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Melter Testing for Technetium Removal Using Goethite Precipitation, VSL-11R2300-1, Rev. 0

Prepared for the U.S. Department of Energy
Assistant Secretary for Environmental Management



P.O. Box 550
Richland, Washington 99352

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Final Report

**Melter Testing for Technetium Removal Using
Goethite Precipitation**

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Completeness of Testing:

This report describes the results of work and testing specified by WRPS. The work and any associated testing followed established quality assurance requirements. The descriptions provided in this test report are an accurate account of both the conduct of the work and the data collected. Results required by the test program are reported. Also reported are any unusual or anomalous occurrences that are different from the starting hypotheses. The test results and this report have been reviewed and verified.

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TABLE OF CONTENTS

LIST OF TABLES.....	4
LIST OF FIGURES.....	5
LIST OF ABBREVIATIONS	6
SECTION 1.0 INTRODUCTION	7
1.1 TEST OBJECTIVES	7
1.2 QUALITY ASSURANCE.....	8
1.3 MELTER SYSTEM DESCRIPTION	9
1.3.1 Feed System	9
1.3.2 Melter System.....	9
1.3.3 Off-Gas System	9
1.4 EXPERIMENTAL AND ANALYTICAL METHODS.....	10
1.4.1 Glass Preparation.....	10
1.4.2 Compositional Analysis	10
1.4.3 Mössbauer Spectroscopy	10
1.4.4 Feed Rheology	11
1.4.5 Melter Exhaust Sampling and Analysis	11
SECTION 2.0 WASTE SIMULANT AND GLASS FORMULATIONS	12
2.1 HLW AZ-101 WASTE SIMULANT	12
2.2 GLASS AND FEED FORMULATION.....	12
2.3 PNNL IRON-RHENIUM PRECIPITATE	13
2.4 ANALYSIS OF FEED SAMPLES.....	14
2.4.1 General Properties	14
2.4.2 Rheology.....	14
2.4.3 Chemical Composition.....	15
SECTION 3.0 MELTER OPERATIONS.....	16
3.1 MELTER OPERATIONS DATA	17
SECTION 4.0 GLASS PRODUCTS	19
4.1 DISCHARGE GLASSES.....	19
4.2 GLASS POOL SAMPLES.....	20
SECTION 5.0 MONITORED OFF-GAS EMISSIONS	21
SECTION 6.0 MASS BALANCE FOR RHENIUM IN GLASS AND EMISSIONS.....	23
SECTION 7.0 SUMMARY AND CONCLUSIONS	25
SECTION 8.0 REFERENCES	26

List of Tables

	<u>Page No.</u>
Table 2.1 Analyzed Compositions of AZ-101 Envelope D Waste.	T-1
Table 2.2 Compositions of the AZ-101 Waste (Oxide Basis) and the HLW Waste Simulant to Produce 100 kg of Waste Oxides (21.5 wt% total solids).	T-2
Table 2.3 Composition of Nominal AZ-101 HLW Glass Formulation (wt%).	T-3
Table 2.4 Summary of AZ-101 HLW Characterization Data for Crucible Glass.	T-4
Table 2.5 Compositions of the Nominal AZ-101 HLW Glass Formulation and the Glass Former Mix to Produce 100 g of AZ-101 HLW Glass.	T-5
Table 2.6 Characteristics of PNNL Iron-Rhenium Slurries for Melter Testing.	T-6
Table 2.7 Characteristics of Melter Feed Samples from DM10 Goethite/Rhenium Tests.	T-7
Table 2.8 XRF Analyzed Compositions for Vitrified Melter Feed Samples from DM10 Tests (wt%).	T-8
Table 3.1 Summary of DM10 Melter Tests.	T-9
Table 4.1 Listing of Glasses, Masses, Cumulative Masses, and Rhenium Concentrations from DM10 Tests.	T-11
Table 4.2 XRF Analyzed Compositions for Glass Discharged During DM10 Tests (wt%).	T-13
Table 4.3 List of Glass Pool Samples with Sampling Times, Measured Iron Oxidation State, Measured ReO ₂ Concentrations and Observations of Secondary Phases.	T-19
Table 4.4 Comparison of XRF Analyzed Compositions for Glass Pool and Discharge Glass Samples Taken from the end of DM10 Tests (wt%).	T-20
Table 5.1 Results from DM10 Test 1 Off-Gas Emission Samples.	T-21
Table 5.2 Results from DM10 Test 2 Off-Gas Emission Samples.	T-22
Table 5.3 Results from DM10 Test 3 Off-Gas Emission Samples.	T-23
Table 5.4 Results from DM10 Test 4 Off-Gas Emission Samples.	T-24
Table 5.5 Results from DM10 Test 5 Off-Gas Emission Samples.	T-25
Table 5.6 Average Emissions Rates and Carry-over for DM10 Tests.	T-26
Table 6.1 Mass Distribution of Rhenium Based on Alternative Feed Analyses and Glass Background Corrections.	T-27

List of Figures

	<u>Page No.</u>
Figure 1.1 DM10 melter and feed tank; off-gas system is in the background to the left.	F-1
Figure 1.2 Schematic diagram of DM 10 vitrification system.	F-2
Figure 2.1 Feed composed of heated goethite slurry prior to mixing in additional water.	F-3
Figure 2.2 Feed composed of heated goethite slurry prior to mixing in additional water.	F-4
Figure 2.3 Viscosity versus shear rate for each feed with each iron and rhenium source.	F-5
Figure 3.1 Glass production (cumulative) and bubbling rates for DM10 tests with goethite and rhenium.	F-6
Figure 3.2 Glass temperatures for DM10 tests with goethite and rhenium.	F-7
Figure 3.3 Plenum temperatures for DM10 tests with goethite and rhenium.	F-8
Figure 3.4 Electrode power and glass pool resistance for DM10 tests with goethite and rhenium.	F-9
Figure 4.1 DM10 product and target glass soda and silica concentrations determined by XRF.	F-10
Figure 4.2 DM10 product and target glass aluminum and iron oxide concentrations determined by XRF.	F-11
Figure 4.3 DM10 product and target glass zinc and zirconium oxide concentrations determined by XRF.	F-12
Figure 4.4 DM10 product and target glass chromium and cadmium oxide concentrations determined by XRF.	F-13
Figure 4.5 DM10 product and target glass cesium and cerium oxide concentrations determined by XRF.	F-14
Figure 4.6 DM10 product and target glass rhenium oxide concentrations determined by XRF.	F-15

List of Abbreviations

ASME	American Society of Mechanical Engineers
CUA	The Catholic University of America
DCP-AES	Direct Current Plasma Atomic Emission Spectroscopy
DM	DuraMelter
DOE	Department of Energy
DWPF-EA	Defense Waste Processing Facility-Environmental Assessment
EDS	Energy Dispersive X-ray Spectroscopy
HEPA	High-Efficiency Particulate Air Filter
HLW	High Level Waste
IC	Ion Chromatography
ICP	Inductively Coupled Plasma
LAW	Low Activity Waste
M	Molarity
N	Normality
NIST	National Institute of Standards and Technology
NQA	Nuclear Quality Assurance
PCT	Product Consistency Test
PNNL	Pacific Northwest National Laboratory
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QARD	Quality Assurance Requirements and Description
RPP	River Protection Project
RPP-WTP	River Protection Project-Waste Treatment Plant
SEM	Scanning Electron Microscopy
TCLP	Toxicity Characteristic Leaching Procedure
UTS	Universal Treatment Standards
VSL	Vitreous State Laboratory
WRPS	Washington River Protection <i>Solutions</i> , LLC
WTP	Hanford Tank Waste Treatment and Immobilization Plant
XRD	X-Ray Diffraction
XRF	X-Ray Fluorescence

SECTION 1.0 INTRODUCTION

During vitrification of low activity waste (LAW) at the Hanford Tank Waste Treatment and Immobilization Plant (WTP), liquid effluents are produced in the primary off-gas treatment system that processes the exhaust gases from the LAW melter. That effluent stream, which contains captured hazardous and radioactive species from the melter exhaust, is then recycled and eventually becomes part of subsequent melter feed batches. Other constituents, such as sulfur and halides, are also recycled in this process and this can impact the LAW glass loadings that are achievable. Thus, breaking the melter recycle loop could have advantages with respect to reducing LAW glass volumes. However, this recycle stream cannot currently be diverted to liquid secondary waste treatment because of the levels of several constituents in the scrub solutions, particularly technetium. Researchers at Pacific Northwest National Laboratory (PNNL) have been investigating a process whereby the technetium could be precipitated from the off-gas recycle stream in the mineral goethite ((Tc, Fe)OOH). It could then be diverted to the high level waste (HLW) stream through the WTP pretreatment facility and the decontaminated liquid stream could be sent to secondary waste treatment. As part of the test program to develop the goethite precipitation process, it is necessary to assess the impacts of including this material in the HLW melter feed and the extent to which the captured technetium is volatilized during HLW vitrification. To support such testing, sufficiently large batches of material were prepared at PNNL using rhenium as a surrogate for technetium. These materials were shipped to the Vitreous State Laboratory (VSL) of the Catholic University of America (CUA) for testing on a DuraMelter 10 (DM10) small-scale joule-heated ceramic melter system. This report describes the testing that was performed with those materials.

