured vs. targeted.

NM = not measured; NA = not applicable; ND = not detected. MgO, SO<sub>3</sub>, ZnO, and ZrO<sub>2</sub> are listed as impurities.

#### 6.3 Crystallinity by Scanning Electron Microscopy and X-Ray Diffraction

Figure 6.2 is an SEM back-scattered electron micrograph of a cross-section of a quenched specimen (glass sample ID: RSM-EWG8-044) together with the results of the energy dispersive X-ray spectroscopy (EDS) analysis of designated regions. The micrograph reveals a few clusters of crystals ranging in size up to ~10  $\mu$ m (EDS regions 2-5) and several smaller isolated crystals that are on the order of 1  $\mu$ m in the surrounding glass matrix (EDS region 1). The crystals in regions 2-5, while very similar in composition to one another, were enriched in Fe, Ni, Mg, Mn, and Cr, which are the major constituents of spinel phases typically detected in waste glass (Barnes and Larson 1981; Matlack et al. 2009; Rose et al. 2011) as compared to the glass matrix composition in region 1.

The micrograph in Figure 6.3 shows a cross section of a specimen from the same sample (RSM-EWG8-044) that underwent CCC heat treatment. After CCC heat treatment, the crystal formations (regions 3-6) were very similar to those in the quenched specimen described above. The relative difference in chemical composition as compared to the surrounding glass matrix (region 1) was also similar with the same elements, showing increased or decreased concentrations in the crystals. As compared to the crystalline phase composition in the quenched glass, the crystals present after CCC generally exhibited lower Cr and Mn concentrations and higher levels of Na, Al, and Si. The main difference in morphology between the quenched and CCC glasses was the presence of what appears to be a large, ~80-µm faceted crystal that has the same dark gray contrast as the glass matrix and that appeared to encapsulate some of the smaller, lighter-colored crystals. EDS shows that this area contains higher concentrations of Na, Al, and Si, which are the major constituents of nepheline, and lower concentrations of K, Ca, Mn, and Fe than the glass matrix.



Spectrum	1	2	3	4	5
0	63.85	55.37	56.16	55.55	55.38
Na	8.40	0.48	0.02		0.39
Mg		0.34	0.22	0.28	0.42
Al	9.19	2.19	2.12	2.17	2.25
Si	12.39	0.60	0.30	0.02	0.30
К	3.98	0.18	0.14		0.13
Са	0.56				
Cr		25.34	24.75	26.53	25.74
Mn	0.28	4.80	4.48	4.68	4.61
Fe	1.36	9.11	9.80	9.24	9.29
Ni		1.59	2.01	1.54	1.49

Figure 6.2. Back-Scattered Electron Micrograph and EDS of Quenched Glass Analysis of a Polished Cross-Section of a Quenched Specimen from Sample RSM-EWG8-044



Spectrum	1	2	3	4	5	6
0	67.88	60.64	62.61	60.53	63.07	62.78
Na	8.34	11.36	1.02	3.33	3.88	2.10
Mg			0.31	0.15	0.30	0.27
Al	8.01	11.23	1.83	3.61	4.17	2.85
Si	11.11	13.78	0.86	2.30	3.39	1.67
К	3.22	2.44	0.12	0.34	0.50	0.37
Са	0.43	0.00				
Cr			15.88	15.72	12.15	15.37
Mn	0.26	0.06	3.95	3.53	2.87	3.55
Fe	0.74	0.48	11.87	9.21	8.58	9.88
Ni			1.54	1.29	1.08	1.16

Figure 6.3. Back-Scattered Electron Micrograph and EDS of CCC Glass Analysis of a Polished Cross-Section of a CCC Specimen from Sample RSM-EWG8-044 XRD patterns were collected using a Bruker D8 X-ray diffractometer by scanning over the range of  $5-80^{\circ} 2\theta$  with a step size of  $0.015^{\circ}$  and a 53.1-s dwell at each step. Data were analyzed using EVA v.14 and TOPAS v.4.2 software (Bruker AXS, Karlsruhe, Germany) for phase identification. For the purposes of semiquantitative analysis, 5 wt% CaF<sub>2</sub> was added to each sample as an internal standard. The crystalline content was determined by Rietveld refinement of the XRD patterns using TOPAS software.

Figure 6.4 is an XRD pattern of quenched glass taken from the same sample as the SEM-EDS sample shown in Figure 6.2. The XRD pattern confirmed the microscopy results, identifying that the spinel crystal structure was indeed present in a concentration of 2.46 wt% (calculated by the 4.99 wt% CaF<sub>2</sub> standard). The best match in the XRD pattern database was trevorite (NiFe<sub>2</sub>O<sub>4</sub>), but based on the EDS results discussed above, the spinel crystals are likely a solid solution of trevorite, chromite (FeCr<sub>2</sub>O<sub>4</sub>), nichromite (NiCr<sub>2</sub>O<sub>4</sub>), manganochromite (MnCr<sub>2</sub>O<sub>4</sub>), and magnesiochromite (MgCr<sub>2</sub>O<sub>4</sub>) all of which are forms of spinel.

Figure 6.5 is an XRD pattern of CCC-treated glass taken from the same sample as the SEM-EDS sample shown in Figure 6.3. A spinel phase was again detected, this time at a slightly higher concentration of 3.40 wt% (calculated based on the 4.99 wt% CaF<sub>2</sub> standard). Nepheline was also detected, at a concentration of 2.56 wt% (calculated based on the 4.99 wt% CaF<sub>2</sub> standard). This likely represents the large crystal found microscopically in Figure 6.3.

#### EWG-RPT-028, Rev. 0.0 PNNL-30141, Rev. 0.0



Figure 6.4. XRD Pattern of Quenched Glass (sample ID RSM-EWG8-044) Examined microscopically in Figure 6.2

#### EWG-RPT-028, Rev. 0.0 PNNL-30141, Rev. 0.0



Figure 6.5. XRD Pattern of CCC Glass (sample ID RSM-EWG8-044) Examined Microscopically in Figure 6.3

01-084-0686 (N) - Nepheline, syn - K.48Na3.48(Al.99Si1.01O4)4 - Y: 26.99 % - a 9.98900 - b 9.98900 - c 8.38000 - alpha 90.

#### 6.4 Liquidus Temperature

XRD analysis of sample RSM-EWG8-044 was used to determine the liquidus temperature of crystalline content as a function of temperature. XRD patterns were collected after the glass had been heat treated at 950°C, 1000°C, and 1050°C for 20 h. The resulting data are shown in Figure 6.6. From extrapolation of the linear fit of temperature versus crystal wt%, the estimated liquidus temperature was 2075°C. The temperature at 1 vol% spinel was 1786°C. It should be noted that a fourth data point at 1100°C was removed as an outlier; the liquidus temperature by fitting three data points was for reference only.



Figure 6.6. Temperature vs. Spinel Content for HLW-HCr-16 Glass Melted at PNNL

#### 6.5 Toxicity Characteristic Leaching Procedure

A quenched sample of HLW-HCr-16 glass with sample ID RSM-EWG8-018 was sent to SwRI for TCLP analysis, following EPA Test Method 1311 (EPA 1992). Table 6.4 shows that the quenched glass is well below EPA regulatory limits for all regulated elements. However, the CCC glass with the extra crystallization was not tested.

CAS No.	Analyte	Concentration (ppb)	EPA Regulatory Limits <sup>(a)</sup> (ppb)
7440-38-2	As	20	5000
7440-39-3	Ba	50	100000
7440-43-9	Cd	5	1000
7440-47-3	Cr	17.6	5000
7439-92-1	Pb	329	5000
7439-97-6	Hg	0.20	200
7782-49-2	Se	10.0	1000
7440-22-4	Ag	10.0	5000
(a) 40 CFR 2	261.25, 1997.		

Table 6.4. TCLP Concentrations for HLW-HCr-16 Glass (sample ID RSM-EWG8-018)

### 6.6 Electrical Conductivity

The electrical conductivity of the high-Cr glass was measured at temperatures between 940°C and 1240°C on a mixture of glass collected from several pours during the melter run that were crushed together. Impedance spectroscopy was performed using a Solartron 1470E potentiostat/galvanostat coupled with a Solartron 1400 frequency analyzer (Solartron Analytical, Oak Ridge, TN) while cooling through the temperature range.<sup>6</sup> Measurements were performed with the glass in a Pt-10% Rh crucible inside a DT-31-RS furnace (Deltech, Denver, CO). The impedance probe used for these measurements consisted of two paddles made of Pt-10% Rh (7 mm wide  $\times$  12.7 mm long) spaced 9.32 mm apart. Duplicate or triplicate impedance measurements were performed at several temperatures. Prior to the measurements, the cell constant for the impedance analyzer was determined with 0.1 M and 1 M KCl reference solutions at room temperature. The conductivity results for the HLW-HCr-16 glass are presented in Table 6.5 and Figure 6.7 together with the conductivities measured for the DWPF Environmental Assessment Standard Reference Material glass. The electrical conductivity of the HLW-HCr-16 glass is slightly lower than that of the DWPF reference but has a very similar activation energy. At 1140°C (slightly off from target temperature of 1150°C), the electrical conductivity is ~22.3 S/m.

<sup>&</sup>lt;sup>6</sup> Crum JV. 2012. *PNWD Procedure: High-Temperature Electrical Conductivity*. GDL-Elec-Test-01, Rev. 0, Pacific Northwest National Laboratory, Richland, WA.

	Temperature	Impedance, 1 kHz	EC
Glass	(°C)	(ohms)	(S/m)
	1187	0.926	28.64
	1187	0.922	28.77
	1188	0.92	28.83
DWDE	1089	1.293	20.51
DWPF	1089	1.297	20.45
	1089	1.299	20.42
	990	1.989	13.34
	990	2.049	12.95
	1236	0.946	29.55
	1236	0.944	29.59
	1140	1.255	22.26
HLW-HCr-16	1042	1.726	16.19
	1041	1.726	16.19
	983	2.685	10.40
	983	2.69	10.39

Table 6.5. Electrical Conductivity of HLW-HCr-16 Glass and DWPF Reference Glass



Figure 6.7. Electrical Conductivity of HLW-HCr-16 Glass and DWPF Reference Glass

### 6.7 Viscosity

Viscosity ( $\eta$ ) of the glass was measured as a function of temperature using a rotating-spindle digital viscometer according to PNNL procedure GDL-Visc-Test-01.<sup>7</sup> Prior to viscosity measurements, the instrument was calibrated using DWPF startup frit (Crum et al. 2012) and PNNL procedure GDL-Visc-Test-01. GDL-Visc-Test-01 complies with ASTM C965 Method A such that the spindle was rotated, and the crucible was fixed. A total of 50 mL of glass from the sample labeled RSM-EWG8-044 was heated to ~1150°C in a Pt-10% Rh crucible and maintained until thermal equilibrium was reached. An initial torque reading at a constant spindle speed was taken at 1150°C, with measurements taken after the glass was cooled to 1050°C and again at 950°C. The glass was then heated back to 1150°C and further to 1250°C, then cooled back to 1150°C, with additional measurements made at each temperature. The ramp rates were 10°C/min. The measured viscosities at the different temperatures are shown in Table 6.6. The viscosity data are plotted in logarithmic scale as a function of 1/T in Figure 6.8. Arrhenius equation was applied to fit the data. Calculated by the fitting parameter, the viscosity at 1150°C is 5.8 Pa-s (fitted by Arrhenius equation as shown in Figure 6.8), which is within the acceptable range of 4 to 6 Pa-s for HLW glass (Vienna et al. 2013).

Set Temperature	Measured Temperature	Viscosity
(°C)	(°C)	(Pa-s)
1150	1126	6.576
1050	1032	17.087
950	935	55.002
1150	1115	6.948
1250	1219	3.496
1150	1150	6.443

Table 6.6. Viscosity of HLW-HCr-16 Glass (sample ID RSM-EWG8-044)

<sup>&</sup>lt;sup>7</sup> Crum JV. 2012. *PNWD Procedure: High-Temperature Viscosity Measurement*. GDL-Visc-Test-01, Rev. 0, Pacific Northwest National Laboratory, Richland, WA



Figure 6.8. Viscosity of HLW-HCr-16 Glass as a Function of Temperature (Sample RSM-EWG8-044)

## 7.0 Scrubber Solution Analysis

As described in Section 2.2 and shown in the flow diagram in Figure 2.1, the EVS condenses water from the melter exhaust and samples were collected. This section summarizes chemical analysis results of scrubber solution or EVS solution samples.

Like the feed samples, EVS samples were analyzed by ICP-OES and IC at SwRI and PNNL. At SwRI, samples were digested using hydrochloric and nitric acids in an open vessel. The digestates for the scrub solution samples did not contain residues. At PNNL, the samples were filtered with a 0.2-micron filter and analyzed directly.

The results of ICP-OES and IC analyses of five EVS solution specimens at PNNL and one specimen at SwRI are given in Table 7.1 and show that the concentrations of most of the measured elements in the scrubber solution were less than 10 parts per million (ppm). Only F, Bi, B, Li, K, Na, and S were detected at levels above this threshold, and their concentrations increased with time due to accumulation in the scrubber tank, as expected. There was agreement between most of the concentrations measured for sample RSM-EWG8-058 at PNNL and SwRI.

Measuring Lab:			PNNL			SwRI
Sample ID:	RSM-EWG8- 002	RSM-EWG8- 019	RSM-EWG8- 029	RSM-EWG8- 045	RSM-EWG8- 058	RSM-EWG8- 058
Al	0.247	0.445	0.369	0.369	0.604	ND
Bi	ND	ND	ND	ND	ND	85.2
В	25	139	160	193	216	222
Cd	0.233	ND	ND	ND	ND	0.4
Ca	19.6	8.51	4.68	5.23	2.14	4.88
Cr	0.587	1.58	1.5	1.65	1.95	2.98
F <sup>(a)</sup>	ND	58.5	70.5	65.5	78.5	68.3
Fe	ND	ND	ND	ND	ND	10
Pb	ND	ND	ND	ND	ND	2.84
Li	4.51	18.8	20.3	23.8	25.7	28.4
Mg	6.77	4.81	4.14	3.45	2.95	3.15
Mn	ND	0.101	0.115	0.115	0.104	2.85
Mo	0.219	0.234	0.258	0.245	0.26	ND
Ni	ND	0.519	0.592	0.682	0.728	1.23
NO <sub>2</sub> <sup>(a)</sup>	ND	ND	ND	ND	ND	78.5
K	4.36	105	122	156	180	182
Si	3.92	5.02	4.44	3.88	4.08	13.9
Na	38.7	107	113	131	141	162
Sr	0.186	ND	ND	ND	ND	ND
S	10.1	38	44	51.6	59.1	63.4
W	NM	NM	NM	NM	NM	0.95

Table 7.1. EVS Solution Sample Compositions (ppm) Measured by ICP-OES

ND = not detected; NM = not measured.