1.1 Test Objectives

The principal objectives of the DM10 tests at the VSL were to demonstrate processing of a Hanford HLW waste stream combined with two different forms of iron-containing materials that were produced during the precipitation of rhenium from simulated LAW recycle streams. A simulant of an AZ-101 HLW composition based on an actual waste sample analysis, which was previously processed on a continuously fed melter, was selected for these tests. The goal of these tests was to compare Hanford HLW melter feed simulants, containing rhenium added directly or in various types of iron compounds, with respect to processing properties, rhenium retention, and off-gas compositions.

Specific objectives of these tests were to:

- Demonstrate on the DM10 melter acceptable processing (glass production rate, feed distribution across molten glass, glass redox, off-gas characteristics, melter feed mixing/pumping) of a Hanford HLW high-iron waste stream (AZ-101) with the iron

oxide source substituted with various forms of iron-containing materials produced at PNNL during rhenium precipitation.

- Determine rhenium retention in the DM10 glass product with three different iron sources and two different rhenium sources: (i) iron hydroxide with perrhenic acid (baseline); (ii) PNNL heated iron precipitate loaded with rhenium; and (iii) PNNL unheated iron precipitate loaded with rhenium.
- Determine rhenium emissions from the DM10 with the same three iron and rhenium sources.
- Characterize the chemical composition of each discharge glass with particular emphasis on rhenium and determine the iron oxidation state for glass from the end of each melter test for each feed composition.
- Sample and characterize discharge glass samples at the end of each test segment.
- Characterize the melter emissions (particulate, aerosol, and gaseous) to permit material mass balance across the melter for each iron and rhenium source, with particular emphasis on rhenium.

It should be noted that the testing involved complete replacement of the iron source in the melter feed by each of the rhenium-loaded goethite materials provided by PNNL. This approach was used in order to maximize any differences in rhenium retention and thereby optimize the sensitivity of the tests. However, if such a precipitation process were implemented, a more likely scenario in actual WTP operations would be that the goethite precipitate would form an additional iron stream to the melter feed, over and above that derived from the HLW solids.

1.2 Quality Assurance

Testing was performed according to the existing quality assurance program that is in place at VSL. That program is compliant with applicable criteria of 10 CFR 830.120; Office of Civilian Waste Management DOE/RW-0333P, Quality Assurance Requirements and Description (QARD) Revision 20; the American Society of Mechanical Engineers (ASME) NQA-1, 2004; and DOE Order 414.1 C, Quality Assurance. The requirements of DOE/RW-0333P were applicable to the following specific aspects of this work:

- Crucible melt preparation of HLW glasses
- Analysis of HLW crucible melt glasses

The program is supplemented by a Quality Assurance Project Plan for WRPS work [1] that is conducted at VSL. Test and procedure requirements by which the testing activities are planned and controlled are also defined in this plan. The program is supported by VSL standard operating procedures that were used for this work [2].

1.3 Melter System Description

Testing was conducted on one of the two DM10 melter systems installed at the VSL, shown in Figure 1.1. A schematic diagram of the DM10 system is shown in Figure 1.2 and the principal components of the system are described in the following sections.

1.3.1 Feed System

The feed container is mounted on a load cell for weight monitoring and is stirred continuously except for periodic, momentary interruptions during which the weight is recorded. The material in the feed container is constantly recirculated, which provides additional mixing. The recirculation loop extends to the top of the melter where feed is diverted from the recirculation loop into the melter through a Teflon-lined feed line and water-cooled feed tube. The feed rate is regulated by a peristaltic pump that is located in between the recirculation loop and the feed tube.

1.3.2 Melter System

A DuraMelter 10 (DM10) system was used for this work. The Monofrax K3 ceramic refractory-lined melter includes two MA 758 plate electrodes that are used for joule-heating of the glass pool and a bubbler for mixing the melt. The DM10 melter has a melt surface area of 0.02 m² and glass inventory of about 8 kg. The glass product is removed from the melter by means of an air-lift discharge system.

1.3.3 Off-Gas System

For operational simplicity, the DM10 is equipped with a dry off-gas treatment system involving gas filtration operations only. Exhaust gases leave the melter plenum through a film cooler device that minimizes the formation of solid deposits. The film-cooler air has constant flow rate and its temperature is thermostatically controlled. Consequently, the exhaust gases passing through the transition line (between the melter and the first filtration device) can be sampled at constant temperature and air flow rate. The geometry of the transition line conforms to the requirements of the 40-CFR-60 air sampling techniques. Immediately downstream of the transition line are cyclonic filters followed by conventional pre-filters and HEPA filters. The temperature of the cyclonic filters is maintained above 150°C while the HEPAs are held above 100°C to prevent moisture condensation. The entire train of gas filtration operations is duplicated and each train is used alternately. An induced draft fan completes the system.

1.4 Experimental and Analytical Methods

The measurements and analyses that were performed in this work are detailed in controlled VSL technical procedures [2], which constitute part of the VSL QA program. This section provides a brief description of the equipment and experimental methods that were used.

1.4.1 Glass Preparation

Each crucible-melt glass was prepared from reagent grade or higher purity chemicals to produce a batch size of approximately 400 g. Crucible melts were prepared by melting the appropriate combination of well-mixed chemicals at 1150°C for 120 minutes in a platinum or platinum-gold crucible. Mixing of the melt was accomplished mechanically with a platinum stirrer, beginning 15 minutes after the furnace temperature reached 1150°C and continuing for the next 100 minutes. The molten glass was poured onto a graphite plate to cool, and the resulting glass was then distributed for analyses.

1.4.2 Compositional Analysis

Each glass sample was powdered and sieved to give -200 mesh material before analysis by X-ray fluorescence spectroscopy (XRF). An ARL 9400 wavelength dispersive XRF spectrometer running UniQuant™ was used for analysis. An additional analysis to quantify rhenium more precisely was also conducted using the XRF. This method involved analyzing a specific spectral line and the associated background for extended periods instead of using the UniQuant™ program. Boron and lithium were determined by total acid dissolution of ground glass samples in HF/HNO₃ and subjecting the resulting solutions to DCP-AES analysis.

1.4.3 Mössbauer Spectroscopy

Mössbauer spectroscopy is an analytical technique that utilizes the recoil-free emission and resonant absorption of gamma rays by nuclei bound in solids. The energies of the gamma ray emitter (source) and absorber (sample) have to be closely matched and, therefore, the number of elements that can be studied using this technique is limited. One of the elements that has been studied extensively using Mössbauer spectroscopy is iron. In this work, Mössbauer spectroscopy was used to measure the fraction of iron in the Fe²⁺ and Fe³⁺ states, which can be used as a measure of the redox state of the glass sample.

Mössbauer spectra were collected using an American Magnetic/Ranger Scientific MS-1200 system equipped with a ⁵⁷Co source in a rhodium matrix with glass powder as the sample. Both divalent and trivalent iron show doublet peaks in the Mössbauer spectra and the peak areas are proportional to the concentrations of the respective species in the glass. Even though the peaks overlap partially, software can be used to deconvolute the peaks and calculate the peak areas. The ratio of the areas of the Fe²⁺ and Fe³⁺ peaks is equal to the ratio of their

concentrations in the glass. The redox measurements are calibrated using a set of six standard glass samples ranging in Fe^{2+} to Fe_{total} values from 7 to 90%. The standards include a NIST traceable Obsidian Rock (SRM 278), five standard glasses analyzed by Corning, Inc. for their redox state, and the SRL-EA glass. The NIST Standard iron foil (SRM 1541) was used to calibrate the instrument and determine the zero velocity channels. Since Doppler shifts in energy are measured in Mössbauer spectroscopy, the velocity is a measure of the shift in energy; knowing the zero velocity channel therefore helps in identifying the Fe^{2+} and Fe^{3+} peaks.

1.4.4 Feed Rheology

A Haake rheometer (Model RS600) was used to measure the yield stress and the dependence of shear stress on shear rate of melter feeds. For measurement of shear stress vs. shear rate, the instrument employs a cylindrical rotor and a matching sample cup and is operated in controlled shear rate mode. The steady-state shear stress is determined for a range of shear rates from 0.1 s^{-1} to 1000 s^{-1} . The rheology data are analyzed for the onset of Taylor vortices and the affected data are so noted. Yield stress is determined using a vane rotor and matching sample cup. The measured torque versus time is monitored at a given shear rate and the maximum torque is used to calculate the yield stress. All measurements were made at 25°C ; previous work [3], which examined a range of temperatures, showed a relatively weak effect of temperature.

1.4.5 Melter Exhaust Sampling and Analysis

The melter exhaust was sampled for metals/particles according to 40-CFR-60 Methods 3, 5, and 29 at steady-state operating conditions during each test segment. The concentrations of off-gas species that are present as particulates and gaseous species that are collected in impinger solutions were derived from laboratory data on solutions extracted from air samples (filters and various solutions) together with measurements of the volume of air sampled. Particulate collection required isokinetic sampling, which entails removing gas from the exhaust at the same velocity that the air is flowing in the duct (40-CFR-60, Methods 1-5). Typically, a sample size of 30 dscf was taken at a rate of between 0.5 and 0.75 dscfm. Total particulate loading was determined by combining gravimetric analysis of the standard particle filter and chemical analysis of probe rinse solutions. An additional impinger containing 2 N NaOH was added to the sampling train to ensure complete scrubbing of all acid gases. The collected materials were analyzed using direct current plasma atomic emission spectroscopy for the majority of the constituents and ion chromatography (IC) for anions.

SECTION 2.0 WASTE SIMULANT AND GLASS FORMULATIONS

2.1 HLW AZ-101 Waste Simulant

In a previous study, actual waste solids from tank AZ-101 were pretreated and analyzed at PNNL. The pretreatment started with washing of the solids (insoluble solids = 317.9 g) twice with 0.01 M NaOH solution (1000 g each), which was followed by caustic leaching with approximately 3 M NaOH at 85°C. After 8 hours of leaching, the slurry was then batch rinsed three times with 0.01 M NaOH (1200 g each). The resulting slurry, which had a solids content of 10.9 wt% with 130.2 g of total insoluble solids, was analyzed; the resulting chemical composition data are listed in Table 2.1. This composition [4] was the basis for the development of suitable glass composition for this waste for subsequent testing [5].

The AZ-101 simulant composition used for recent melter tests [6, 7] was also based on the actual waste analysis provided in Table 2.1. Uranium and most constituents at less than about half a weight percent oxide were omitted. Exceptions were chromium and sulfur, which were added to the waste simulant. Non-radioactive cesium was also added at the designated level of half a weight percent. The recipe for the resulting AZ-101 simulant is provided in Table 2.2. For the purpose of the present work, the concentrations of the volatile components (i.e., carbonate, nitrite, nitrate, and organic carbon) are assumed to be similar to those found for the AZ-102 HLW waste [8]. With the waste compositions defined, formulation of the HLW waste simulant proceeds in a straightforward fashion. In general, oxides and hydroxides are used as the starting materials, with slurry of iron (III) hydroxide (13% by weight) as the major constituent. Volatile inorganic components are added as the sodium salts, whereas organic carbon is added as oxalic acid.

2.2 Glass and Feed Formulation

Several glasses were developed and evaluated as candidates for the AZ-101 waste stream based on the actual waste sample analysis [5]. The glass deemed most suitable for the waste (HLW98-95) is compared to the nominal glass composition used for the previous [7] and current tests in Table 2.3. The additive type and oxide percentage, 68.25%, are the same in the two glasses. The small differences in the glass product composition are the result of removal of uranium and several constituents at low concentrations, the exclusion of cesium and technetium pretreatment products, and renormalization of the waste oxide composition. The only difference between the glass previously processed and the glass processed in the current tests is the exclusion of RuO₂ from the waste composition in the present tests. A summary of the properties of the HLW98-95 glass is provided in Table 2.4. The measured glass properties indicate that the glass meets all WTP processability, product quality, and contract requirements.

NOAH Technologies Corporation produced the waste simulant blended with glass forming additives under sub-contract to VSL according to the formulation provided in Table 2.5 for the tests with the nominal feed composition. NOAH was the supplier of simulant and feed samples used in previous testing for the WTP performed at VSL on the DM100 and DM1200 melter systems [8-19]. The waste simulant was received in 55-gallon drums and was thoroughly homogenized prior to use. The chemical additives that were used to produce the melter feeds were selected based on previous testing and the RPP-WTP Project baseline glass forming chemicals. Concentrated perhenic acid was added to the baseline feed to achieve a rhenium concentration in the target glass of 0.05 wt% ReO_2 if all of it were to be retained in the glass product.

2.3 PNNL Iron-Rhenium Precipitate

Two samples of the iron-rhenium precipitate material prepared at PNNL were shipped to VSL for melter testing. These samples consisted of approximately 28 kg of “heated” material and 25 kg of “unheated” material. The heating process was intended to convert the precipitate to the goethite form. The results from analysis of the received material are provided in Table 2.6. Both materials are composed of about 89% water and 11% solids. The dried material was analyzed by x-ray fluorescence spectroscopy (XRF) and x-ray diffraction (XRD) to determine chemical composition and iron mineral type. As expected from the PNNL analysis and description, the material was mostly iron oxide (97.5%), with about half a weight percent rhenium oxide and about two percent contaminants in the form of aluminum, calcium, and chlorine. Additional analysis of the dried material involving microwave aided acid dissolution followed by direct current plasma – atomic emission spectroscopy (DCP-AES) solution analysis was also conducted at VSL. The results of this analysis are in good agreement with the XRF analysis with slightly higher aluminum contents and trace amounts of magnesium, sodium, and silicon. The results from direct XRF analysis of the dried solids for rhenium are about a third higher than the PNNL inductively coupled plasma (ICP) analysis. Given the low rhenium concentrations and the fact that the XRF calibration is designed primarily for a glass matrix, the ICP analysis was considered more accurate and was therefore used to calculate target rhenium concentrations for the feeds constituted with goethite slurries. The dominant mineral phases present in slurries estimated by PNNL (magnetite and goethite) were confirmed by XRD analysis at VSL. However, the iron hydroxide assumed to be present in the unheated slurry could not be confirmed by the XRD method; furthermore, any amorphous phases that may be present would not be detected.

The heated and un-heated materials received from PNNL were used to produce two separate melter feed batches. In each case, the thirteen percent $\text{Fe}(\text{OH})_3$ slurry shown in Table 2.2 was replaced on a molar basis with each of the iron-rhenium slurries received from PNNL. The average solids content of eleven percent and the XRF measured iron content of 97.5 wt% iron oxide was used to calculate the amounts of slurry required for each of the feed batches. The small amounts of aluminum, calcium, and chlorine will result in a change of less than 0.1 wt% in the product glass. The concentrations of rhenium in the target glass were 0.043 and 0.042 wt% ReO_2 if all of it were to be retained in the glass product for the heated and unheated material,

respectively. Previous melter testing with rhenium at similar target concentrations has demonstrated that these concentrations are suitable for reliable measurements of rhenium concentrations in both the melter exhaust and product glass [20]. The iron oxide slurries were combined with the remaining constituents in the waste simulant, shown in Table 2.2 as well as glass forming additives shown in Table 2.5, and were thoroughly blended before transfer to the melter feed tank. At this juncture, the feed produced with goethite slurries were too viscous to be pumped into the feed tank, as shown in Figures 2.1 and 2.2. Water was added to each of the feeds until they were pumpable, resulting in an absolute increase in water content of about three percent.

2.4 Analysis of Feed Samples

2.4.1 General Properties

Feed samples from each unique feed composition were analyzed to confirm physical properties and chemical composition. Samples were taken at the end of the first four melter tests upon removal of remaining feed from the melter feed tank. Sample names, sampling dates, and measured properties are given in Table 2.7. Samples from similar iron sources have very similar measured pH, density, and glass conversion ratio. As expected, higher water contents and lower glass conversion ratios were measured for feeds formulated with the goethite slurries due to the required additions of water to the feeds using goethite slurries as the iron source. The measured glass conversion ratios were within ten percent of the target; however, the nominal feed was on average three percent above the target value and the feeds with the goethite slurries were on average six percent below the target value. For the purposes of calculating production rates from feed rates, the average measured glass conversion ratios of 0.36 and 0.33 were used for nominal feed and for the feeds from the goethite slurries, respectively.