## 8.0 Offgas Sample Analysis

Section 7.0 reports the RSM exhaust processed by the EVS system. The melter offgas was also directly sampled and analyzed. The offgas sampling port within the RSM system is shown in Figure 2.1 and Figure 8.1. This section presents analyses of particulate matter (PM) and gas species in the RSM offgas.



Figure 8.1. Sampling Locations. (a) PM sampling port on top of the RSM (next to the 90° bend in exhaust pipe); (b) PM sampling port with PM filter installed; (c) PM filter, the orange arrows mark the PM sampling Line, with the main exhaust pipe on the bottom; (d) gas sampling port in horizontal section of exhaust pipe

### 8.1 Sampling and Analytical Procedures

AmTest Air Quality (Auburn, WA) was contracted by PNNL to perform offgas sample analysis of the RSM exhaust. Testing was performed in accordance with test methods outlined in 40 CFR 60, Appendix A, and the Emission Measurement Technical Information Center's website, Test Methods section.<sup>8</sup> The following equipment and analytical methods were used during the analysis.

Flow rate:	Hot wire anemometer supplied by PNNL
CO <sub>2</sub> and O <sub>2</sub> :	EPA Method 3A [nondispersive infrared (NDIR) and paramagnetic analyzers]
Moisture:	EPA Method 4 (incorporated w/ isokinetic sampling method)
PM:	EPA Method 5 (filterable PM; single point isokinetic sampling)
NO <sub>x</sub> :	EPA Method 7E (chemiluminescent analyzer)
CO:	EPA Method 10 (gas filter correlation NDIR analyzer)

Offgas emissions in the RSM exhaust were analyzed for nine separate 70- to 100-min sample periods. The EPA Method 5 sampling rate ( $\Delta P$ ) was back-calculated using cubic feet per minute data from PNNL's mass flow meter, which reported wet standard cubic feet per minute (wscfm) corrected to 70°F. The average flow rate during each test period was corrected to dry standard cubic feet per minute (dscfm) at 68°F to be consistent with EPA test methods. The stack gas velocity was back-calculated in feet per second and converted to airflow in actual cubic feet per minute. The back-calculated  $\Delta P$  and measured gas temperature were used to calculate the percent isokinetics for each run. Because of the complexity of the back-calculations, the percent isokinetics for these tests may not be as meaningful as traditional EPA Method 5 sampling performed in larger area stacks. Since this testing was performed with the nozzle inserted into a 2-in. duct, the normal acceptance criterion for isokinetics (100 ± 10%) should be less stringent. The percent isokinetics for each test period is shown in Table 8.1. This table also summarizes average feed rate and stack gas moisture, and lists data analyzed for each test. Feed rate of the nine tests is plotted in Figure 8.2. Feed rate shows large variations during the test periods; however, the average feed rates of all tests are in a reasonable range as listed in Table 8.1.

Test periods #1 and #2 were considered trial runs because the team was fine-tuning how the backcalculations for the isokinetics would be performed and the RSM was experiencing clogging issues with the feeder system during this time. The offgas samples taken during test period 3 also may not be representative since the RSM was not feeding product during portions of the test period due to continuing feed clogging issues. As shown in Figure 8.2, tests 4 and 5 show large fluctuations of feed rate. After examining the offgas analysis data, tests 4 and 5 were only used for comparing averaged NO<sub>x</sub> and CO with averaged feed rate in the following Section 8.2.

Samples taken during test periods 6 through 9 were determined to be the most appropriate for the gas analysis and PM chemical analysis because the RSM was operating in a stable condition during this time. Approximately 31 dry standard cubic feet (dscf) of melter exhaust gas was sampled at a rate between 0.31 and 0.38 dscfm during these four test periods. Particulate emissions are particularly sensitive to cold-cap coverage in the melt cavity, and the feed rate and bubbler flow rates were adjusted as necessary to control cold-cap coverage. The cold-cap coverage ranged from 75% to 85% during the four test periods, which was reasonably close to the target cold-cap coverage of 80% to 90% as desired for continuous melter operation.

<sup>&</sup>lt;sup>8</sup> https://www.epa.gov/emc/emc-promulgated-test-methods

Test	Date	e/Time		Test Period	Average feed	Isokinetics	Gas Moisture	Data						
Period #	Test Date	Start	End	(min)	rate (g/min)	(%)	(%)	analyzed						
1	9/10/2013	1313	1423	70	78.78	187	21.01	None						
2	9/10/2013	1513	1633	80	78.40	152	22.62	None						
3	9/11/2013	0837	0957	80	56.57	93	19.82	None						
4	9/11/2013	1032	1147	75	38.54	111	18.66	Offgas <sup>(a)</sup>						
5	9/11/2013	1244	1414	90	68.62	99	17.18	Offgas <sup>(a)</sup>						
6	9/11/2013	1446	1616	90	77.97	89	17.55	Offgas, PM						
7	9/12/2013	0754	0934	100	49.53	88	14.65	Offgas, PM						
8	9/12/2013	1003	1123	80	58.08	98	13.76	Offgas, PM						
9	9/12/2013	1155	1320	85	57.87	100	14.99	Offgas, PM						
(a) O	nly average N	$NO_x$ and	CO con	centration was	(a) Only average $NO_x$ and CO concentration was used for Test 4 and 5.									

Table 8.1. RSM PM Sampling Conditions



Figure 8.2. Feed Rate (30-min average, sections of Figure 4.1) of Offgas Test Periods (TPs)

### 8.2 NO<sub>x</sub> and CO Emissions

EPA Methods 3A, 7E, and 10 (see Section 8.1) were performed on the RSM exhaust gas during nine sample periods, lasting 70 to 100 min each, to quantify  $O_2$ ,  $CO_2$ ,  $NO_x$ , and CO concentrations. Airflow data collected during concurrent PM sampling test periods were used to calculate  $NO_x$  and CO emission rates.  $NO_x$  and CO emission concentrations in dry parts per million by volume (ppmvd) were measured using a chemiluminescent analyzer and a gas filter correlation NDIR analyzer, respectively. Oxygen and  $CO_2$  concentrations in dry percent were measured using a paramagnetic analyzer and NDIR analyzer, respectively. The data were recorded using an automated data acquisition system and the concentrations were recorded once per minute during each test period. A schematic of the  $NO_x$  and CO sampling system is shown in Figure 8.3.



Figure 8.3. EPA Methods 3A, 7E, and 10 Analyzer Sample System Diagram

Data collected during test periods 1 through 3 were not analyzed because of the melter feed clogging issues mentioned earlier. Feed rate was unstable for test periods 4 and 5. Gaseous emissions rates of  $NO_x$  and CO for test periods 6 through 9 represented the most reliable set of data and are shown in Table 8.2. CO emissions are considerably higher than  $NO_x$  emissions during all test periods. Average gaseous emissions of  $NO_x$  and CO increased with increasing feed rate, as shown in Figure 8.4 and Figure 8.5.

	1	Test Period 6	j		r	Test Period 7	7	
	09/11	1/13, 1446 –	1616,	-	09/11/13, 0754 – 0934, Avg. Feed Rate 49.53 g/min			
	Avg. Fe	eed Rate 77.9	7 g/min	-				
	Avg.	Min.	Max.	_	Avg.	Min.	Max.	
NO <sub>x</sub> (ppmvd)	94.19	56.12	160.58		70.21	33.52	127.13	
CO (ppmvd)	421.55	288.86	758.83		268.93	126.49	496.20	
$H_2O \ vol\%^{(a)}$	17.55	_	_		14.65	_	_	
CO <sub>2</sub> vol%	0.98	0.88	1.10		0.59	0.30	0.90	
	Test Period 8				Test Period 9			
	09/12	2/13, 1003 – 1	1123,		09/12/13, 1155 – 1320,			
	Avg. Fe	eed Rate 58.0	8 g/min	_	Avg. Fe	ed Rate 57.8	7 g/min	
	Avg.	Min.	Max.	_	Avg.	Min.	Max.	
NO <sub>x</sub> (ppmvd)	68.70	39.98	156.32	_	91.68	40.46	146.64	
CO (ppmvd)	236.53	120.35	511.14		400.97	211.61	604.78	
$H_2O \ vol\%^{(a)}$	13.76 – –			14.99	_	_		
CO <sub>2</sub> vol%	0.56	0.42	0.86		0.77	0.48	1.09	
(a) Only average v	ol% of H <sub>2</sub> O	was obtained						

Table 8.2. Gaseous Emissions Rates for Test Periods 6 through 9



Figure 8.4. Feed Rate vs. NO<sub>x</sub> Concentration in RSM Offgas



Figure 8.5. Feed Rate vs. CO Concentration in RSM Offgas

#### 8.3 Particulate Matter Emission Analysis

For each test period, PM was withdrawn isokinetically from the source gas within the RSM exhaust pipe and collected on a fiber filter maintained at a temperature of  $120 \pm 14^{\circ}$ C ( $248 \pm 25^{\circ}$ F). The mass of PM during each test period was determined gravimetrically after the removal of uncombined water from the material accumulated on the filter. PM emission rates were calculated in milligrams per minute using the percent isokinetics and sample nozzle/exhaust stack ratio information. A schematic of the PM sample train is shown in Figure 8.6. It should be noted that the flow meter used during the test was a PNNL installed device not provided by the offgas sampling contractor.



Figure 8.6. EPA Method 5 Particulate Sample Train Diagram

PM filter materials were analyzed by Antech (Corbett, OR) using ICP-OES for test periods 6 through 9, and the results are shown in Table 8.3. Particulate emissions contained an average of 0.74% of feed solids for the detected elements. The average decontamination factor (DF) during the four test periods was determined to be 134 with an average slurry feed rate of 60.7 g/min (2.57 L/h, lower than the stable processing feed rate of 2.98 L/h). Bismuth, potassium, sulfur, and lead exhibited the highest emission rates of the elements measured.

	Te	est Period 6				Test Period 7					
	09/11/	13 1446 – 1616,			09	09/12/13 0754 - 0934,					
	17.5	5% Moisture,				14.65% Moisture,					
	89%	6 Isokinetics				88% Isokinetics,					
	Feed Solids	Emissions	%		Feed Solids	Emissions	%				
	Rate (mg/min)	Rate (mg/min)	Feed	DF	Rate (mg/min)	Rate (mg/min)	Feed	DF			
Total	13874	99.64	0.72	139	8813	49.82	0.57	177			
Al	2994	ND	NC	NC	1902	ND	NC	NC			
Bi	238	53.75	22.60	4.4	151	15.18	10.05	10.0			
Ca	207	0.19	0.09	1083	131	0.04	0.03	3385			
Cr	225	0.37	0.17	602	143	0.48	0.34	295			
Fe	1099	2.29	0.21	480	698	1.56	0.22	449			
Κ	1232	22.58	1.83	55	783	18.02	2.30	43			
Li	518	1.69	0.33	307	329	1.34	0.41	246			
Mg	13.3	0.36	2.72	37	8.4	0.03	0.33	303			
Mn	296	0.49	0.17	600	188	0.38	0.20	501			
Na	2027	10.78	0.53	188	1288	8.62	0.67	149			
Ni	55	0.11	0.21	478	35	0.07	0.21	473			
Р	51	0.14	0.28	356	32	0.09	0.29	349			
Pb	78	1.68	2.16	46	49	0.66	1.33	75			
S	18	5.16	27.93	3.6	12	3.32	28.31	3.5			
Si	3400	ND	NC	NC	2160	ND	NC	NC			
	Te	est Period 8				Test Period 9					
	09/12/	13 1003 – 1123,			09	/12/13 1155 – 132	0,				
	13.7	6% Moisture,				14.99% Moisture,					
	98%	6 Isokinetics				100% Isokinetics					
	Feed Solids	Emissions	%		Feed Solids	Emissions	%				
	Rate (mg/min)	Rate (mg/min)	Feed	DF	Rate (mg/min)	Rate (mg/min)	Feed	DF			
Total	10334	116.17	1.12	89	10298	61.74	0.60	167			
Al	2230	7.50	0.34	297	2222	ND	NC	NC			
Bi	177	19.83	11.19	8.9	177	19.48	11.04	9.1			
Ca	154	0.05	0.03	3155	153	0.06	0.04	2769			
Cr	167	0.69	0.41	241	167	0.56	0.34	296			
Fe	819	3.49	0.43	235	816	1.53	0.19	533			
K	918	25.31	2.76	36	914	22.49	2.46	41			
Li	386	2.21	0.57	175	384	1.59	0.41	242			
Mg	9.9	1.88	18.99	5	9.8	ND	NC	NC			
Mn	220	0.79	0.36	278	219	0.38	0.17	581			
Na	1510	13.74	0.91	110	1505	10.31	0.69	146			
Ni	41	0.15	0.37	268	41	0.09	0.21	466			
Р	38	0.18	0.48	208	38	0.07	0.17	574			
Dh	50	0.92	1.58	63	58	0.96	1.66	60			
FU	30	0.72						-			
S	38 14	5.14	37.33	2.7	14	4.23	30.86	3.2			
S Si	14 2532	5.14 24.28	37.33 0.96	2.7 104	14 2523	4.23 ND	30.86 NC	3.2 NC			

Table 8.3. RSM PM Emissions Summary

Comparison of PM and offgas line deposit samples are shown in Table 8.4. As shown in Figure 4.6, solid deposits from the melter were collected from the offgas line. The three analyzed samples showed similar chemical compositions (Table 4.2). The averaged composition was calculated and compared with the composition of filter PM samples. It should be noted that small amounts of Cl and F are not included in the comparison.

The average PM emission rate during test periods 6 through 9 was used to estimate the total PM emissions generated during the entire RSM test. The average feed rate of the four selected PM test periods was 60.7 g/min (2.57 L/h) which is lower than the stable processing feed rate is 70.4 g/min (2.98 L/h, Table 4.1). Considering the large variation of the feed rate over the entire run, this difference is fairly small; and the PM emission rate averaged in a short period can be used to compare with other data averaged based on the entire run (e.g. averaged feed rate, glass pour rate, and EVS emission rate) for the mass balance calculation.

There are some components that were not included in the analysis, such as  $Bi_2O_3$  for offgas line deposits and  $B_2O_3$  for PM, which caused the sum of the concentrations to be less than 100 mass%.  $Al_2O_3$ ,  $Na_2O_3$ ,  $K_2O_3$ , and  $SiO_2$  are other major components were analyzed in the samples. PM samples were collected by filters down-stream which should be finer particles. The composition of PM samples is high in  $K_2O$  and  $SO_3$ . On the other hand, offgas line deposits are larger particles from the melter, which contain higher  $Al_2O_2$ , and  $Na_2O$ . The total mass of samples are small fractions of the total feed processed. The total mass of PM was calculated by measured mass emission rate during sample periods and overall feed rate; while the total mass of the offgas line deposits was measured from the collected samples of the whole run. The total emission from the melter, including offgas line deposits, PM, and EVS solution will be discussed in Section 9.0.

Compound mass <sup>0</sup> /		Offgas Line D	eposits		DM
Compound, mass%	Film Cooler	Long Tube	90° Bend	Average	PM
Al <sub>2</sub> O <sub>3</sub>	13.61	11.27	8.89	11.20	4.59
$B_2O_3$	18.50	13.20	10.00	13.75	NM
Bi <sub>2</sub> O <sub>3</sub>	NM	NM	NM	NM	14.43
CaO	0.71	0.82	0.79	0.78	0.06
Cr <sub>2</sub> O <sub>3</sub>	1.80	1.49	1.46	1.57	0.37
Fe <sub>2</sub> O <sub>3</sub>	5.44	4.58	4.33	4.75	1.49
K <sub>2</sub> O	7.33	6.48	6.45	6.72	12.63
Li <sub>2</sub> O	1.90	1.41	1.36	1.54	1.73
MgO	0.06	0.04	0.04	0.05	0.52
MnO	1.42	0.91	0.88	1.05	0.31
Na <sub>2</sub> O	11.06	9.14	8.05	9.36	6.92
NiO	NM	NM	NM	NM	0.06
P <sub>2</sub> O <sub>5</sub>	0.38	0.93	0.87	0.75	0.13
PbO	NM	NM	NM	0.00	0.54
SO <sub>3</sub>	0.34	ND	0.28	0.19	5.28
SiO <sub>2</sub>	21.35	17.82	14.75	17.88	16.84
SrO	NM	NM	NM	NM	0.02
TiO <sub>2</sub>	NM	NM	NM	NM	0.16
$ZrO_2$	0.05	0.24	0.32	0.21	0.02
Total mass% <sup>(a)</sup>	83.97	68.33	58.44	69.80	66.10
Total sample weight, g	56.55	72.29	61.12	189.96	74.13
(a) Concentrations sum to	ess than 100% b	because some e	lements coul	d not be ana	lyzed.
ND = not detected; NM = not	ot measured.				

Table 8.4. Comparison of PM and Offgas Line Deposits

### 9.0 Process Mass Balance

During the RSM operation, offgas streams including deposits in the exhaust line, PM emission, and scrubber (EVS) solution were characterized for completeness. The compositional data of process streams previously discussed and summarized were combined.

Total water mass in the feed was calculated and compared with the mass of water accumulated in the EVS tank (Table 9.1). It should be noted that there are two reference values of the water mass% in the slurry feed (target and analyzed). The analyzed water content in the feed sample is ~7% higher than the target water content of the feed formulation, which should be caused by experimental error. As expected, the water mass% calculated by EVS vs. feed basically matches the reference values, which indicates that the EVS solution accumulated all water- and water-soluble components in the offgas stream (expect the offgas line deposits).

Feed processed, kg	388.01
EVS solution accumulated in tank, kg	228.85
Water mass% recovered by EVS <sup>(a)</sup>	59.0
<i>Reference water mass%, Target</i> <sup>(b)</sup>	55.9
Reference water mass%, Analyzed feed sample <sup>(c)</sup>	60.1
(a) Calculated by EVS solution mass (ignore mass of soluble comp	ponent in the solution)

#### Table 9.1. Water Mass Calculation

divided by feed mass.

(b) Calculated by feed formulation, including water from hydroxides.

(c) Calculated by an analyzed feed sample (sample ID, RSM-EWG8-041,

(d) Table 3.2), by subtracting total dry mass of analyzed components from total mass of the slurry feed sample

Italics are reference water mass%.

The mass balance is measured by comparing input and output of the RSM. The results for the overall RSM run are summarized in Table 9.2. For each element, the mass from the slurry feed was the input, and the output mass included the poured glass, the offgas line deposit, and EVS solution. For the feed and glass samples, analytical compositional data from SwRI were used whenever possible; otherwise, PNNL data were used.

PM samples were not counted in the output mass. The PM sampling process was performed for a total of 355 min (Table 8.1), and the sampling stream was  $\sim 2\%$  of the total offgas stream (PM filter run fraction was  $\sim 2\%$ , calculated based on the ratio of sampling nozzle area and the main line area), which means most emission from the melter went to the offgas line deposit and the EVS. Thus, the mass of PM samples was negligible for the overall mass balance. However, the PM results are used to estimate the element emissions in terms of emission rate later in this section.

The volatile  $NO_x$ , CO, and CO<sub>2</sub> were directly measured by continuous gas monitors as described in Section 8.2; they were not included in the mass balance calculation because of that 100% of the output was in the gas emission.

The recovery of elements throughout the RSM system was calculated for the targeted components as elements. There are some elements measured in the feed and glass samples as impurities (not in the target formulation). Some of those impurities, Mg, S, Sr, Ti, Zn, and Zr, are included in the mass balance calculation.

Overall, the tracked elements show good recovery as shown in Table 9.2. Major components such as Al, B, Na, and K had about 90% to 110% recoveries. The recoveries of Fe and Si were 84% and 135% respectively, which is likely due to experimental error. Fluorine was lower than the detection limit in the produced glass samples; however, the recovery was estimated by the ICP-OES detection limit. The estimated F recovery was below 65.2%; this low mass balance closure is related to the larger relative errors for the components with lower concentration. It should be noted that some components were not analyzed in collected samples, such as Bi in offgas deposits, which led to lower recovery percentages.

Furthermore, the feed rate and emission rate of the elements were calculated by the masses and feeding time, and the proportion of emission were estimated (Table 9.3). F and S show over 10% total emission. Considering that F was not analyzed in PM, its total emission percentage might be underestimated. The percentage of volatile F and S can be used to compare with other melter studies. The total emission of elements from the melter is 0.39%.

The PM filter was effective in collecting solids from the emission, which can be approximately used as the total deposits of the offgas system. As shown in Table 9.3, the total offgas line deposits were calculated by subtracting the EVS emission rate from the PM emission rate. As expected, most of the analyzed components show that the calculated masses exceed the masses recovered from the three offgas line deposit samples (film cooler, the 90° bend, and the long tube) except for Ca, which was likely caused by experimental error. Comparing the calculated and measured total offgas line deposits rates in mg/min, the source of differences is deposits in the offgas system outside of the three sampling locations (Figure 4.6). The value of Mg (an impurity) is very low and is likely due to experiment error in the chemical analyses.

In PM samples, a significant amount of Bi, 12.59% of total Bi from feed, was found; however, Bi was not measured in the offgas line deposits. This shows Bi emission might be under-estimated by the analytical result of EVS solution. Bi was found in filtered solid samples of EVS solution (data not reported, because sample mass could not be determined precisely).

In summary, the melter emission could not be 100% recovered by offgas line deposit samples and EVS samples; the unrecovered proportion was investigated by comparing with PM results. Even though it is more effective, PM still has downsides. First, the film cooler and 90° bend deposits could not be collected by PM filter. Also, the PM sampling periods were intermittent, which was affected by the changes of feeding rate and bubbling rate. The comparison results of different emission samples provide important information of undetected emissions from (1) offgas line deposits that were hard to collect and (2) probable precipitates not collected in the bottom of the EVS tank.

Element	Feed <sup>(a)</sup> ,	388.01 kg	Offgas Li 0.1	ine deposit <sup>(b)</sup> , 190 kg	EVS Sol 228.8	ution <sup>(c)</sup> , 5 kg	Glass <sup>(d)</sup> ,	140.81 kg	% of mass accounted for
	mg/kg	mass, kg	mg/kg	mass, kg	mg/kg	mass, kg	mg/kg	mass, kg	
Al	38400	14.900	59272	0.01126	0.41	0.00009	100350	14.130	94.9%
В	18100	7.023	42697	0.00811	221.50	0.05069	44300	6.238	89.7%
Bi	3050	1.183	_	-	85.15	0.01949	5315	0.748	64.9%
Ca	2650	1.028	5561	0.00106	4.88	0.00112	6815	0.960	93.5%
Cr	2880	1.117	10763	0.00204	2.98	0.00068	11150	1.570	140.7%
F <sup>(e)</sup>	157	0.061	5227	0.00099	68.30	0.01563	< 164	< 0.023	< 65.2%
Fe	14100	5.471	33235	0.00631	10.00	0.00229	32650	4.597	84.2%
Κ	15800	6.131	55795	0.01060	181.50	0.04154	47450	6.681	109.8%
Li	6640	2.576	7171	0.00136	28.35	0.00649	17600	2.478	96.5%
$Mg^{(f)}$	170	0.066	272	0.00005	3.15	0.00072	143	0.020	31.7%
Mn	3790	1.471	8139	0.00155	2.85	0.00065	9250	1.302	88.7%
Na	26000	10.088	69432	0.01319	161.50	0.03696	69500	9.786	97.5%
Ni	703	0.273	-	-	1.23	0.00028	2055	0.289	106.2%
Р	656	0.255	3266	0.00062	_	_	1190	0.168	66.1%
Pb	998	0.387	-	-	2.84	0.00065	2155	0.303	78.5%
S <sup>(f)</sup>	237	0.092	773	0.00015	63.40	0.01451	245	0.034	53.5%
Si	43600	16.917	83591	0.01588	13.90	0.00318	162500	22.882	135.4%
Sr <sup>(f)</sup>	4.87	0.002	_	_	-	_	8	0.001	58.1%
Ti <sup>(f)</sup>	69.1	0.027	_	_			162	0.023	84.8%
W	212	0.082	_	_	0.95	0.00022	741	0.104	127.1%
Zn <sup>(f)</sup>	15.6	0.006	-	-	-	-	42	0.006	98.5%
Zr <sup>(f)</sup>	16.8	0.007	_	_	_	_	84	0.012	181.2%
Total	NA	69.163	NA	0.072	NA	0.1952	NA	72.335	105.0%

Table 9.2. Mass Balance Calculation

(a) Feed Solids analysis from RSM-EWG8-041 (

(b) Table 3.2).

(c) Offgas line deposits analysis by XRF and ICP-OES (Table 4.2).

(d) EVS Solution from analysis of sample RSM-EWG8-058 except Al, which was averaged by four samples (Table 7.1).

(e) Glass composition from average of ICP-OES analysis of two samples, RSM-EWG8-018 and RSM-EWG8-035

(Table 6.2).(f) Used detection limit to estimate the maximum concentrating in glass.

(g) Impurities.

"-" = not measured or not detected; NA = not applicable.

Element	Feed	Feed Offgas Line Deposits, measured		EV	EVS		SUM		PM		Offgas Line Deposits, calculated <sup>(a)</sup>		% measured vs calculated offs-gas line	% recovered
	mg/min	mg/min	% feed	mg/min	%feed	mg/min	% feed		mg/min	% feed	mg/min	% feed	deposits <sup>(b)</sup>	emission
Al	2703	2.04	0.08	0.017	0.001	2.06	0.08		7.50	0.28	7.48	0.28	27.3%	27.5%
В	1274	1.47	0.12	9.19	0.72	10.66	0.84		-	-	_	_	_	_
Bi	215	-	_	3.53	1.65	3.53	1.65		27.04	12.59	23.50	10.95	-	13.1%
Ca	187	0.19	0.10	0.20	0.11	0.39	0.21		0.08	0.04	_	_	_	_
Cr	203	0.37	0.18	0.12	0.06	0.49	0.24		0.52	0.26	0.40	0.20	93.1%	94.8%
F	11	0.18	1.63	2.83	25.65	2.83	27.28		_	-	_	_	_	_
Fe	992	1.14	0.12	0.42	0.04	1.56	0.16		2.17	0.22	1.76	0.18	65.2%	71.8%
K	1112	1.92	0.17	7.53	0.68	9.46	0.85		21.89	1.97	14.36	1.29	13.4%	43.2%
Li	467	0.25	0.05	1.18	0.25	1.42	0.30		1.68	0.36	0.51	0.11	48.9%	84.6%
Mg	12	0.01	0.08	0.13	1.09	0.14	1.17		0.69	5.73	0.56	4.64	1.7%	20.4%
Mn	267	0.28	0.11	0.12	0.04	0.40	0.15		0.50	0.19	0.38	0.14	73.5%	79.8%
Na	1830	2.39	0.13	6.70	0.37	9.09	0.50		10.73	0.59	4.02	0.22	59.5%	84.8%
Ni	49	_	-	0.05	0.10	0.05	0.10		0.11	0.21	0.05	0.11	_	48.4%
Р	46	0.11	0.24	_	_	0.11	0.24		0.12	0.26	_	_	_	93.9%
Pb	70	-	_	0.12	0.17	0.12	0.17		1.05	1.49	0.93	1.33	_	11.2%
S	17	0.03	0.16	2.63	15.77	2.66	15.93		4.42	26.47	1.78	10.70	1.5%	60.2%
Si	3069	2.88	0.09	0.58	0.02	3.46	0.11		24.28	0.79	23.71	0.77	12.1%	14.2%
W	15	_	_	0.04	0.26	0.04	0.26		_	_	_	_	_	_
Total	12547	13.09	0.10	35.40	0.28	48.487	0.39		103.13	0.82	79.44	0.63	16.5%	47.0%

Table 9.3. Total Estimated Cation and Anion Emissions (Offgas Line Deposits, PM, and EVS Solution Analyses Combined)

(a) Calculated offgas line deposits by subtracting EVS masses from PM masses.