2.4.2 Rheology

Samples of the melter feeds that were used for these tests were also subjected to rheological characterization. The results from rheological characterization of a variety of other melter feeds and waste simulants, as well as the effects of a range of test variables, are described in detail in a separate report [3]. Rheograms for the melter feeds, which show the feed viscosity versus shear rate, are presented in Figure 2.3; measured values for viscosity at selected shear rates and the yield stress are shown in Table 2.7. The measured range of feed viscosity and yield stress was relatively small compared to feed samples from previous tests, which included feeds that were deliberately adjusted to be more viscous or diluted with water to simulate lower waste solids content [16]. Yield stress values were higher for feeds with the goethite slurries (24-28 Pa vs. 17 Pa) than for the nominal feeds. Viscosity differences were smaller and only apparent at the lowest shear rates. Clearly, the yield stress and viscosity of the material prior to the addition of water to the feed (see Figures 2.1 and 2.2) were considerably higher than those of the feeds processed during the tests. Increases in feed viscosity have previously been observed in HLW melter feeds formulated with boehmite in place of $\text{Al}(\text{OH})_3$ (yield stress of 159 Pa vs. 1 Pa) [21] in a similar fashion to the increases in viscosity for feeds in current tests formulated with goethite

in place of $\text{Fe}(\text{OH})_3$. The goethite and iron hydroxide slurries showed similar pH values as did the boehmite and aluminum hydroxide slurries. Addition of about 2 wt% of water to the goethite slurry resulted in a slurry with viscosity and yield stress values similar to those of the iron hydroxide slurry. Additional work is needed to determine if the cause of the difference in slurry properties with goethite and boehmite is something unique to the chemical form or some other factor such as the particle size. It should be noted that the material referred to as “goethite” was actually about 60-65% magnetite with the remaining being goethite or goethite and iron hydroxide.

2.4.3 Chemical Composition

The chemical compositions of the feed samples were determined by first making a glass from the feed via crucible melt. The glass was subsequently crushed and analyzed directly by XRF. The boron and lithium concentrations were determined by DCP analysis of solutions generated by microwave aided acid dissolution. Data are compared to the target composition in Table 2.8. The results generally corroborate the consistency of the feed composition and show good agreement with the target composition for the major components. Of the oxides with a target concentration of one percent or greater, only the XRF values for iron oxide in the feed produced by NOAH and lithium oxide for feed formulated with goethite slurries had deviations of greater than 10% from target. No oxides with target concentrations of one percent or greater had deviations greater than fifteen percent from target. The absolute deviations for iron and lithium were less than 1.6 and 0.6 wt%, respectively. Rhenium was added to the NOAH feed in the form of perrhenic acid and therefore the surplus iron has no effect on the rhenium concentration. Boron and lithium concentrations measured by DCP were within nine and four percent of the target for feed produced from NOAH, respectively, and within three and fifteen percent of the target for feed constituted from goethite slurry, respectively, validating the use of the target values for normalizing the XRF data. Several oxides targeted at low concentrations in the glass including Ca, Cd, Cr, Mn, P, and S were observed in the feed at concentrations higher than the target, particularly in the feed obtained from NOAH. Similarly, potassium, magnesium, and titanium were measured at low concentrations in feed made by NOAH. The volatile trace element sulfur was measured at concentrations higher than target concentrations suggesting that sulfur is present as a contaminant in the glass forming additives or chemicals used to make the simulant. Given the low target concentrations, these surpluses are not expected to have any significant effect on glass properties or rhenium retention. Rhenium was also measured at low concentrations (0.004 wt%) in feed in which it was not intentionally included, consistent with the analysis of the glass product (see Section 4.1); therefore corrections were made to the measured rhenium concentration of glasses for determination of rhenium retention in the glass and total rhenium balance. In feed samples with rhenium added as perrhenic acid or originating with the goethite slurry, much of the rhenium is volatilized during crucible melting, as expected.

SECTION 3.0 MELTER OPERATIONS

Melter tests were conducted with HLW AZ-101 waste simulants containing various sources of iron and rhenium on the DM10 between 2/28/10 and 3/4/10. These tests produced over 100 kg of glass from almost 300 kg of feed. The tests, ranging from 10 to 24 hours in duration, were divided as follows:

- Pre-Test: Processed nominal AZ-101 feed ($\text{Fe}(\text{OH})_3$ as iron source) with no added rhenium for 9 hours.
- Test 1: Processed nominal AZ-101 feed ($\text{Fe}(\text{OH})_3$ as iron source) with no added rhenium for 24 hours to define rhenium background.
- Test 2: Processed nominal AZ-101 feed ($\text{Fe}(\text{OH})_3$ as iron source) with rhenium added as perrhenic acid targeting 0.05 wt% ReO_2 in the glass product. Duration of 23.25 hours.
- Test 3: Processed AZ-101 feed with heated goethite obtained from PNNL as the iron source. Based on analysis of the material, the rhenium content was targeted at 0.043 wt% ReO_2 in the glass product. Duration was 19.5 hours, determined by the amount of heated goethite material received.
- Test 4: Processed AZ-101 feed with unheated goethite obtained from PNNL as the iron source. Based on analysis of the material, the rhenium content was targeted at 0.042 wt% ReO_2 in the glass product. Duration was 16.25 hours, determined by the amount of unheated goethite material received.
- Test 5: Processed nominal AZ-101 feed ($\text{Fe}(\text{OH})_3$ as iron source) with rhenium added as perrhenic acid targeting 0.05 wt% ReO_2 in the glass product. Duration of 9.75 hours with bubbling increased to give maximum attainable product rate.

Attempts were made to replicate the melter configuration and operating conditions used for previous melter tests with HLW simulants [12-15, 21-24]. These conditions include a near complete cold cap, which is between 80-95% melt surface coverage for the DM10 since a 100% cold cap tends to lead to "bridging" in smaller melters. The bubbling rate was held constant at 1 lpm in all but the last test and the feed rate was adjusted to provide the desired complete cold cap (90-100% of melt surface covered with feed). In Test 5, the bubbling rate was adjusted to obtain the highest attainable production rate. Power was supplied to the electrodes to maintain a glass temperature of 1150°C throughout the tests. All tests targeted the same glass composition (HLW98-95 without U, minor constituents, and RuO_2) with the difference in feed composition being the iron and rhenium sources. This approach permitted the direct comparison of each iron source with respect to rhenium retention in the glass product at constant operating conditions. It also allowed the determination of ease of processing feed with the different sources of iron.

3.1 Melter Operations Data

Production rates, run conditions, and measured melter parameters for the five melter test segments are summarized in Table 3.1. Production rates, bubbling rates, glass temperatures, plenum temperatures, electrode power, and glass resistance are depicted over the course of the tests in Figures 3.1 – 3.4. The average production rates ranged between 850 and 1700 kg/m²/day and increased with the use of the goethite slurry as an iron source and melt pool bubbling. At a constant bubbling rate of 1 lpm, glass production rates increased by 59% from an average of about 950 to 1500 kg/m²/day with the use of the goethite slurry as an iron source. The significant increase in production rate was unexpected and potentially complicates comparisons of rhenium retention between the various feeds; Test 5 was therefore added to provide an additional basis for comparison. The increase in production rate occurred despite the goethite feed having a higher water content, which in previous melter tests has been demonstrated to decrease glass production rates [9, 13, 15, 21-23]. This increase is also about twice the increase observed in DM100 tests at constant bubbling while processing HLW feeds with Al(OH)₃ and boehmite (950 to 1200 kg/m²/day). It is not clear whether the increase in the processing rate with goethite and boehmite as compared to the corresponding hydroxides is unique to the chemical form of the materials, or some other factor such as particle size. Additional work would be useful to investigate this point. As noted above, the material identified as “goethite” was actually about 60-65% magnetite with the remaining being goethite or goethite and iron hydroxide. In tests with high-Al glass formulations, feeds with aluminum oxide as the aluminum source showed the lowest processing rate, feeds with aluminum hydroxide showed higher rates, and feeds with boehmite showed the highest rates [21].

The increase in production rate with increase in melt pool bubbling rate in Test 5 was expected based on numerous melter tests documenting the effect of bubbling on processing rate [8-18, 22, 23]. The glass production rate in this test was closer to that observed in the tests with the goethite feeds, providing an additional basis for comparison.

Occasionally during the tests, dried feed bridged from the sides of the melter, thermowell, and electrodes, necessitating dislodging with a rod through the view port on top of the melter. Feed bridging is commonly observed in small melters and is not judged to be related to the feed formulations that were used in the melter tests; furthermore, this would not be expected to be an issue in larger melters where the walls are farther apart. Glass temperatures (2 and 4 inches from the melt pool floor) averaged within 8°C of the target glass temperatures throughout the tests. The glass temperature 4 inches from the melt floor varied more with the level of glass in the melter and changes in the cold cap than did the temperatures measured lower in the melt pool. The discharge temperature was maintained above 1000°C throughout the tests to prevent the freezing of glass in the chamber during discharge. Test average plenum temperature measurements were between 524 and 610°C in the thermowell and about 30 to 90°C cooler at the exposed thermocouple. This difference is opposite to that in most previous tests, indicating that the exposed thermocouple was partially shielded, or the thermowell was closer to a bubbling outlet. The gas temperature at the film cooler averaged between 266-292°C, as determined by the plenum temperature, the amount of added film cooler air, and the temperature of the added film cooler air. Power supplied to the melter increased from near 4 kW to almost 7 kW with

increasing feed rates. Glass pool resistance decreased by about 0.01 ohm when processing feed formulated with the goethite slurry. A vacuum of about 1 inch of water was maintained on the melter throughout the tests.