(b) Measured offgas line deposit mass rates (three collected samples in Table 4.2) divided by calculated mass rates offgas line deposit.

(c) Calculated by total measured masses rates (offgas line deposits and EVS) divided by PM mass rates.

"-" = not measured or not detected.

### 10.0 PNNL RSM and VSL DM100 Comparisons

The HLW-HCr-16 glass was produced in the RSM operated by PNNL (this work) and the DM100 melter operated by the VSL (Matlack et al. 2014). The SY101/102 simulants used by PNNL and VSL were produced by the same supplier from the same batch and were below the target value for sodium (detailed description in Section 3.0). VSL and PNNL made batch adjustments to increase sodium in the melter feed. PNNL made adjustments by adding NaOH pellets. It was not indicated in the corresponding report (Matlack et al. 2014) how the adjustments were made.

Melter testing by VSL included two melter runs totaling 100 h of feed time, 580 kg of glass, and feeding over 1600 kg of melter feed. PNNL melter testing, by comparison, consisted of 92 hours of feed time, 140.8 kg of glass, and feeding 388 kg of melter feed. The glass production average reported by VSL for the DM100 was nearly 100% greater (1100 and 1550 kg/day/m<sup>2</sup> for DM100 and 654 kg/day/m<sup>2</sup> for the RSM). However, the peak steady state production in the RSM was 822. The two runs in the DM100 were distinguished by optimized processing parameters to increase production, namely bubbling rates. In VSL's DM100 test 1, the goal was optimized bubbling to achieve greater production with a bubbling flux of 144 L/min/m<sup>2</sup>, which resulted in a production rate of 1550 kg/day/m<sup>2</sup>. In VSL's DM100 test 2, bubbler air flow was held at a constant value with a bubbling flux of 83 L/min/m<sup>2</sup> and a glass production rate of 1100 kg/day/m<sup>2</sup> (Matlack et al. 2014).

	RSM	DM100 test 1, Optimized Bubbling <sup>(a)</sup>	DM100 test 2, Fixed Bubbling <sup>(b)</sup>
Melt surface area, m <sup>2</sup>	0.05	0.108	0.108
Average glass temperature, °C	1137	1140	1142
Average plenum temperature, °C	598	492	444
Average bubbling rate, L/min	4.2	15.6	9.0
Average bubbling rate, L/min/m <sup>2</sup>	84	144	83
Feeding duration, h	92	50	50
Feed processed, kg	388	982	697
Glass produced, kg	140.8	327.3	253.3
Feed rate, average (steady state), L/h <sup>(c)</sup>	2.98(3.33)	13.8	9.8
Glass production rate, average (steady state), $kg/day/m^{2(d)}$	654(822)	1550	1100
Total DF	134	143	162

Table 10.1. Production Value Comparison, PNNL-RSM and VSL-DM100 (Matlack et al. 2014)

(a) Data for optimized production run with enhanced bubbling.

(b) Data for baseline production run.

(c) Based on feed density of  $1.416 \text{ g/cm}^3$ .

(d) Due to feeding problems, the average glass production rate of PNNL RSM is lower than the steady-state glass production rate.

HLW-HCr-16 VSL glass compositions, shown in Matlack et. al. 2014 (Table 4.7), were comparable with the PNNL glass (Table 6.2 and Table 6.3 of this report) in terms of the major glass-forming components of  $Al_2O_3$ ,  $B_2O_3$ ,  $Na_2O$ , and  $SiO_2$  (Table 10.2). It is noticeable that Bi is much lower in the PNNL glass, and evidence of Bi emission was observed. On the other hand, loss of Bi was not as significant in the VSL glass. Sodium in the PNNL glass was below target and below VSL numbers. This is thought to be

related to undissolved NaOH pellets that were removed from the system when resolving frequent line plugs.

~	· · · · · · · · · · · · · · · · · · ·	,	
Component	Target	PNNL <sup>(a)</sup>	VSL <sup>(0)</sup>
	(mass%)	(mass%)	(mass%)
$Al_2O_3$	19.83	18.955	17.09
$B_2O_3$	15.06	14.27	14.34
Bi <sub>2</sub> O <sub>3</sub>	0.91	0.59	1.00
CaO	0.98	0.955	0.92
Cr <sub>2</sub> O <sub>3</sub>	1.82	1.63	1.42
F	0.06	< 0.016	0.01
Fe <sub>2</sub> O <sub>3</sub>	5.04	4.67	5.88
K <sub>2</sub> O	6.39	5.715	4.95
Li <sub>2</sub> O	4.00	3.79	3.82
MgO	_(c)	0.02	0.15
MnO	1.29	1.195	1.04
Na <sub>2</sub> O	10.69	9.37	10.32
NiO	0.25	0.265	0.41
$P_2O_5$	0.38	0.27	0.46
PbO	0.31	0.23	0.23
SO <sub>3</sub>	_(c)	0.03	0.09
SiO <sub>2</sub>	32.89	34.83	37.12
TiO <sub>2</sub>	_(c)	0.03	0.04
WO <sub>3</sub>	0.09	0.09	0.10
ZnO	_(c)	0.01	0.20
ZrO <sub>2</sub>	_(c)	0.01	0.42
Total	100	96.88	100.00

Table 10.2. Glass Composition Comparison, HLW-HCr-16 Produced in PNNL-RSM and VSL-DM100 (Matlack et al. 2014)

(a) Average of two PNNL RSM samples, RSM-EWG8-018 and RSM-EWG8-035, from Table 6.2.

(b) Averaged of three VSL DM100 samples, MBL-D-39A, MBL-D-78A, and MBL-D-97A, reported in Matlack et al. 2014.
(c) Map. So. Tio. Trop. and Trop. are not tanget constituents.

(c) MgO, SO<sub>3</sub>, TiO<sub>2</sub>, ZnO, and  $ZrO_2$  are not target constituents.

The analytical results from EPA Method 5 sampling were also comparable. VSL reported an overall melter DF of 143 and 162 for tests 1 and 2, respectively. PNNL reported an overall DF of 134 averaged for the four test periods. Notable exceptions were DFs for Al, Ca, Cr, and Si, which were significantly higher in the DM100, and Fe, Mn, and Li, which were significantly lower. The reasons for these differences are unclear.

Gaseous emissions from the melter were also sampled and analyzed by PNNL and VSL, with the results given in Table 8.2 of this report and Table 5.2 of Matlack et. al. 2014, respectively. Notable differences were the higher levels of  $NO_x$  (81.2 vs. 35.5 ppmv) and CO (331.9 vs. 138.1 ppmv) during the PNNL RSM run. This can be explained by the higher plenum temperatures and larger variations in cold cap coverage during the RSM offgas testing period compared to the larger DM100 melter.

## **11.0 Conclusions**

The RSM tests in this study provided glass samples and basic operational data while processing a high-Cr waste simulant feed composition, HLW-HCr-16. It was demonstrated that acceptable processing rates and characteristics could be achieved when the RSM was operated under parameters that were generally aligned with those of larger melters on a melt-surface-area-adjusted basis. The tests demonstrated the process flowsheet through a small-scale integrated process which provided feedback and data and during operations.

Steady-state processing is essential in a vitrification process to maintain glass quality and reduce the challenges to the offgas system from particulate, aerosols, and noxious gasses. During this test, undissolved NaOH pellets frequently plugged the feed injection line to the melter. As would be expected, stopping feed to the melter reduced the durations for collecting steady state data as well as impacting glass chemistry results. As a result, it was difficult to make firm conclusions about the data and make fair comparisons with other melter tests.

This test was carried out in a period of ~103 h; several interruptions caused by plugging of the feeding system were resolved in the first 24 h. The RSM was operated with a target glass temperature of 1150°C and target plenum temperature between 550°C and 700°C. The average glass melt temperature measured in the RSM was 1137°C. Measured plenum temperatures ranged from 515°C to 622°C during steady-state operation and the average surface-area-specific bubbling rate was 84 L/min/m<sup>2</sup>. The average glass production rate was 1.36 kg/h in the RSM, resulting in an overall average melter surface-area-normalized glass generation rate of 654 kg/day/m<sup>2</sup>. During periods of steady-state operation in the RSM, the production rate as calculated from the feed rate was 822 kg/day/m<sup>2</sup>. The average power use for glass production was 109.2 kW/m<sup>2</sup>. Overall, 140.8 kg of glass was produced during operation of the RSM.

One glass (RSM-EWG8-044) was subsampled and isothermally heat treated at different temperatures as well as following the HLW CCC profile. The crystal phases in the quenched and heat-treated samples were analyzed by SEM-EDS and XRD. Some spinel crystals were identified in the quenched glass and also in the heat-treated glasses. The crystal fraction of spinel in the glass increased slightly after heat treatments. Nepheline was also identified in the heat-treated glass. The temperature at 1 vol% spinel determined by the isothermal heat-treatment tests was estimated to be 1786°C. The resulting glass product had TCLP responses below the regulatory limits. At ~1150°C, viscosity and electrical conductivity of the glass were 5.8 Pa-s and 22.3 S/m, respectively. The viscosity was within the acceptable range and the electrical conductivity was close to the DWPF reference glass.

Offgas sample analysis determined the  $H_2O$ ,  $CO_2$ ,  $NO_x$ , and CO concentrations in the emission gas.  $H_2O$  and  $CO_2$  were 13.76 – 17.55 vol% and 0.56 – 0.98 vol% in the emission gas respectively, with the rest being bubbled air.  $NO_x$  and CO, as minor components of interest in the emission, were 68.70 – 94.19 ppmvd and 236.53 – 421.55 ppmvd respectively. The total DF for the RSM test was determined to be 134 by the PM analysis results.

Process mass balance was investigated focusing on the emission of various elements. Bi, F, and S are the three elements that had significant levels of emission during the melter run. Combination of offgas line deposits and EVS solution recovered 27.28% of F and 15.93% of S; while PM method recovered 12.59% of Bi and 26.47% of S. Emission ratios of the elements other than Bi, F, and S were all less than 2% (disregarding data from some impurities, which had low concentration and large uncertainties, such as Mg). Only a portion of the offgas line deposit samples was collected, and the unrecovered deposits were

estimated by PM results. EVS captured majority of alkalis, B, Cr, F, and S, while Si, Al, and Pb are found mainly in the solid deposits in the offgas line.

Comparing the RSM and VSL DM100, RSM is smaller and has a slower glass production rate. The analytical results from offgas systems of the two melters were very similar and the glass samples from the two melter tests were both reasonably close with the target composition, as expected. Improvements can be made to the RSM feeding and bubbling operations.

Overall, this work demonstrates that the smaller-scale RSM can be used to support and supplement waste vitrification research, providing comparable experimental results while requiring less material and shorter turnaround times than the larger melters. In future tests, consideration should be given to validating the prepared feed before commencing operations.

## 12.0 References

10 CFR 830, *Nuclear Safety Management*, Subpart A – Quality Assurance Requirements. Code of Federal Regulations. Accessed September 14, 2015 at

http://www.gpo.gov/fdsys/search/pagedetails.action?collectionCode=CFR&searchPath=Title+10%2FCha pter+III%2FPart+830%2FSubpart+A&granuleId=CFR-2011-title10-vol4-part830&packageId=CFR-2011-title10vol4&oldPath=Title+10%2FChapter+III%2FPart+830%2FSubpart+A&fromPageDetails=true&collapse= false&vcord=1136.

40 CFR 261, Identification and Listing of Hazardous Waste. Code of Federal Regulations.

40 CFR 60, Standards of Performance for New Stationary Sources. Code of Federal Regulations.

ASME NQA-1-2000, *Quality Assurance Requirements for Nuclear Facility Applications*. American Society of Mechanical Engineers, New York, NY.

ASME NQA-1-2012, *Quality Assurance Requirements for Nuclear Facility Applications*. American Society of Mechanical Engineers, New York, NY.

ASTM C 1285-02(2008), Standard Test Methods for Determining Chemical Durability of Nuclear, Hazardous, and Mixed Waste Glasses and Multiphase Glass Ceramics: The Product Consistency Test (PCT). American Society of Testing and Materials, West Conshohocken, PA.

ASTM C965-94(1994), *Standard Practice for Measuring Viscosity of Glass Above the Softening Point*. American Society of Testing and Materials, West Conshohocken, PA.

Barnes SM and DE Larson. 1981. *Materials Design Experience in a Slurry-Fed Electric Glass Melter*. PNL-3959, Pacific Northwest Laboratory, Richland, WA.

Crum JV, TB Edwards, RL Russell, PJ Workman, MJ Schweiger, RF Schumacher, DE Smith, DK Peeler, and JD Vienna. 2012. "DWPF Startup Frit Viscosity Measurement Round Robin Results." *J. Am. Ceram. Soc.* 95(7):2196-2205.

DOE—U.S. Department of Energy. 2000. *Design, Construction, and Commissioning of the Hanford Tank Waste Treatment and Immobilization Plant*. Contract DE-AC27-01RV14136, as amended, U.S. Department of Energy, Office of River Protection, Richland, WA.

DOE—U.S. Department of Energy. 2014. *Hanford Analytical Services Quality Assurance Requirements Document*. DOE/RL-96-68, Rev. 4, Richland Operations Office, Richland, WA.

DOE Order 414.1D, *Quality Assurance*. U.S. Department of Energy, Washington, D.C. Accessed September 14, 2015, at <u>https://www.directives.doe.gov/directives-documents/400-series/0414.1-BOrder-d</u>.

EPA—U.S. Environmental Protection Agency. 1992. *Method 1311: Toxicity Characteristic Leaching Procedure*. Washington, D.C.

EPA—U.S. Environmental Protection Agency. 1994. *Method* 9056: *Determination of Inorganic Anions by Ion Chromatography*. Washington, D.C.

Goles RW and AJ Schmidt. 1992. Evaluation of Liquid-Fed Ceramic Melter Off-Gas System Technologies for the Hanford Waste Vitrification Plant. PNL-8109, Pacific Northwest Laboratory, Richland, WA.

Kim D, MJ Schweiger, WC Buchmiller, and J Matyas. 2011. *Laboratory-Scale Melter for Determination of Melting Rate of Waste Glass Feeds*. PNNL-21005 (EMSP-RPT-012), Pacific Northwest National Laboratory, Richland, WA.

Matlack KS, WK Kot, W Gong, W Lutze, IL Pegg, and I Joseph. 2009. *Effects of High Spinel and Chromium Oxide Crystal Contents on Simulated HLW Vitrification in DM100 Melter Tests*. VSL-09R1520-1, Vitreous State Laboratory, The Catholic University of America, Washington, DC.

Matlack KS, H Gan, M Chaudhuri, WK Kot, IL Pegg, and I Joseph. 2014. *High Waste Loading Glass Formulation Development for High-Cr HLW*. 14R3060-1, Rev. 0, Vitreous State Laboratory, The Catholic University of America, Washington, DC.

Rose PB, DI Woodward, MI Ojovan, NC Hyatt, and WE Lee. 2011. "Crystallization of a simulated borosilicate high-level waste glass produced on a full-scale vitrification line." *J. Non-Cryst. Solids*, 357(15):2989-3001.

Vienna JD, D Kim, DC Skorski, and J Matyas. 2013. *Glass Property Models and Constraints for Estimating the Glass to be Produced at Hanford by Implementing Current Advanced Glass Formulation Efforts*. PNNL-22631, Rev. 1 (ORP-58289), Pacific Northwest National Laboratory, Richland, WA.

# Appendix A – Manual Log (Melter Operating Parameter)

Temperature and Electrical	Opera	tor (initials):	11.1	MU	mul.	DED	DEL	DER	mul	JL,	
Data (HMI)		Date:	09.09.13	9-9-13	9-9-13	9/1/13	9/5/13	9/9/13	9/9/13	9/9/13	
Data (IIII)		Start time:	0529	0629	0736	0828	0938	1036	1136	1242	
DESCRIPTION	UNITS	RANGE									
Melt Temperature (T1)	°C	1125-1175	1148	1121	1115	1126	1120	1121	1129	1131	
Melt Temperature (T2)	°C	1125-1175	1157	1144	1148	1152	1150	1149	1147	1149	
Melt (Electrode) Temp Setpoint	°C	1150	1160	1150	1150	1150	1150	1150	1156	1150	
Secondary Electrode Current	Amps		102.9	102	98.8	98,9	92.4	94.8	98.1	97.4	
Secondary Electrode Potential	Volts		48.1	61	53.2	53.3	44.1	47.5	52.5	48.2	
Secondary Electrode Power (calc)	kW (V×A)		4,95	6,22	5.26	5.27	4.07	4.51	5.15	4,69	
Melt Resistance (calc)	Ω (V/A)		0.468	0.598	0,538	0,53	0.48	0.50	0.54	0.495	1
Electrode Power Output	%		57.9	60.1	55.6	55.8	52.7	53.9	54.5	53.0	1
Electrode Power Control	A or M		Д	A	A	A	A	A	A	iA-	1
Melter Temperature Control		T1 or T2	TZ	TZ	T2	てる	t2	T2	T2_	To	1
Kiln Monitored (Middle) Temp	°C		849	790	743	771	784	776	775	798	1
Kiln Top Temp	°C		811	659	685	714	724	731	726	751	1
Kiln Bottom Temps	°C		697	214	877	930	965	925	933	858	1
Kiln Temp Setpoint	°C		800	SOD	800	800	800	011 .	ALAH 780	780	1
Kiln Power	kW		22610-	1.3	3.9	3.9	3.86	1	3.87	0	1.
Kiln Power Output	%		6.6		93.0	93.0	93.0	V	93.4	17	1
Kiln Control	A or M		A	A	A	A	14		Á	A	1
Discharge Canister Temp	°C	750 - 850	1	1	off	Off	ofe		DCF	off	1
Discharge Canister Temp Setpoint	°C				)	1	1			1	1
Discharge Canister Power	kW		(								1
Discharge Canister Power Output	%		1		1	1	1 I	1	11	V	1
Pourspout Heater Actual Temp	°C	1000-1100	1059	1174	1097	1095	1083	985	1055	1048	1
Pourspout Heater Temp Setpoint	°C		1100	1100	1100	1100	(100	018	1050	1050	1
Pourspout Heater Power	kW		1.67	3.4	2.18	1.77	1.78	1	1.53	1.87	1
Pourspout Heater Power Output	%		362.	53	457	42.5	38.5		.39.2	439	1
Plenum Temperature	°C	500-700	576	665	547	543	402	599	619	\$51	1
eed Nozzle Temp (FNT)	°C	<50	43	34	32	31	31	32	33	39	1
Off-Gas Temp (OGT 1)	°C	<350	374	2,4	269	282	249	244	266	715	1
Off-Gas Temp (OGT #2)	°C		271	145	226	245	208	202	215	186	1
Scrub Liquid Temp (SLT)	°C	<40	27	27	37	34	34	31	9)	28	1
Post EVS Off-Gas Temp	°C	<50	29	360	38	38	39	34	34	7.9	1
leat Xfer Temp	°C	<30	?	1.1	12	29	24	25	25	24	1
Post HEME Temp	°C		25	36	31	32	39	30	290	27	1
Subbler Flow Rate (total)	sccm		4200	4200	42.09	4202	4201	4197	4203	4700	1
Plenum Vacuum	in. H <sub>2</sub> O	0.1 - 2.5	1.3	H	1.0	1.0	0.9	04	1.0	3	54
		Finish time:	0571	OFUL	0.200	0034	ОСИ	10.44	1111	1240	ſ

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Date: 10-21-13

Tanan I at a s	Oper	ator (initials)	1 - 21	1 1 1 1 1 1 1	1	THE GHC	To Chemoto	Por or ONTO	ye.	
Temperature and Electrical	Open	ator (Initials)	alatio	mer	PPS	PPS	PPS #	PPS	PPS	115
Data (HMI)		Start time	12000	7/9/13	9/9/13	9/9/13	9/9/13	9/9/13	9/9/13	19106
DESCRIPTION	UNITS	RANGE	12173	1/50/	1 16.03	17:04	18:03	22:01	23:02	12:05
Melt Temperature (T1)	°C	1125-1175	1141	1124	1129	1.121	A LONG	I IIII	Sector Sector	
Melt Temperature (T2)	°C	1125-1175	1150	my hogg	1141	1131	1139	1147	1124	1075
Melt (Electrode) Temp Setpoint	°C	1150	1100	1.48	1148	1152	1144	1150	1150	1106
Secondary Electrode Current	Amps	1150	907	115	1130	1150	1150	1150	1150	1150
Secondary Electrode Potential	Volts		10.1	JI.L	103.7	102.7	110.7	111.6	106.6	98
Secondary Electrode Power (calc)	kW (V×A)		4119	50.7	51.5	50.3	52.9	51.6	51.9	49
Melt Resistance (calc)	0 (1//4)		7.47	9.86	2.35	5.17	5.86	5.76	5.53	4-31
Electrode Power Output	0/0		C.TT	0.52	0.496	0.49	0.48	0.46	0.49	0,55
Electrode Power Control	AorM		5505	54.5	58.5	58.3	62.5	62.6	60.0	49.
Melter Temperature Control	AUTH	T1 or T2	14	H	A	A	A	A	A	A
Kiln Monitored (Middle) Temp	00	110/12	12	12	T2	72	+2	T2	T2	TZ
Kiln Top Temp	°C		851	922	805	756	727	787	759	760
Kiln Bottom Temps	°C		801	819	769	711	679	747	714	715
Kiln Temp Setpoint	°C		856	830	812	787	770	788	772	756
Kiln Power	LAN/		799.9	854	805	756	727	787	759	760
Kiln Power Output	<u> </u>		. 0	0	0	0	0	0	0	0
Kiln Control	. 90		6	0	0	0	0	0	0	0
Discharge Canister Temp		750 050	H	DH	ott	off	off	off	off	M
Discharge Canister Temp Setpoint	90	/50 - 850	ap	OFF	ott	off	off	otf	off	1
Discharge Canister Power	Lan I			1.	1	1	1		1	1
Discharge Canister Power Output	KVV				'N/A	MA	N/A	N/A	N/A	
Pourspout Heater Actual Tomp	%	1000 1100	1	1		/	1			1
Pourspout Heater Tomp Sotnoint	<u> </u>	1000-1100	1050	1050	1053	1050	1050	1049	1051	1040
Pourspout Heater Power	50		1050	1050	1050	1050	1050	1050	1050	1000
Pourspout Heater Power	KW		1.97	1.97	2.12	2.22	2,36	2.06	2.10	2 78
Denum Temporaturo	%		44.9	45.0	46.5	47.8	49.0	44.0	464	46.7
eed Nozzla Tomp (ENT)	30	500-700	955	825	682	612	560	727	679	(AI
Off-Gas Tomp (OCT 1)	<u></u>	<50	48	40	35	33	32	38	36	7.1
Off-Gas Temp (OGT_#2)	<u> </u>	<350	147	350	326	315	276	331	330	194
Scrub Liquid Tomp (SLT)	00		117	249	262	253	248	266	257	7.51
Post EVS Off-Cas Tomp	00	<40	25	27	31	33	33	29	31	30
leat Xfer Temp		<50	26	30	35	37	37	32	34	34
Post HEME Temp		<30	23	24	28	25	25	24	25	15
Subbler Flow Pate (total)	°C		26	26	25	31	31	25	27	110
lenum Vacuum	sccm	0.1.0.7	4201	4202	4204	4193	4195	4200	4197	4.7.11
	In. H <sub>2</sub> O	0.1 - 2.5	-3	1.0	1.1	1.0	1.0	1.0	11	00
		Finish time:	1350	1514	16:08	17:07	18:08	22:04	22:05	1111

Date: \_\_\_\_\_\_3

Temperature and Electrical	Opera	ator (initials):	110	ar	115	CDL	mile	7-	SL	mul
Data (HMI)		Date:	9/10/13	9/ch	91013	9 Acm	9/10/17	2 9Moto	9/10/13	9/10/13
		Start time:	0112	02:03	0310	04:17	0720	6915	1011	1112
DESCRIPTION	UNITS	RANGE			And South	Statutes and	and the second	1 112	1VIA	11113
Melt Temperature (T1)	°C	1125-1175	1054 ×	1123	1124	1/28	1090	114.2	1154	1117
Melt Temperature (T2)	°C	1125-1175	1076	1147	1138	11100	1281	1153	1143	1151
Melt (Electrode) Temp Setpoint	°C	1150	1150	1150	1150	1150	1150	1152	1150	1150
Secondary Electrode Current	Amps		96.3	1023	109	90.5	969	102.6	90,	62.0
Secondary Electrode Potential	Volts		57.3	53 4	53	471	573	624	5/2	710
Secondary Electrode Power (calc)	kW (V×A)		5.52	5.516	5178	4.26	5.55	5.42	511	59.9
Melt Resistance (calc)	Ω (V/A)		0-595	6.517	0.486	0.520	12,591	0.506	0.515	2.01
Electrode Power Output	%		54.0	587	600	60.9	541	523	513	01959
Electrode Power Control	A or M		A	A	A	A	A	54.0	262 A	35.6
Melter Temperature Control		T1 or T2	T7.	177	72	10	To	177	4	A
Kiln Monitored (Middle) Temp	°C		719	807	851	800	847	011	710	12
Kiln Top Temp	°C		671	742	264	717	011	7010	161	135
Kiln Bottom Temps	°C		241	934	677	0711	814	191	131	689
Kiln Temp Setpoint	°C		-	800	800	027	835	744	194	767
Kiln Power	kW		-	101	1.34	800	050	846	160	731
Kiln Power Output	%		(	8.1	55.1	1.63	1.91	0	0	0
Kiln Control	A or M		M	a		00.9	6310	0	0	0
Discharge Canister Temp	°C	750 - 850	1	1	- Printe	13	H	1VI	Ma	M
Discharge Canister Temp Setpoint	°C						off	off	off	0/-
Discharge Canister Power	kW		1							100
Discharge Canister Power Output	%									
ourspout Heater Actual Temp	°C	1000-1100	1051	1051	IAUT	105.	1000	67	1000	1
ourspout Heater Temp Setpoint	°C	1000 1100	INCO	1051	1050	1051	1053	1051	1050	1051
ourspout Heater Power	kW		2 73	1020	7.06	1050	1050	1050	1050	1050
ourspout Heater Power Output	%		41	470	HHI	1.89	1.96	1.75	1.96	2.11
lenum Temperature	°C	500-700	642	E Q.	150	44.0	44.5	47.8	44.9	46.7
eed Nozzle Temp (FNT)	°C	<50	21	21	1033	641	275	735	640	561
Off-Gas Temp (OGT_1)	°C	<350	208	200	36	52	35	35	3/	30
ff-Gas Temp (OGT_#2)	°C		220	240	2111	Loy	302	30/	237	228
crub Liquid Temp (SLT)	°C	<40	20	174	596	201	242	243	194	185
ost EVS Off-Gas Temp	°C	<50	24	25	20	36	29	33	33	34
eat Xfer Temp	°C	< 30	260	31	15	35	32	37	37	38
ost HEME Temp	°C		12	20	27	45	15	75	26	26
ubbler Flow Rate (total)	sccm		N DI DE	112	61	57	24	29	30	30
lenum Vacuum	in. H <sub>2</sub> O	0.1 - 2.5	1.0	4 400	4100	4600	4200	4204	4199	4207
	-2-	Finish time:	0110	07:07	0115	0.3	1.1	.7	# 3'	O. Y
21			* Lus du	0 4,0 1	0363	CALL	0757	9120	1072	1125
eviewed by:	Man		Yo Cal					-	quil.	15
Page No. 4/12