SECTION 4.0 GLASS PRODUCTS

Over one hundred kilograms of glass was produced in these tests. The glass was discharged from the melter periodically using an airlift system and collected in custom fabricated square carbon steel cans. The discharged product glass was sampled at the end of each test by removing sufficient glass from the top of the cans for total inorganic analysis. Product glass masses, discharge date, and measured rhenium content are given in Table 4.1. The glass pool samples were obtained by dipping a rod into the glass melt at the end of each test

4.1 Discharge Glasses

Discharge glass samples were crushed and analyzed directly by XRF. The target values for boron and lithium oxides, which are not determined by XRF, were used for normalizing the XRF data to 100 wt%. The XRF analyzed compositions of all discharged glass samples are provided in Table 4.2. The majority of the XRF analysis results compared very favorably to their corresponding target values and also corroborated much of the feed sample analyses (see Section 2.4.3). Of the oxides with a target concentration of one percent or greater, only the XRF values for aluminum, zinc, and zirconium oxides had deviations of greater than 10% from target in glass from Test 1, zinc from Test 2, and zirconium from Test 4. Deviations observed in glass from Test 1 occurred due to a lack of complete melter turnover during the test. Zinc and zirconium deviations in other tests were less than half an absolute weight percent and therefore are not expected to have any significant effect on glass properties. Minor constituent such as calcium, cadmium, chromium, manganese and phosphorous were over-represented in the glass product at about the same frequency and magnitude as in the feed samples (see Section 2.4.3). Potassium, magnesium, and titanium were measured at low concentrations in glasses, particularly in glass produced from feed made by NOAH. The volatile trace element sulfur was measured at concentrations higher than target concentrations while processing feed supplied from NOAH suggesting that sulfur is present as a contaminant in the glass forming additives or chemicals used to make the simulant.

The discharge glass compositions over the course of testing are illustrated in Figures 4.1-4.6. Most oxides approximate their respective target values and varied little during testing after three melt pool turnovers had been completed for each composition. At the beginning of testing, oxides of Na, Al, Fe, Zn, Zr, Cd, Cs, Ce and P increase in concentration at the expense of Si, Cr, B, and Li as the glass pool transitions to the target HLW AZ-101 composition. Many of the oxides such as those of silicon and sodium reach steady state concentrations during testing and vary little over the remainder of the tests. Examples of small differences between glass generated from feeds produced by VSL (Tests 3 and 4) and NOAH (Tests 1, 2, and 5) are higher concentrations of iron and cadmium while processing feed from NOAH and higher concentrations of cerium and cesium while processing feed made at VSL with goethite slurries.

Rhenium was measured at low concentrations (0.002 to 0.005 wt% ReO_2) prior to the intentional introduction in the feed due to trace level feed contamination or residual rhenium from previous melter tests. Upon addition to the feed in Test 2, the concentration in the glass increased to 0.014 wt% ReO_2 through the majority of Test 3 while processing the heated goethite slurry. The concentration of ReO_2 increases again to 0.017 wt% during Test 4 while processing feed with unheated goethite slurry and then again to 0.018 wt% during Test 5 while processing nominal feed containing rhenium added as perrhenic acid. As expected, rhenium concentrations were well below target concentrations as a result of volatilization from the cold cap and glass pool.

4.2 Glass Pool Samples

Glass pool dip samples were obtained at the end of each test to verify the composition of the glass pool, detect any secondary phases on the glass pool surface, and to determine the melt level to quantify the amount of glass in the melt pool. A list of all dip samples including sample names, sampling dates, measured rhenium content, measured iron oxidation state, glass pool depth, and secondary phase observations are given in Table 4.3. There was no visual evidence of secondary phases in any of the dip samples. The analysis of the glass pool samples corroborates the composition of the discharge glasses as shown in Table 4.4. The measured rhenium concentrations in the glass pool samples were the same as, or an absolute 0.001 wt% oxide higher than the discharge glass. No reduced iron was measured in glass from the end of tests processing nominal feed, as expected since all iron in that feed was trivalent. About two percent of the total iron in glass samples from the tests processing feed containing the goethite slurry was determined to be divalent. Approximately two thirds of the iron in the goethite slurry is in the form of magnetite (see Table 2.6), resulting in twenty two percent of the iron in the feed being divalent; therefore, twenty percent of the feed iron was oxidized during the vitrification process.

SECTION 5.0 MONITORED OFF-GAS EMISSIONS

The melter exhaust was sampled for metals/particles according to 40-CFR-60 Methods 3, 5, and 29 at steady-state operating conditions during each test segment. The concentrations of off-gas species that are present as particulates and gaseous species that are collected in impinger solutions were derived from laboratory data on solutions extracted from air samples (filters and various solutions) together with measurements of the volume of air sampled. Particulate collection required isokinetic sampling, which entails removing gas from the exhaust at the same velocity that the air is flowing in the duct (40-CFR-60, Methods 1-5). Typically, a sample size of 30 dscf was taken at a rate of between 0.5 and 0.75 dscfm. Total particulate loading was determined by combining gravimetric analysis of the standard particle filter and chemical analysis of probe rinse solutions. An additional impinger containing 2 N NaOH was added to the sampling train to ensure complete scrubbing of all acid gases. The collected materials were analyzed using DCP-AES for the majority of the constituents and ion chromatography (IC) for anions. Cesium was measured in solutions using atomic absorption. Melter emission fluxes are compared to feed fluxes in Tables 5.1 – 5.6. Notice the distinction that is made between constituents sampled as particles and as "gas". The "gaseous" constituents are operationally defined as those species that are scrubbed in the impinger solutions after the air stream has passed through a 0.3 μ m heated filter. All thirteen samples are within the 90 – 110% limits for isokinetic sampling.

Particulate emissions from the melter constituted 0.43 to 0.75 percent of feed solids during tests with nominal feed and 0.19 to 0.35 of feed solids during tests with feed containing the goethite slurry. The level of carry-over for tests processing nominal feed is mostly within the range measured previously for iron rich, HLW simulants processed on the DM100 [10, 19] (0.57 - 1.47 percent) and on the DM10 [24] (0.39 - 1.29 percent). Conversely, overall solids carryover while processing feed constituted with the goethite slurry was below that measured in previous tests as well as the tests in this study with nominal feed. Much of the difference between nominal and feed with goethite slurry is attributable to iron carryover; 0.67 percent of iron is carried over in tests with the nominal feed as opposed to an average of only 0.05 percent in tests with the goethite slurry. The carryover of solids and most elements was highest during the test with elevated bubbling, as expected. The feed element emitted at the highest rate was clearly rhenium. Calculated sulfur carryover from the melter was also high; however, the measured values were affected by low target feed concentrations and probable low level sulfur contamination in the feeds, particularly the nominal feed (see Section 2.4.3). Other elements exhibiting volatile behavior include chromium, alkali metals, cadmium, and boron. It should be noted that while indicative, melter sampling results from the DM10 have the potential to be biased somewhat by frequent bridging of feed across the melt pool surface and the need to mechanically dislodge the deposits and therefore DM10 results should be verified in future testing on larger melters, as is typically done. Boron and sulfur were the only elements detected in the impinger solutions

collected downstream of the heated particle filter in the sampling train, which constitutes the “gas” fraction of the melter emissions.

SECTION 6.0

MASS BALANCE FOR RHENIUM IN GLASS AND EMISSIONS

The principal goal of this work was to determine a mass balance for rhenium while processing feed containing goethite slurries used to remove rhenium from solution for comparison with that for the baseline feed. During tests processing feeds with the different iron and rhenium sources, sufficient glass and melter exhaust samples were taken and analyzed to complete a rhenium mass balance for each feed and operating condition. A complete rhenium mass balance for the tests is given in Table 6.1. Two to three times more rhenium was detected in the melter exhaust as compared to the glass for each of the feed compositions and operating conditions tested. The total rhenium recoveries ranged from 79 to 132 percent depending on the method used for determining the amount of rhenium in the feed and for assessing the background amount of rhenium in the feed and melter system.

Total mass balance closure is not only dependent on the amounts measured in the glass product and melter exhaust but also the amount of rhenium in the feed and the amount of rhenium contamination in the melter system or in the feed streams. Initial rhenium mass balance calculations (see Table 3.1) were based on the PNNL analysis (see Table 2.6) in the tests with the goethite slurry and the background rhenium concentration measured in glass discharged from the DM10 while feeding the target glass composition with no added rhenium. Subsequent analysis of melter feed samples suggests that the rhenium observed in the glasses discharged during the initial test originated as contamination in the feed provided by NOAH used in Tests 1, 2, and 5 rather than as residual rhenium remaining in the melter. Also, VSL analysis of the goethite slurry indicated that the rhenium concentrations were 25 and 36 percent higher than indicated by the PNNL analysis. The effect of the assumptions used for the feed rhenium concentration and the background corrections are evaluated in Table 6.1. Also evaluated is the exclusion of an outlier rhenium emission result for Test 3. Three samples were taken showing rhenium emission rates as 7.73 mg/min, 7.00 mg/min, and 4.47 mg/min (see Table 5.3); average percent feed emitted was calculated with and without the low outlier. The total rhenium recoveries for samples using the PNNL analysis of the goethite slurries are all above 100%, and 119% or greater when omitting the low emission outlier from Test 3. Correcting the glass concentrations by subtracting the rhenium measured during Test 1 reduces the percentage retention in the glass by an absolute 6 to 8 percent. Exclusion of the low emission outlier from Test 3 results in an absolute increase in the amount of rhenium measured in the emissions and total recovery of 14 percent.

The effects of using either of the two feed sample analyses taken together with the calculated total recoveries suggest that the best approach for tests processing feed including the goethite slurries is the use of the VSL rhenium analysis without background correction of the discharged glass. The background correction should be applied to the tests with the NOAH feed since the feed samples indicate that the origin of the rhenium background appears to be trace level contamination of the NOAH feed.

Overall, the amount of rhenium retained in the glass for the tests varied within a relatively narrow range, indicating that the iron and rhenium sources do not have a large effect on rhenium retention in the glass product. Direct comparisons are complicated by the fact that the feeds made using goethite processed at a significantly higher rate than the baseline feed under the same conditions. Nevertheless, the rhenium retention values are generally bounded by those for the baseline feeds at (i) lower production rate and the same bubbling rate (Test 1) and (ii) approximately the same production rate but higher bubbling rate (Test 5). There appears to be no significant difference between the heated and unheated goethite slurries with respect to rhenium retention in the glass product.

SECTION 7.0 SUMMARY AND CONCLUSIONS

A series of melter tests were conducted on the DM10 using an AZ-101 HLW composition to evaluate the effect of the iron and rhenium source on rhenium retention in the glass product. The tests employed melter feeds with four different iron and rhenium sources: the baseline iron hydroxide slurry without rhenium, the baseline iron hydroxide slurry with rhenium added as perrhenic acid, PNNL magnetite/goethite slurry with adsorbed rhenium, and PNNL heated magnetite/goethite slurry with adsorbed rhenium. In four of the five tests, the bubbling rate was set at 1 lpm and the feed rate adjusted to provide the desired complete cold cap (90-100% of melt surface covered with feed) and target plenum temperature of 550 – 650°C for each of the four feed types. One test using the baseline iron hydroxide slurry with rhenium added as perrhenic acid was conducted at a higher bubbling rate targeting the highest achievable production rate. Measurements of glass production rates, melter operating conditions (temperatures, pressures, power, flows, etc.) were made throughout the tests. Qualitative observations of the cold cap and feed characteristics such as pumpability and spreading across the melt surface as well as ease of discharging glass were made throughout the tests. In addition, particulate loading and composition as well as acid gas concentrations were determined for the purposes of calculating a material mass balance around the melter during each test. Glass samples taken throughout the tests from the melt pool and the air-lift discharge were visually examined for secondary phases and analyzed for chemical composition. Particular emphasis was placed on rhenium distribution between the glass and the off-gas.

The melter tests resulted in the production of over 100 kg of glass from feeds with iron hydroxide and goethite slurries. Notable observations made during the processing of the different feeds were as follows:

- Feed with goethite slurries as the iron source form a significantly more viscous melter feed. Dilution of the feed with additional water was required to produce a melter feed from the goethite slurries that can be processed. However, surprisingly little added water was required.
- Feed made with goethite slurries as the iron source processed about 60% faster than feed using iron hydroxide as an iron source when processed at a constant bubbling rate.
- The retention of rhenium in the glass product was essentially the same for feeds with iron hydroxide and perrhenic acid, heated goethite slurries with rhenium, and unheated goethite slurries with rhenium.
- The majority of the divalent iron present as magnetite in the goethite slurry was oxidized during the vitrification process.
- Solids carryover from the melter for feed with goethite slurries as the iron source was about half that measured for feed with iron hydroxide as the iron source.

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Table 2.1. Analyzed Compositions of AZ-101 Envelope D Waste.

Analyte	AZ-101 Solid (µg/g dry solid)	Oxide [#]	Analyzed AZ-101 Solid (wt%)
Ag	902	Ag ₂ O	0.12%
Al	99873	Al ₂ O ₃	23.71%
As	—	As ₂ O ₃	—
B	91	B ₂ O ₃	0.04%
Ba	1510	BaO	0.21%
Be	26	BeO	0.01%
Bi	150	Bi ₂ O ₃	0.02%
Ca	7505	CaO	1.32%
Cd	14500	CdO	2.08%
Ce	5240	Ce ₂ O ₃	0.77%
Cl	703	Cl	0.09%
Co	128	CoO	0.02%
Cr	2285	Cr ₂ O ₃	0.42%
Cs	—	Cs ₂ O	—
Cu	584	CuO	0.09%
F	390	F	0.05%
Fe	202384	Fe ₂ O ₃	36.35%
K	2000	K ₂ O	0.30%
La	5808	La ₂ O ₃	0.86%
Li	115	Li ₂ O	0.03%
Mg	1540	MgO	0.32%
Mn	5364	MnO ₂	1.07%
Mo	67	MoO ₃	0.01%
Na	54545	Na ₂ O	9.24%
Nd	4290	Nd ₂ O ₃	0.63%
Ni	9992	NiO	1.60%
P	4505	P ₂ O ₅	1.30%
Pb	1728	PbO	0.23%
Pd	2300	PdO	0.33%
Rh	513	Rh ₂ O ₃	0.08%
Ru	1600	Ru ₂ O ₃	0.25%
SO ₄	2410	SO ₃	0.25%
Sb	—	Sb ₂ O ₅	—
Se	—	SeO ₂	—
Si	13055	SiO ₂	3.51%
Sn	3600	SnO ₂	0.37%
Sr	3412	SrO	0.51%
Te	—	TeO ₂	—
Ti	178	TiO ₂	0.04%
U	18500	UO ₂	2.64%
Y	385	Y ₂ O ₃	0.06%
Zn	278	ZnO	0.04%
Zr	65050	ZrO ₂	11.05%
TOTAL	537186	TOTAL	100.0%

- Empty data field. [#] Oxide forms listed are those provided by the WTP Project

Table 2.2. Compositions of the AZ-101 Waste (Oxide Basis) and the HLW Waste Simulant to Produce 100 kg of Waste Oxides (21.5 wt% total solids).

AZ-101 HLW Waste Composition		AZ-101 HLW Waste Simulant	
Waste Oxide	Wt%	Starting Material	Target Weight (kg) *
Al ₂ O ₃	24.62%	Al(OH) ₃	37.995
CaO	1.40%	CaO	1.429
CdO	2.16%	CdO	2.184
Ce ₂ O ₃	0.80%	CeO ₂	0.847
Cr ₂ O ₃	0.46%	Cr ₂ O ₃	0.469
Cs ₂ O	0.50%	CsOH (50% solution)	1.064
Fe ₂ O ₃	37.73%	Fe(OH) ₃ (13% slurry)	372.255
La ₂ O ₃	0.89%	La ₂ O ₃	0.899
MnO	0.91%	MnO ₂	1.127
Na ₂ O	10.60%	NaOH	10.402
Nd ₂ O ₃	0.65%	Nd ₂ O ₃	0.657
NiO	1.66%	Ni(OH) ₂	2.135
P ₂ O ₅	1.34%	FePO ₄ ·xH ₂ O	3.560
SO ₃	0.38%	Na ₂ SO ₄	0.682
SiO ₂	3.78%	SiO ₂	3.808
SnO ₂	0.66%	SnO ₂	0.667
ZrO ₂	11.46%	Zr(OH) ₄ ·xH ₂ O	29.565
Carbonate	1.20 [#]	Na ₂ CO ₃	2.130
Nitrite	0.50 [#]	NaNO ₂	0.769
Nitrate	2.00 [#]	NaNO ₃	2.459
Organic Carbon	0.05 [#]	H ₂ C ₂ O ₄ ·2H ₂ O	0.264
		Water	155.610
Oxide Total	100.00%	TOTAL	630.977

* Target weights adjusted for assay information of starting materials.

[#] Unit for volatile components is g/100 g of waste oxide.

Table 2.3. Composition of Nominal AZ-101 HLW Glass Formulation (wt%).