Temperature and Electrical	Opera	ator (initials):	36	52.	34	SL.	51,	3L.	PPS	PPS
Data (HMI)		Date:	110/13	9/10/13	9/10/13	9/10/13	9/10/13	9/10/B	9/10/13	9/10/13
		Start time:	1215	1322	1911	1524	1600	1700	18:01	19:6
DESCRIPTION	UNITS	RANGE	ALL DEFENSE		,	And the second second		State of the second	and the second second	
Melt Temperature (T1)	°C	1125-1175	1/24	1107	1139	1/20	1107	1110	1095	1117
Melt Temperature (T2)	°C	1125-1175	1155	1152	1151	1147	1150	1149	1149	1158
Melt (Electrode) Temp Setpoint	°C	1150	1150	1150	1150	1152	1150	1150	1150	1160
Secondary Electrode Current	Amps		92,0	96,1	1022	99,8	99,2	95.5	95.1	103.3
Secondary Electrode Potential	Volts		51,3	48.1	53,1	52.2	54.2	57.7	51.6	53.9
Secondary Electrode Power (calc)	kW (V×A)		4-72	4.62	5.42	5.21	5,38	5.03	4.91	5.57
Melt Resistance (calc)	Ω (V/A)		0.558	0, 5005	0,51%	0 523	0,546	0.552	0.54.3	0.52
Electrode Power Output	%		52,2	54,4	57.8	56.8	56-1	541	54.6	58.7
Electrode Power Control	A or M		A	A	A	A	A	A	A	A
Melter Temperature Control		T1 or T2	TZ	TZ	TZ	TZ	TL	12	T2.	TA
Kiln Monitored (Middle) Temp	°C		709	695	707	704	710	710	703	696
Kiln Top Temp	°C		664	649	663	659	663	664	655	648
Kiln Bottom Temps	°C		752	742	741	747	749	745	778	73
Kiln Temp Setpoint	°C		709	694	707	723	709	710	703	696
Kiln Power	kW		0	0	0	0	0	0	0	0
Kiln Power Output	%		6	0	0	0	0	3	0	0
Kiln Control	A or M		M	M	m	M	M	W	M	N
Discharge Canister Temp	°C	750 - 850	A	aff_	off	At	off	ER		- CC
Discharge Canister Temp Setpoint	- °C		1		1	1		011	0++	OFF
Discharge Canister Power	kW							-/	1	
Discharge Canister Power Output	%	1		/	V	1 V	V	N		
Pourspout Heater Actual Temp	°C	1000-1100	1049	1060	Inci	10199	1051	1001	1051	1000
Pourspout Heater Temp Setpoint	°C		1050	1050	1050	1050	1252	1051	1051	1055
Pourspout Heater Power	kW		2.37	7.31	777	777	746	1000	1030	220
Pourspout Heater Power Output	%		48.9	485	422	57.5	601	460	194	477
Plenum Temperature	°C	500-700	574	129	570	515	201	aut	41.7	77.F
Feed Nozzle Temp (FNT)	°C	<50	19	31	30	30	20	20	330	597
Off-Gas Temp (OGT 1)	°C	<350	755	190	739	731	239	20	30	30
Off-Gas Temp (OGT #2)	°C		714	115	201	199	108	201	297	228
Scrub Liquid Temp (SLT)	°C	<40	33	37	37	31	37	27	Lac	228
Post EVS Off-Gas Temp	°C	<50	3.7	33	31	36	36	27	55	33
Heat Xfer Temp	°C	<30	5-20213	76	75	26	11	2/	ST	ST
Post HEME Temp	°C		30	30	30	31	40	21	26	10
Bubbler Flow Rate (total)	sccm		4196	4100	4199	4202	1100	lim	51	JI
Plenum Vacuum	in. H <sub>2</sub> O	0.1 - 2.5	419 Mg	19 5	2	1200	4/17	Im	4176	4204
The start of the s		Finish time:	1219	1370	1410	107-7	1102	1607	10	0.6
			1 haven 1	10/3	11111	17/1	Incis	1001	18 05	19.06

Date: \_\_\_\_\_\_7-1/-13

Temperature and Electrical	Opera	ator (initials)	PPS	1115	NON	11/31	0	111	1/0	1.00.1
Data (HMT)		Date	9/10/13	GIDIA	0/11/17	011112	00	12	NUN -	mul
		Start time:	20:02	1315	60:40	0177	CRIVIS	4/011	1 9/1VIS	19/11/1
DESCRIPTION	UNITS	RANGE		10/12	100.10	10000	102.10	LOSOU	ICID:05	107 2-
Melt Temperature (T1)	°C	1125-1175	1115	1119	110.77	1002	10.90	1.10	WEAR LAND	
Melt Temperature (T2)	°C	1125-1175	1161	1152	1107	1127	10-29	1115	1117	1133
Melt (Electrode) Temp Setpoint	°C	1150	1160	1150	1100	1150	1179	1197	1150	1156
Secondary Electrode Current	Amps		98.9	110.3	1128	074	1150	1150	1150	160
Secondary Electrode Potential	Volts		517	444	010	9100	44.6	100.7	100.9	107.7
Secondary Electrode Power (calc)	kW (V×A)		5.11	11 99	1 2 721	56.1	1100	56.2	76.9	49.8
Melt Resistance (calc)	$\Omega$ (V/A)		0.573	0.497	1212	3,00	7.80	3,26	4.71	5136
Electrode Power Output	%		567	51.3	101	0.934	0.484	0.518	0.467	0,462
Electrode Power Control	A or M		A	1	121.7	55,0	228	26.0	264	57.5
Melter Temperature Control	201	T1 or T2	T2		1-13-	A	A	4	A	A
Kiln Monitored (Middle) Temp	°C	120112	693	775	1705	12	72	42	72	Ta
Kiln Top Temp	°C		142 0	150	102	686	695	718	1079	675
Kiln Bottom Temps	°C		777	690	1020	631	1044	672	130	627
Kiln Temp Setpoint	°C		TST	164	143	720	134	745	30	724
Kiln Power	kW		615	-			-	-	+	
Kiln Power Output	%		0	0		0	-	0		-
Kiln Control	AorM		M	0	~	0	-	0	-	
Discharge Canister Temp	°C	750 - 850	00	M	m	M	M	pa	P	100
Discharge Canister Temp Setpoint	°C	750-050	off			1		)	-	al.
Discharge Canister Power	kW							1	and the second	
Discharge Canister Power Output	0/0		1						-	5.00 ·
ourspout Heater Actual Temp	°C	1000-1100	1-10		1			(	-	
ourspout Heater Temp Setpoint	°C	1000-1100	1048	105	1050	1050	1046	1030	10SC	1051
ourspout Heater Power	kw/		1050	1050	1050	1050	1050	1050	1050	1000
ourspout Heater Power Output	0/0		2,50	2.46	240	2.45	307	3.40	147	2 77
lenum Temperature	°C	500 700	50.8	49.6	57.3	49.7	50,3	62.2	49.6	48.0
eed Nozzle Temp (FNT)	°C	500-700	248	6 34	512	519	1028	500	yn	574
ff-Gas Temp (OGT 1)	°C	<30	30	31	28	28	2/01	28	18	28
ff-Gas Temp (OGT #2)	°C	<350	246	271	232	232	2743	230	202	254
crub Liquid Temp (SLT)	°C	110	232	246	219	220 +	2425	219	200	732
ost EVS Off-Gas Temp	°C	<40	33	32	33	32	31	33	33	22 0
eat Xfer Temp	°C	< 50	37	56	36	36	34	38	310	36
ost HEME Temp	°C	<30	2.6	26	26	25	29	26	25	25
ubbler Flow Rate (total)	Com		32	30	30	28	21	38	-3	20
enum Vacuum	in H o	01 25	4197	4201	4200	4203	4702	4201	4100	4202
the second states and the second states and	III. H <sub>2</sub> U	0.1 - 2.5	0	0.1	6.9	1.0	1.0	1.0	100	100
	ł	inish time:	20:06	2318	GO'SU	0711 0	12.12	O STALL F	Naila	1.0

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Page No. <u>6/12</u>

Temperature and Electrical	Oper	ator (initials)	: MUL	36	Th	1-31	131	101	10	1 via ma
Data (HMI)		Date	: alulis	9/11/13	911113	allit	3 aluti	3 alin	alatura	24
		Start time	108 24	0931	1330	114341	1 19 43	1746	3 JULP	9/1/13
DESCRIPTION	UNITS	RANGE		A Long Long	1105	1101	1015	13-13	1751	1526
Melt Temperature (T1)	°C	1125-1175	1129	1124	1128	1135	1 1117	1 1107	1.1.01	
Melt Temperature (T2)	°C	1125-1175	1162	1158	1169	lika	1150	1176	1146	1131
Melt (Electrode) Temp Setpoint	°C	1150	1160	1110	110	1110	111	1161	1156	1164
Secondary Electrode Current	Amps		102.7	1066	IN G	1112	116	1/62	1160	1160
Secondary Electrode Potential	Volts		49.9	5361	6413	1111	101.8	110.0	110.2	99.9
Secondary Electrode Power (calc)	kW (V×A)		5 120	5 697	5 0001	5100	ZUIF	501	53,1	49.5
Melt Resistance (calc)	Ω (V/A)		486	5009	2.85	2.695	5111	5611	5,851	4.945
Electrode Power Output	%		5711	103	1200	0460	1466	0455	0.982	0,495
Electrode Power Control	A or M		21.9	60.5	617	64.8	628	61.9	62,4	35.9
Melter Temperature Control		T1 or T2	01	T	H	A	IT	14	A	A
Kiln Monitored (Middle) Temp	°C	110112	12	12	12	TZ	124	72	TI	T2
Kiln Top Temp	°C		641	681	693	710	111	723	721	918
Kiln Bottom Temps	°C		640	651	644	660	674	675	675	662
Kiln Temp Setpoint	°C		128	119	730	735	746	749	755	751
Kiln Power	LW/		01+	oft	SFF	off	OF	off	aff	off
Kiln Power Output	0/6		0	0	0	Ó	9	2	0	0
Kiln Control	A or M		0	9	0	0	0	0	0	D
Discharge Canister Temp	A 01 PT	750 050	m	M	m	M	M	m	M	M
Discharge Canister Temp Setpoint	00	750 - 850	N/A	NA	NA.	MA	NA	MIA	NIA	IN
Discharge Canister Power	L'IN		-	-	-	1	-	1		
Discharge Canister Power Output	04		-	-	-	-	~	-	-	
Pourspout Heater Actual Temp	90	1000 4400		-	-	-	-	-	-	2
Pourspout Heater Temp Setpoint	- °C	1000-1100	1052	1050	1051	1051	1252	1050	1051	1000
Pourspout Heater Power	Lan I		1050	1050	1050	1050	120	1050	1001	1050
Pourspout Heater Power Output	KVV		2.34	2.35	2.38	1.37	7.36	775	1030	2 30
Plenum Temperature	%		48.5	48.7	49,1	49.1	419.1	487	490	5.72
Feed Nozzle Temp (ENT)	<u></u>	500-700	57.3	557	609	670%	678	125	18:7	9817
Off-Gas Temp (OCT 1)	<u> </u>	<50	29	30	30	31	33	5453Z-	201	575
Off-Gas Temp (OGT_#2)	<u> </u>	<350	242	249	160	7.66	1893	107	7.6	30
Scrub Liquid Tomp (SLT)	°C		221	730	731	730	165	710	160	249
Post EVS Off-Cos Toma	°C	<40	33	33	33	33	31	27	ho-holen	230
leat Xfer Tomp	°C	<50	36	37	37	36	26	26	34	34
Post HEME Tomp	°C	<30	2.5	25	25	25	70	21	38	38
Bubbler Flow Date (total)	°C		28	29	30	30	30	10	66	30
Penum Vacuum	sccm		4201	4198	4207	4101	Unit	1	37	35
	In. H <sub>2</sub> O	0.1 - 2.5	1.0	1.1	a	a	1001	4207	4202	\$200
06 Elas		inish time:	\$33 .	09341	1227	11/36	1021	1.0	1.0	1.0
AG HIOM	,		-	-/-/1	DEL	7/11	125	1398	1436	1531.
eviewed by:				~	-	Hilb	22,08	20.42	19.19	2/13

	1 6		1 1000	0	00	3	00	(50)	0	0
Temperature and Electrical	Ope	rator (initials)	DA	KY	H	19,	FF	HA I	N N	10
Data (HMI)		Date	9-11	9-11	9-11	9/11	9/11	94	911	qui
DESCRIPTION	LINITTO	Start time	1610	1725	(904	1859	2007	2109	220	1300
Melt Temperature (T1)	UNITS	RANGE	1121	1151	1	Alla martine		1	Ren Lines	
Melt Temperature (T2)	00	1125-1175	1/36	1106	((0)	1134	1129	1(32	1130	1134
Melt (Electrode) Temp Satpoint	0°C	1125-11/5	1150	(134	(122	1161	1162	1160	1152	1163
Secondary Electrode Current	L .	1150	1/50	1140	1130	1160	160	1160	1160	1160
Secondary Electrode Corrent	Amps		112	105	101.3	[01.]	99.5	103.9	113.5	107.3
Secondary Electrode Potential	VOITS		5413	63.6	56.0	51.2	50.4	50.0	53.4	51.0
Melt Resistance (calc)	KW (VXA)	)	6108	5.63	5167	5,18	5.01	5.20	6.06	5.50
Electrode Power Output	<u>S2 (V/A)</u>		0,485	0.510	0,55	0.51	0.51	0,48	0.47	Dout
Electrode Power Control	%		63.4	59,2	57.6	57.8	56-5	58.8	63.8	60.10
Melter Temperature Control	A or M	-	A	A	A	A	A	A	A	A
Kiln Monitorod (Middle) Terre	A State State State	T1 or T2	72	12	72	T2	Tr	12	Th.	12
Kiln Ton Temp	<u> </u>		705	696	692	685	694	701	710	717
Kiln Bottom Tompo	00		656	648	643	636	645	656	112	117
Kiln Temp Setpoint	°C		748	735	732	728	733	735	ZUV	7419
Kiln Power	<u> </u>		öft	16ff	OFF	OFF	DFF	OFF	OFF	OFE
Kiln Power Output	kW		0	0	Ø	0	D	0	0	0
(in Control	%		0	0	0	0	0	0	Ő	0
Discharge Capister Temp	A or M		M	m	14	M	M	NI	N	M
Discharge Canister Temp	<u>°C</u>	750 - 850	off	off	OFF.	OFF	OFF	OFE	INFE	N
Discharge Canister Temp Setpoint	°C		1					PI	M	OFF
Discharge Canister Power	kW							Topp	-	
Pourspout Heater Advert	%		1054	10ff	IDFF	OFF	OFF	1000	DEE	OFF
Pourspout Heater Actual Temp	°C	1000-1100	1052	1053	1051	1050	1050	1054	1053	1 OFF
Ourspout Heater Temp Setpoint	°C	and i	1050	1050	1050	10.50	1057	1000	1050	1059
Ourspout Heater Power	kW	20/3127	483	2.20	2.34	2 29	7,29	2 22	7 02	1050
Consport Heater Power Output	%	1	48,3	48-1	48.8	118.5	LA.F	(18.9	180	2:10
lenum remperature	°C	500-700	569	572	567	537	555	701	40:1	44.2
eed Nozzle Temp (FNT)	°C	<50	30	29	30	30	79	306	244	DDL
Off-Gas Temp (OGT_1)	°C	<350	256	231	224	221	9110	201	4	30
m-Gas Temp (OGI_#2)	°C		245	235	232	226	240	259	245	236
crub Liquid Temp (SLT)	°C	<40	34	33	33	34	22	251	258	22
ost EVS Off-Gas Temp	°C	<50	37	27	27	37	22	3	34	33
eat Afer Temp	°C	<30	30	25	26	25	75	30	38	57
USL HEME Temp	°C	1	330	32	32	32	22	25	20	26
ubbler Flow Rate (total)	sccm		4203	4200	11201	LIGI	100	50	32	31
lenum vacuum	in. H <sub>2</sub> O	0.1 - 2.5	0,9	1.0	1.1	09	4200	4198	4190	4173
		Finish time:	1615	1220	1506	1907	0001	1.0	10	1.1
	OG	Flow 1	20.6	05.11	22.50	2521	2014	2/11	2203	23/1
eviewed by: Thank Di		1	an e v	-214	-2600	40.TI	62.31	28.02	27.71	23.44