	AZ-101 Waste			Glass Composition		
Oxide #	Actual Blended Waste [4, 5]	Next Generation Melter Tests [7]	Goethite Melter Tests	Crucible Melt [5]	Next Generation Melter Tests [7]	Goethite Melter Tests
Ag ₂ O	0.12%	-	-	0.04%	-	-
Al ₂ O ₃	23.06%	24.58%	24.62%	7.32%	7.80%	7.81%
B ₂ O ₃	0.43%	-	-	10.64%	10.50%	10.50%
BaO	0.21%	-	-	0.07%	-	-
BeO	0.01%	-	-	0.00%	-	-
Bi ₂ O ₃	0.02%	-	-	0.01%	-	-
CaO	1.34%	1.40%	1.40%	0.43%	0.44%	0.44%
CdO	2.02%	2.16%	2.16%	0.64%	0.69%	0.69%
Ce ₂ O ₃	0.75%	0.80%	0.80%	0.24%	0.25%	0.25%
Cl	0.18%	-	-	0.06%	-	-
Cr ₂ O ₃	0.45%	0.46%	0.46%	0.14%	0.15%	0.15%
Cs ₂ O	0.00%	0.50%	0.50%	0.00%	0.16%	0.16%
CuO	0.09%	-	-	0.03%	-	-
F	0.05%	-	-	0.02%	-	-
Fe ₂ O ₃	35.31%	37.67%	37.73%	11.21%	11.96%	11.98%
K ₂ O	0.43%	-	-	0.14%	-	-
La ₂ O ₃	0.83%	0.89%	0.89%	0.26%	0.28%	0.28%
Li ₂ O	0.03%	-	-	3.76%	3.75%	3.75%
MgO	0.31%	-	-	0.10%	-	-
MnO ₂	1.04%	0.91%	0.91%	0.33%	0.29%	0.29%
MoO ₃	0.01%	-	-	0.00%	-	-
Na ₂ O	10.80%	10.58%	10.60%	11.93%	11.86%	11.87%
Nd ₂ O ₃	0.61%	0.65%	0.65%	0.19%	0.21%	0.21%
NiO	1.55%	1.66%	1.66%	0.49%	0.53%	0.53%
P ₂ O ₅	1.26%	1.34%	1.34%	0.40%	0.43%	0.43%
PbO	0.23%	-	-	0.07%	-	-
PdO	0.32%	-	-	0.10%	-	-
Rh ₂ O ₃	0.08%	-	-	0.02%	-	-
RuO ₂	0.24%	0.15%	-	0.08%	0.05%	-
SO ₃	0.37%	0.38%	0.38%	0.12%	0.12%	0.12%
SiO ₂	3.80%	3.77%	3.78%	44.71%	44.70%	44.70%
SnO ₂	-	0.66%	0.66%	-	0.21%	0.21%
SrO	0.49%	-	-	0.16%	-	-
TiO ₂	0.04%	-	-	0.01%	-	-
UO ₂	2.76%	-	-	0.88%	-	-
ZnO	0.04%	-	-	2.01%	2.00%	2.00%
ZrO ₂	10.72%	11.44%	11.46%	3.40%	3.63%	3.64%
TOTAL	100.00%	100.00%	100.00%	100.00%	100.00%	100.00%

- Empty data field. # Oxide forms listed are those employed by the WTP Project

Table 2.4. Summary of AZ-101 HLW Characterization Data for Crucible Glass.

Glass ID		HLW98-95
Oxides from AZ-101 Solids		30.69%
Oxides from Cs Pretreatment		0.81%
Oxides from Tc Pretreatment		0.25%
TOTAL Waste Loading		31.75%
Melt Temperature		1150 °C
Crucible Used		Pt/Au
Visual Observation of As-Melted Glass		Dark brown homogeneous translucent glass
SEM/EDS Observations after Heat Treatment at 950 °C for 70 hours (Premelt 1200 °C/1 hour)		Spinel crystals found concentrated at the bottom of the crucible. Rh and Ru incorporated into spinel crystals. 0.5 vol % of crystals
Meets Liquidus Temp. Requirement?		Yes
Viscosity (P) at	1000 °C	226
	1050 °C	144
	1100 °C	85.9
	1150 °C	54.1
	1200 °C	35.6
Conductivity (S/cm) at	1000 °C	0.203
	1050 °C	0.254
	1100 °C	0.308
	1150 °C	0.367
	1200 °C	0.428
7-Day PCT Normalized Concentration (g/l)	B	0.554 (DWPF-EA – 16.83)
	Li	0.585 (DWPF-EA – 9.51)
	Na	0.461 (DWPF-EA – 11.59)
	Si	0.317 (DWPF-EA – 3.53)
7-Day PCT Normalized Leach Rate (g/(m ² -d))	B	0.0395 (DWPF-EA – 1.20)
	Li	0.0418 (DWPF-EA – 0.68)
	Na	0.0329 (DWPF-EA – 0.83)
	Si	0.0226 (DWPF-EA – 0.25)
pH after 7 days of PCT Leaching		10.34
TCLP Data and Associated UTS Limits (ppm)	Ag	<0.003 (UTS – 0.14)
	Ba	<0.4 (UTS – 21.00)
	Cd	0.10 (UTS – 0.11)
	Cr	0.01 (UTS – 0.60)
	Ni	0.07 (UTS – 11.00)
	Pb	<0.02 (UTS – 0.75)
Glass Transition Temperature (°C)		463
Density (g/ml)		2.713

Table 2.5. Compositions of the Nominal AZ-101 HLW Glass Formulation and the Glass Former Mix to Produce 100 g of AZ-101 HLW Glass.

Oxide #	AZ-101 Blended Waste (wt%)	Oxides from Glass Formers (as wt% in Glass)	Nominal AZ-101 Glass Composition (wt%)	Glass Forming Additives	Typical Assay	Amount (g) to Produce 100 g of Glass
Al ₂ O ₃	24.58%	-	7.81%	-	-	-
B ₂ O ₃	-	10.50%	10.50%	Na ₂ B ₄ O ₇ ·10H ₂ O	0.99	29.049
CaO	1.40%	-	0.44%	-	-	-
CdO	2.16%	-	0.69%	-	-	-
Ce ₂ O ₃	0.80%	-	0.25%	-	-	-
Cr ₂ O ₃	0.46%	-	0.15%	-	-	-
Cs ₂ O	0.50%	-	0.16%	-	-	-
Fe ₂ O ₃	37.67%	-	11.98%	-	-	-
La ₂ O ₃	0.89%	-	0.28%	-	-	-
Li ₂ O	-	3.75%	3.75%	Li ₂ CO ₃	0.975	9.512
MnO ₂	0.91%	-	0.29%	-	-	-
Na ₂ O	10.58%	8.50%	11.87%	Na ₂ CO ₃	0.99	6.609
Nd ₂ O ₃	0.65%	-	0.21%	-	-	-
NiO	1.66%	-	0.53%	-	-	-
P ₂ O ₅	1.34%	-	0.43%	-	-	-
RuO ₂	0.15%	-	0.05%	-	-	-
SO ₃	0.38%	-	0.12%	-	-	-
SiO ₂	3.77%	43.50%	44.70%	SiO ₂	0.99	43.939
SnO ₂	0.66%		0.21%			
ZnO	-	2.00%	2.00%	ZnO	0.99	2.020
ZrO ₂	11.44%	-	3.64%	-	-	-
TOTAL	100.00%	68.25%	100.00%	-	TOTAL	91.130

Oxide forms listed are those provided by the WTP Project

- Empty data field

Table 2.6. Characteristics of PNNL Iron-Rhenium Slurries for Melter Testing.

–		Heated Material	Unheated Material
Amount of slurry available for melter and small scale testing		28.0 kg	25.2 kg
PNNL	Measured solids content	13.0 wt%, 11.84 wt%	11.0 wt%
	Measured Density	1.09 g/ml	1.09 g/ml
	Measured Re content	3.044 mg/g	2.812 mg/g
	Approximate pH	8.5	8.5
	Estimated iron speciation	65-70% magnetite, 30-35% goethite	65-70% magnetite, 30-35% goethite and iron hydroxide
VSL	Measured solids content	10.83 wt%	11.14 wt%
	XRD measured mineral speciation*	59% magnetite, 41% goethite	66% magnetite, 34% goethite
	XRF Measured Composition [#]	Al ₂ O ₃	0.83 wt%
		CaO	0.46 wt%
		Cl	0.26 wt%
		Cr ₂ O ₃	0.03 wt%
		Fe ₂ O ₃	97.5 wt%
		MnO	0.05 wt%
		ReO ₂	0.46 wt%
	DCP Measured Composition [#]	Al ₂ O ₃	0.95 wt%
		CaO	0.47 wt%
		Cl	NA
		Cr ₂ O ₃	0.03 wt%
		Fe ₂ O ₃	97.4 wt%
		MnO	0.05 wt%
		ReO ₂	0.45 wt%
		MgO	0.07 wt%
		Na ₂ O	0.41 wt%
		SiO ₂	0.05 wt%

* Does not include amorphous iron phases; data obtained by Reitveld refinement of XRD patterns.