			MA	200			(1)			aluli
Temperature and Electrical	Opera	ator (initials):	MJ.	OPL	119	On	115	$(\Omega)$	11	100116 -
Data (HMI)		Date:	eq12/13	21212	19/12/1	3 2M/	18 9/12/1	S DAVIS	andis	4/12/1
DESCRIPTION	LINITC	Start time:	0000	DITE	0225	03:13	0411	05.10	OGIL	6730
Melt Temperature (T1)	00115	THE THE	11.2	This of	and a set	1111 St. 15			and the second	
Melt Temperature (T2)	°C	1125-1175	ILLS	1129	1125	[111	1120	1096	1102	112/
Melt (Electrode) Temp Setpoint	°C	1125-11/5	1151	1105	1149	1163	1157	1166	1158	1153
Secondary Electrode Current	Ampo	1150	1160	1160	1160	1160	1160	146	1160	1160
Secondary Electrode Potential	Volte		106.6	104.2	164.7	87.3	94,3	817	88	110,2
Secondary Electrode Power (calc)	kW (VxA)		40,4	45.5	46.5	923	48.9	41,2	41	41.5
Melt Resistance (calc)	O(V/A)		1,955	7.771	9,868	3.69	4.15	3.37	3.608	4.683
Electrode Power Output	0/0		67575	5,931	0.444	0.485	2.465	2.504	2.466	2.386
Electrode Power Control	A or M		LODIC	78.8	20.1	49.2	55.3	46.C	49.8	61.6
Melter Temperature Control	AUM	T1 or T2	4	A	A	A	A	A	A	A
Kiln Monitored (Middle) Temp	٥٢	110112	TU	120	76	72	TZ	72	TZ	72
Kiln Top Temp	°C		000	10 15	718	70-	691	1891	656	649
Kiln Bottom Temps	°C		TAUD	668	665	652	GWI	1032	615	598
Kiln Temp Setpoint	°C		610	154	910	YTT	745	-135	724	717
Kiln Power	kW		0	-	700	-			-	-
Kiln Power Output	%		6	-	102	-	-	-	ł	-
Kiln Control	AorM		0		24.0	-	~	-	~	-
Discharge Canister Temp	°C	750 - 850	N.	m.	AT	M	M	M	IL	m
Discharge Canister Temp Setpoint	°C	750 050		-			1	-	1	
Discharge Canister Power	kW							5		
Discharge Canister Power Output	%	-		-				-		
ourspout Heater Actual Temp	°C	1000-1100	1071	100	1.12	1	Latur		1	/
ourspout Heater Temp Setpoint	°C	1000 1100	1051	1050	1050	1648	1047	1049	1049	1053
ourspout Heater Power	kW		211	2000	1050	1050 -	100	1044	1049	1049
ourspout Heater Power Output	%		457	Tipz	1-14	2.15	2.54	132	2.38	2037
lenum Temperature	°C	500-700	547	10,0	42.5	50.2	49	48.10	49.2	49.5
eed Nozzle Temp (FNT)	°C	<50	29	4.40 pm	001	49.5	603	490	452	4741
Off-Gas Temp (OGT_1)	°C	<350	227	221	69	68	29	27	27	27
off-Gas Temp (OGT_#2)	°C		729	266	135	118	239	723	207	193
crub Liquid Temp (SLT)	°C	<40	34	211	250	209	1.12	210	196	185
ost EVS Off-Gas Temp	°C	<50	34	20	76	23	251	2	32	32
eat Xfer Temp	°C	<30	26	16	24	20	150	35	35	33
ost HEME Temp	°C		31	27	30	201	26	26	25	25
ubbler Flow Rate (total)	sccm		4206	Dec	117.05	20	En	49	201	29
enum Vacuum	in. H <sub>2</sub> O	0.1 - 2.5	1.1	10	400	4205	7199	165	4204	4202
and the state of the state	F	inish time:	0011	CHIE	0710	mie	1,0	1.0	1.0	0.9
	OGI	LOW	13,47	2201	TUNE	2315	0410	15:13	0613	0738

										-
Temperature and Electrical	Oper	ator (initials)	: mil	JER	36	54	52	171-	mul	R
Data (HMI)		Date	: 9-12-13	9/12/13	3 7/12/13	9/12/13	9/12/13	9/11/1	3 9/12/15	an
DESCRIPTION		Start time	: 08.24	0935	1032	1/33	1234	1339	1001	1 113
DESCRIPTION Molt Tomporature (T1)	UNITS	RANGE			Salation Prov	N.B. Caller	a contemportation		111	1 1676
Melt Temperature (T1)	°C	1125-1175	1097	1118	1126	1139	1116	1/23	1130	1 UIV
Melt Temperature (12)	°C	1125-1175	1104	1100	1158	1150	1162	1159	11100	111
Melt (Electrode) Temp Setpoint	°C	1150	1160	1160	1160	1160	1160	1169	1160	110
Secondary Electrode Current	Amps		Sec. 8	93.1	93.7	111.7	89.1	923	gr. y	a. 4
Secondary Electrode Potential	Volts		43.4	43.4	41,5	47.9	43.9	48.4	40.9	11.4
Secondary Electrode Power (calc)	kW (V×A)		3.77	4.05	3.89	5.35	39/1	In BIT	13741 20	116
Meit Resistance (calc)	Ω (V/A)		05	0.466	0.443	0.419	0 493	2536	A HIC	- Olier
Electrode Power Output	%		48.7	53.8	524	67.6	5.2.6	517	0.765	0.90
Electrode Power Control	A or M		A	A	A	A	A	A	54.5	01. k
Melter Temperature Control	Sector Sector	T1 or T2	T2	T2	TL	177	172	TI	H	1
(In Monitored (Middle) Temp	°C		6.59	678	681	187	192	101	12	12
Kiln Top Temp	°C		608	621	617	120	120	100	692	105
Kiln Bottom Temps	°C		717	727	718	725	72/	637	641	600
Kiln Temp Setpoint	°C		AFF	0++	- CC	112	126	139	733	741
Kiln Power	kW		1	1	OF	off	PIT	art	off	OFF
Kiln Power Output	%				+1				0.9	
Kiln Control	A or M		IM	14	1 M	1/		1		
Discharge Canister Temp	°C	750 - 850	1270	044	and	- AC	10	pa	1	
Discharge Canister Temp Setpoint	°C		75TC	orr	off	off	off	arr	off	
Discharge Canister Power	kW				+/				00	
Discharge Canister Power Output	%			1	1		-/	1		
ourspout Heater Actual Temp	°C	1000-1100	trug	10.96		1107	1	/		1
ourspout Heater Temp Setpoint	°C	1000 1100	1099	1011	1101	TIOL	(100	1299	1100	1104
ourspout Heater Power	kW		1100	1100	1100	1199	1100	1100	1100	1100
ourspout Heater Power Output	0/0		3.28	J. 23	2.90	3.01	2.84	2.86	2.86	3.14
lenum Temperature	°C	500-700	12	35.4	53.9	55.3	53.5	53.5	53.7	56.0
eed Nozzle Temp (FNT)	°C	<50	488	541	480	529	506	535	521	534
ff-Gas Temp (OGT 1)	°C	<350	-0	78	41	21	28	29	28	28
ff-Gas Temp (OGT #2)	°C	1350	215	101	198	190	218	241	223	220
crub Liquid Temp (SLT)	00	<10	207	158	192	1.81	214	236	2.20	221
ost EVS Off-Gas Temp	°C	<50	32	32	32	32	32	32	32	33
eat Xfer Temp	°C	< 30	35	35	35	35	35	36	37	36
ost HEME Temp	00	<30	25	72	25	25	25	25	25	25
ubbler Flow Rate (total)	scom		29	56	29	30	32	31	31	32
enum Vacuum	in H.o	01-25	4200	4201	4201	4195	4201	4103	1201	HZOU
	111 1120	0.1 - 2.5	1.0	1.0	0.9	.3	1.1	1.5	10	1.0
DI EL:	01	Finish time:	0837	0939	1035	1135	1236	1342	14.12	1629
_ G Flow	Ol2	Hourl	1	1	- 001	11187	2000	00111	APT	1001

. . . . .

-				0	0	00	-0	Fage		191-
Temperature and Electrical	Ope	rator (initials)	: 19	KK	R	19	R	31-	131-	1 31
Data (HMI)		Date	1912	9/12	9/12	912	9/12	9/13	9/12/13	9/12/
DESCRIPTION	LINITTO	Start time	1741	1830	2001	2058	2201	2303	1414	010
Melt Temperature (T1)		RANGE	1100	11	a Nelson and		Section	1	- en j	10/0/
Melt Temperature (T2)	00	1125-11/5	1139	1138	1115	1160	1188	1140	1148	1139
Melt (Electrode) Temp Setpoint	-C	1125-11/5	1158	1159	(158	1118	1121	1131	1163	113
Secondary Electrode Current	Amos	1150	1160	1160	1160	1160	1160	1160	1162	11/0
Secondary Electrode Potential	Amps		95.8	98.6	93.0	92.1	93.9	100.9	991	940
Secondary Electrode Power (calc)	VOILS		45.5	46.0	47.6	9 42.6	32.7	59.5	41.1	47
Melt Resistance (calc)	KVV (VXA)		4.30	4:54	4.43	3.93	3.54	5.095	4672	441
Electrode Power Output	52 (V/A)		0.415	0.467	0.512	\$ 0.463	0.40	0.5005	2.475	000
Electrode Power Control	90		53.8	56.0	52.80	\$ 62.0	52.8	56.8	558	56.1
Melter Temperature Control	A or M	-	A	A	Ag	A	A	A	A	1 2011
Kiln Monitored (Middle) Tama	00	T1 or T2	TZ	TZ	120	TLI	-71	72	51	TO
Kiln Ton Temp	00		715	730	725	711	201	741	745	TE
Ciln Bottom Temps	<u> </u>		661	677	674	660	668	199	195	12
Ciln Temp Sotpoint	°C		749	752	754	747	238	354	710	100
Cin Power	°C		OFF	OFF	OFF	OFF	OFE	ant	107	110
Cilp Power Output	kW		1		1			107	lert	217
(in Control	%							+ /	+ -	
Discharge Capister Terre	A or M							+ /	+ /	
Discharge Canister Temp	°C	750 - 850						+	+	
Discharge Canister Temp Setpoint	°C									
Discharge Canister Power	kW							+/		
Pourspout Hoster Astro-LT	%		OFF	OFF	OFE	AFF	dec	all	-10-	-
ourspout Heater Actual Temp	°C	1000-1100	1110	1101	1100	1100	Nac	UT	ort	014
ourspout Heater Temp Setpoint	°C		1100	1100	1100	11 00	1100	1101	110.3	1100
ourspout Heater Power	kW		3.15	2.79	2.74	2 24	1100	1105	1100	1100
lonum T	%		56.6	53.1	576	51 2	180	1.18	2.76	2.9
ienum remperature	°C	500-700	598	Luo	322	660	52.7	52.9	52.7	53.7
eed Nozzle Temp (FNT)	°C	<50	30	31	29	25	641	680	665	680
IT-Gas Temp (OGT_1)	°C	<350	241	261	257	121	20	32	31	31
m-Gas remp (OGT_#2)	°C		236	2411	228	216	0 9 10	677	306	306
Crub Liquid Temp (SLT)	°C	<40	32	31	23	2.	204	149	247	251
ost EVS Off-Gas Temp	°C	<50	34	34	26	2L AS	20	3/	32	32
eat xier Temp	°C	<30	26	25	26	20	3	34	35	35
DST HEME Temp	°C		32	31	81	20	25	25	26	25
Ibbler Flow Rate (total)	sccm		11192	4200	LIGU	6000	29	29	29	29
enum vacuum	in. H <sub>2</sub> O	0.1 - 2.5	5.8	10	0.0	440	4145	4202	4201	4197
		Finish time:	1244	1870	7607	0.1	1.1	1.0	1.0	0.9
	OGF	OW	18101	2125	2005	100	2203	2307	2416	0113
viewed by: Minut			10.00	LILOI	63.041	18.611	12.82	19 68	7111	10 000

Page No. \_\_\_\_\_\_\_

Temperature and Electrical	Ope	rator (initials)	): 51-	1.81	151-	131	131	101		
Data (HMT)		Date	: 9/13/12	19/13/1	3 9/12/1	2 alina	9/10/	aliati	mik	- mil
		Start time	: 0200	2304	0420	2 2625	1/2/	5 7101	9/13/	39/13
DESCRIPTION	UNITS	RANGE		10001	0100	TUSUL	10601	0100	0800	0914
Melt Temperature (T1)	°C	1125-1175	1138	1135	1.120	1113	1111	1 / 1 27	man and and	Sale Sale
Melt Temperature (T2)	°C	1125-1175	1117	1150	112	11/2	1115	1116	1111	1124
Melt (Electrode) Temp Setpoint	°C	1150	1110	1127	1165	1162	1160	116/	1158	1761
Secondary Electrode Current	Amps	1100	1012	1040	116	116	1160	1162	1160	1160
Secondary Electrode Potential	Volts		Lat	LAU	99.0	96,6	98.3	98.3	97.6	102
Secondary Electrode Power (calc)	kW (V×A)		49/0	6177	16200	91.8	48.2	48.7	47.0	9 49.0
Melt Resistance (calc)	$\Omega(V/A)$		1495	2471	1,079	4.593	9.739	41787	4.58	7 570.
Electrode Power Output	9/0		103	500	7,917	5.991	4.490	0.495	0.48	2 0,48
Electrode Power Control	AorM		10,0 h	510	52.8	59,0	55.4	55.1	56.0	57.0
Melter Temperature Control		T1 or T2	72	TO	A	A	A	A	4	A
Kiln Monitored (Middle) Temp	°C	110112	100	14	72	71	T2	72	TZ	17
Kiln Top Temp	°C		10	759	758	736	734	732	725	. 74
Kiln Bottom Temps	°C		676	101	703	686	685	683	1-76	690
Kiln Temp Setpoint	°C		168	187	786	769	764	765	760	769
Kiln Power	L KW		OFT	off	ort	ote	off	off	OFF	all
Kiln Power Output	0/0					ľ	1		1	0,0
Kiln Control	AorM									
Discharge Canister Temp	A0114	750 050								
Discharge Canister Temp Setpoint	°C	750 - 850								
Discharge Canister Power	LIM									
Discharge Canister Power Output	04		1	1						
Pourspout Heater Actual Temp	90	1000 1100	off	off	off	off	off	DAG	d1	
Pourspout Heater Temp Setpoint	90	1000-1100	1101	1100	1122	1100	1100	1103	iPt	2600
Pourspout Heater Power	LIAN		1100	1100	1100	1109	1100	1100	1100	1048
Pourspout Heater Power Output	KVV O/		2.79	3.25	3.58	2.68	2.70	7.71	1100	7/00
Plenum Temperature	%	500 700	52,5	56.5	53,3	51,7	60.1	57.0	6.11	2.04
Feed Nozzle Temp (ENT)	20	500-700	630	655	595	602	677	629	36.1	55,8
Off-Gas Temp (OGT 1)	<u> </u>	<50	30	31	29	30	30	30	666	540
Off-Gas Temp (OGT_#2)	00	<350	282	309	249	235	261	745	31	30
Scrub Liquid Temp (SLT)			241	255	209	210	724	714	632	283
Post EVS Off-Gas Tomp	<u></u>	<40	33	33	33	33	33	28	176	240
leat Xfer Temp	00	<50	37	37	37	36	36	36	21	33
Post HEME Temp	00	<30	26	26	26	26	76	76	26	3)
Subbler Flow Pate (total)	°C		30	20	30	30	79	3.0	16	26
Plenum Vacuum	sccm		4196	4200	4196	4102	4701	4198	27	29
	in. H <sub>2</sub> O	0.1 - 2.5	0.8	41	27	0.7	27	00	4196	4201
	A7	Finish time:	0204	2306	0407	0504	26.22	074	15	1.0
eviewed but	06 FI	Sw 1	20.291	18 87	11.77	1906	20201	107	0838	0915
amam	Alun .	m		and the second se	and the second se	1001	2 60 1	16,11	16.61	18:0

Page No. 12/12

Temperature and Electrical	Oper	ator (initials):	1982	mill	DOG	mol		1	1	_
Data (HMI)		Date:	9/13/13	9/10/1	3 9/17/1	aliz/17				
		Start time:	1022	1110	1216	1221				
DESCRIPTION	UNITS	RANGE		1111/	INP	1276			-	
Melt Temperature (T1)	°C	1125-1175	1122	1119	1120	1115		1 703-1-3034	Same Same	N.S. 122
Melt Temperature (T2)	°C	1125-1175	1155	1112	1100	115		1		
Melt (Electrode) Temp Setpoint	°C	1150	LICA	1110	1110	11/0				
Secondary Electrode Current	Amps		105.8	100 2	102.2	1160	1			
Secondary Electrode Potential	Volts		45.5	490	1103.4	108		1		
Secondary Electrode Power (calc)	kW (V×A)		5.15	Che	75.4	48,2		1		
Melt Resistance (calc)	Ω (V/A)		OMC	3.00	7116	5,4				
Electrode Power Output	%		599	0.50	0.44	0-99		100		
Electrode Power Control	A or M		A	20.5	58.9	61,3		R		
Melter Temperature Control		T1 or T2	To	F	/+	A.		31		
Kiln Monitored (Middle) Temp	°C	110112	724	12	Td	72				1
Kiln Top Temp	°C		142	735	677	695		0		1
(iln Bottom Temps	°C		674	682	646	640		L L		-
Kiln Temp Setpoint	°C		771	773	761	757		T		
(iln Power	LIN/		OTE	off	044	off		1	é	-
(iIn Power Output	0%			00		11.9		V		
(iln Control	AorM								1	
Discharge Canister Temp	°C	750 050							1	
Discharge Canister Temp Setpoint	°C	750 - 850			1.1				1	
Discharge Canister Power	LW/		_						1	
Discharge Canister Power Output	0/								1-	
ourspout Heater Actual Temp	90	1000 1100	1	1		X			1	
ourspout Heater Temp Setpoint	00	1000-1100	1091	1103	1095	1100				
ourspout Heater Power	L'INI		1100	1100	1100	1100				
ourspout Heater Power Output	RVV 0/		3.23	2.84	2,90	2.89				
enum Temperature	90	500	67.0	53.3	53.0	53,8				
eed Nozzle Temp (FNT)	20	500-700	628	536	388	646				
ff-Gas Temp (OGT 1)	20	<50	30	29	26	3/				
ff-Gas Temp (OGT #2)	20	<350	274	263	198	304				+
crub Liquid Temp (SLT)	20		338	232	179	199				+
st EVS Off-Gas Temp		<40	33	35	37	33				1
eat Xfer Temp		<50	37	40	41	34				+
st HEME Temp	30	<30	26	26	26	29				
Ibbler Flow Rate (total)			31	33	41	32				
enum Vacuum	sccm	0.1.0.5	4200	4201	4203	11200				
	III. H <sub>2</sub> O	0.1 - 2.5	1.0	1.0	0.9	0.6				1
	F	inish time:	1027	1121	1231	1340				
vious d h	1	10.			1.00 0.11	110				1

.

## Appendix B – Sample Log Sheet

TI-EWG-0008 p.10)3 Attachment A

			Sample Log	Sheet	
Sample Number	Operator	Date:	Start time:	Description	
RSM-EWG8-					
#sequence	initials	e.g., 11/29/10	e.g., 1400 h	e.g., Glass, melter feed, EVS c	ondensate, HEME
RSM-EWG8-01	CDL	9/9/13	05:07	feed 7	
RSM-EWG8-02	JST	9/9/13	0510	ScrubSoln	15:47 9/9/13
RSM-EWG8-03	nuc	9/9/13	10:10	glass 3	samples collected
RSM-EWG8-04	DER	9/9/13	1520	Fred	by Ben Williams
RSM-EWGB-05	muk	9/9/13	1520	Scrub soln	# RID Teken 10
RSM-EWG8-06	PPS	9/9/13	15:55	glass pour	*
RSM-EWG8-07	PPS	9/9/13	18:06	1/555 CER ~ 18.25	tkg k
RSM-EWG8-08	PPS	9/9/13	21:54	glag par	۲ د ۲
RSM-EWG8-09	WCB	919/13	2310	EVS	4
RSM-EWG8-10	PPS	9/5/13	23:37	Feed sample	×
12 SM -EW68-11	ON	9/10/13	02:24	GLASS SAMPLE	ye.
RSM-EW68-12	56	9/10/13	07:45	scrub	*
RSM-EWG-13	56	9/10/13	07:45	Feed	\$
RSM-EV68-14	JOER	9/10/13	1015	GLASS	¥
RSM-EWG8-15	muk	9/10/13	0224	Glass conster	~ (6.82kg #
RSM-EWG2-16	1 mile	9/10/13	1020	Glass caniter	e gr
RSM-ENGE-17	MIK	9/10/13	1425	Glass consister	ď
R5M-EW68-18	muc	Vio/13	1450	Glass Canota	2 0.98 kg #
RSM - ENG	and muk	9/10/13	1515	Scrub Soln	4
RSM- EWG8- RC	DER	9/10/13	1515	Freed	×
RSM-EWG8-21	PPS	9/10/13	18:27	g ass Dour Giv 90.	25g 🗰
RSM-EWG8-22	PPS	9/10/13	20:10	Glass conister ~ 10.3	kg 🎽
RSM-EWG8-23	PPS	9/10/13	22:54	glass pour Grossi	t: 87.69 x
RSM-ENG8-24	WCB	9/10/13	23:02	SEVS'	¥/
RSM-EW98-25	PPS	9/10/13	23:02	feed sample	¥
	A 1				

Reviewed By: \_\_\_\_\_

Date: 10-2/-13

				TI-EWF-0008
				0.202
				Aile along to
			Sample Log	Shoot ATTACK MENU A
Sample Number	Operator	Date:	Start time:	Description
RSM-EWG8-	Operator	Date.	Start time.	Description
#sequence	initials	e.g., 11/29/10	e.g., 1400 h	e.g., Glass, melter feed, EVS condensate, HEME
RSM-EW68-26	ON	9/11/13	01:08	GLASS CAMSTER 5,14KG *
125M-EWG8-27	or	211/13	04:35	GLASS SAMPLE GW: 74,7/9 #
RSM-EU68-29	5 1 13	9-11-15	0656	Feel Soundle
RSMIE CB-29	115	9-11-13	0657	EVS SITUS She Sauple PH= 8 1
R5M-EW68-30	mil	9-11-13	0750	Glass can ~ 10kg 1
RSM-EW68-31	mik	9-11-13	0835	Glass can Nkg sample. Mass = 1,29 kg x
RSM-ENG8-32	mik	9-11-13	0840	Glass Sample GW 73.17 .
RSM-2068-33	MUK	9-11-13	1030	Glass Canista moss not talents ×
RSM-EW68-34	56	9-11-13	1450	Glass Sample 4
RSM-EWG8-34	WeB	9-11-13	1733	glass carrister
RSM-EWG 8-35	WRB	9-11-13	<del>3</del> 200	glass sample
ROM-EWE836	115	9-11-13	2330	feet sample
RSM-ENG8-37	was	9-11-13	2330	EVS Narc Dolution
RSM ENG8-38	ori	9/12/13	01:22	GLASS CAMISTER
RSM-EW68-39	113	9-12:13	0238	glass sample ,
25M-EW68-40	'OL	9-12-13	0656	EVS SCRUB SAMPE PH 8.5
RSm-BW68-41	A	9.12.13	0656	FBED SAMPLE
RSM-SWEB-42	mik	9-12-13	0730	GLass Sample
RSM-EW68-4	3 min	9-12-13	1003 .	BLASS CANister
RSM-EW68-44	1 201	9-12-13	1140	Glass Sample (large) 1.2kg
RSM-EW68-6	15 1982 V	1213/12/13	1225	EVS SCHUD Scaphe
RSM-E4 )68-46	2 Was	9/12/13	1615	glass sample
RSM-EWG8-47	WCB	19/12/13	1740	allows can GW= 11,067 kg
RSM-EWG8-48	web	9/12/13	2000	your gample
RSM-EW68-49	web	9/12/13	2300	Scrub (EVS)

Réviewed By: Marine Dr. Mrs Javinjary 19/13

Date: 10-7-13

				TI-EWG-0008
				0.303
				Ada ab apart A
				ATTACONMENT AT
0 1 11 1		D	Sample Log S	Sheet
Sample Number	Operator	Date:	Start time:	Description
#sequence	initials	e.g. 11/29/10	e.g. 1400 h	e.g. Glass melter feed EVS condensate HEME
RSM-EUG8-50	AJCB	9/12/12	7310	Melterlad
RSM-EW68-51	FI	9/13/13	2405	Glass Sample
RSM-EWG8-57	5/ .	9/13/13	2125	Glass Sourde
RSM-EW1-8-53	11.)	9,13.13	0558	feed sample
RSM-EWG55	+ (71)	9-13.13	0559	Scrub slu sauple PH= 8.5
RSM-ENG855	JE	9-13-13	0650	Glass camister 15,10, Kg (NOR 201)
RSM-EW68-5	5 DER	9/13/13	0830	white Vasidon ON GLISS CONISTRO 1913
R5m-EWG-8-57	mik	9/13/13	0850	Glass sample
R5m-Eux68-58	melle	9/13/13	1235	Scrubbu sample pH = 7
RSM-EWG8-59	Rady	9/13/13	1240	Ferd 12/13/13
RSM-ENGB-60	PA	9/13/13	1530	Large glass container 12,50kg glass
RGM - EWGB-61	Ing	91/3/13	1300	Melf Strare sample (glass) < 10g
RSM-62-62	30	10/7/13	1320	Melt surface sample (glass) = 50g
		2		
			0100	
			10 2305	
21	1.	5		10 7/12
Reviewed By:	any AN	my	Da	ate: 10-2/-13
	100 mm			10/23/13
	Hours			

## Appendix C – Operating Parameter Data Plots from RSM Tests



Figure C.1. Simulant Feed Pump Set Point and Balance Reading



Figure C.2. Simulant Feed Pump Set Point and Calculated Feed Rate



Figure C.3. Melter and Plenum Temperatures and Electrode Power



Figure C.4. Melter Vacuum



Figure C.5. Electrode Current and Potential

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