[#] Oxidation states not measured.

NA – Not Analyzed

Table 2.7. Characteristics of Melter Feed Samples from DM10 Goethite/Rhenium Tests.

Test	Iron Source	Date	Name	% Water	pH	Density (g/ml)	Glass Yield				Yield Stress	Viscosity (Poise)		
							Measured		Target	% Dev		@1/s	@10/s	@100/s
							(g/l)	(kg/kg)	(kg/kg)					
1	Fe(OH) ₃	3/1/2011	ENM-F-15A	57.88	11.32	1.37	492	0.359	0.350	2.69	17.6	67.3	12.54	1.77
2	Fe(OH) ₃	3/2/2011	ENM-F-21A	58.15	11.20	1.36	491	0.361	0.350	3.14	17.1	68.7	15.23	2.16
3	Heated goethite	3/3/2011	ENM-F-36A	60.62	10.84	1.34	453	0.333	0.350	-4.80	24.3	82.6	13.96	1.72
4	Unheated goethite	3/4/2011	ENM-F-42A	61.42	10.90	1.32	430	0.325	0.350	-7.03	28.8	87.9	19.62	2.15

Table 2.8. XRF Analyzed Compositions for Vitrified Melter Feed Samples from DM10 Tests (wt%).

Test	1			2			3			4	
Sample	Target	ENM-F-15A	% Dev.	Target	ENM-F-21A	% Dev	Target	ENM-F-36A	% Dev	ENM-F-42A	% Dev
Al ₂ O ₃	7.81	7.19	-7.87	7.81	7.15	-8.40	7.81	7.64	-2.13	7.08	-9.35
B ₂ O ₃	10.50	9.68*	-7.80	10.49	9.58*	-8.71	10.49	10.66*	1.58	10.75*	2.44
CaO	0.44	1.38	NC	0.44	1.42	NC	0.44	0.55	NC	0.57	NC
CdO	0.69	0.92	NC	0.69	0.92	NC	0.69	0.63	NC	0.68	NC
Ce ₂ O ₃	0.25	0.25	NC	0.25	0.26	NC	0.25	0.29	NC	0.30	NC
Cr ₂ O ₃	0.15	0.25	NC	0.15	0.24	NC	0.15	0.18	NC	0.19	NC
Cs ₂ O	0.16	0.13	NC	0.16	0.12	NC	0.16	0.16	NC	0.19	NC
Fe ₂ O ₃	11.98	13.45	12.26	11.97	13.57	13.33	11.97	11.74	-1.93	12.97	8.33
K ₂ O	§	0.54	NC	§	0.53	NC	§	<0.01	NC	<0.01	NC
La ₂ O ₃	0.28	0.12	NC	0.28	0.13	NC	0.28	0.22	NC	0.22	NC
Li ₂ O	3.75	3.60*	-3.99	3.75	3.67*	-2.07	3.75	4.29*	14.46	4.21*	12.33
MgO	§	0.18	NC	0.00	0.17	NC	0.00	<0.01	NC	<0.01	NC
MnO	0.29	0.74	NC	0.29	0.81	NC	0.29	0.31	NC	0.32	NC
Na ₂ O	11.87	11.78	-0.79	11.86	11.80	-0.50	11.86	12.47	5.09	11.98	0.96
Nd ₂ O ₃	0.21	0.22	NC	0.21	0.22	NC	0.21	0.18	NC	0.17	NC
NiO	0.53	0.61	NC	0.53	0.61	NC	0.53	0.54	NC	0.54	NC
P ₂ O ₅	0.43	0.71	NC	0.43	0.73	NC	0.43	0.48	NC	0.45	NC
ReO ₂	§	0.004	NC	0.05	0.032	NC	0.043	0.033	NC	0.035	NC
SiO ₂	44.70	42.38	-5.18	44.67	42.04	-5.88	44.68	44.06	-1.38	43.53	-2.57
SnO ₂	0.21	0.23	NC	0.21	0.22	NC	0.21	0.19	NC	0.21	NC
SO ₃	0.12	0.27	NC	0.12	0.26	NC	0.12	0.09	NC	0.16	NC
TiO ₂	§	0.04	NC	§	0.04	NC	§	<0.01	NC	<0.01	NC
ZnO	2.00	1.81	-9.29	2.00	1.86	-7.15	2.00	1.98	-1.10	2.05	2.77
ZrO ₂	3.64	3.52	-3.39	3.64	3.61	-0.76	3.64	3.30	-9.34	3.38	-6.98
Sum	100.00	100.00	NC	100.00	100.00	NC	100.00	100.00	NC	100.00	NC

* DCP-AES analyzed results.

NC – Not calculated

Table 3.1. Summary of DM10 Melter Tests.

Test		1	2	3
	Feeding Interval	2/28/11 19:00 – 3/1/11 19:00	3/1/11 19:45 – 3/2/11 19:00	3/2/11 21:18 – 3/3/11 16:52
	Total	24 hr	23 hr 15 min	19 hr 34 min
	Interruptions	6 min	0 min	10 min
Feed	Origin	NOAH	NOAH + perhenic acid	VSL+ heated goethite
	Glass Yield (kg/kg)	0.36	0.36	0.33
	Rhenium glass target	0	0.05	0.043
	Processed (kg)	62.57	47.80	81.715
	Processing rate (kg/hr)	2.61	2.06	4.15
Glass	Produced from feed (kg)	22.52	17.21	26.97
	Discharged (kg)	18.55	15.12	24.48
	Test Average Production Rate (kg/m ² /day)*	1073	846	1564
	Measured ReO ₂ wt%	0.0035	0.014	0.015
	Background Corrected ReO ₂ wt%	0.0035	0.0105	0.0115
	% Feed Rhenium	NA	21	27
Emissions	Average Rhenium Emission Rate (mg/min)	< 0.10	2.92	6.40
	% Feed Rhenium	NA	58	78
Total Rhenium Recovery (%)		NA	79	105
Test Average Glass Temperature (°C)	2" from floor	1150	1152	1152
	4" from floor	1143	1146	1142
Test Average Plenum Temperature (°C)	Thermowell	538	556	577
	Exposed	493	510	544
Test Average Melt Pool Bubbling (lpm)		1.0	1.0	1.0
Test Average Discharge Chamber Temperature (°C)		1031	1035	1040
Test Average Film Cooler Exhaust Outlet Temperature (°C)		266	270	288
Test Average Melter Pressure (inches water)		-0.90	-0.87	-0.86
Test Average Electrical Properties	Voltage (volts)	27.1	25.9	31.1
	Current (amps)	169	158	207
	Power (kW)	4.59	4.11	6.49
	Glass Pool Resistance (ohms)	0.161	0.164	0.151

* - Calculated from total feed processed

NA – Not Applicable

Table 3.1. Summary of DM10 Melter Tests (continued).

Test		4	5
	Feeding Interval	3/3/11 17:45 – 3/4/11 10:00	3/4/11 10:45 – 3/4/11 20:30
	Total	16 hr 15 min	9 hr 45 min
	Interruptions	0 min	0 min
Feed	Origin	VSL+ goethite	NOAH + perrhenic acid
	Glass Yield (kg/kg)	0.33	0.36
	Rhenium glass target	0.042	0.05
	Processed (kg)	63.915	40.46
	Processing rate (kg/hr)	3.93	4.20
Glass	Produced from feed (kg)	21.09	14.57
	Discharged (kg)	19.08	14.10
	Test Average Production Rate (kg/m ² /day)*	1483	1699
	Measured ReO ₂ wt%	0.017	0.018
	Background Corrected ReO ₂ wt%	0.0135	0.0145
	% Feed Rhenium	32	29
Emissions	Rhenium Emission Rate (mg/min)	7.12	7.66
	% Feed Rhenium	92	76
Total Rhenium Recovery (%)		124	105
Test Average Glass Temperature (°C)	2" from floor	1152	1152
	4" from floor	1144	1148
Test Average Plenum Temperature (°C)	Thermowell	610	524
	Exposed	524	479
Test Average Melt Pool Bubbling (lpm)		1.0	3.2
Test Average Discharge Chamber Temperature (°C)		1039	1035
Test Average Film Cooler Exhaust Outlet Temperature (°C)		292	282
Test Average Melter Pressure (inches water)		-0.92	-0.98
Test Average Electrical Properties	Voltage (volts)	31.3	30.0
	Current (amps)	215	186
	Power (kW)	6.80	5.65
	Glass Pool Resistance (ohms)	0.146	0.161

* - Calculated from total feed processed

Table 4.1. Listing of Glasses, Masses, Cumulative Masses, and Rhenium Concentrations from DM10 Tests.

Test	Iron/Rhenium Source	Date	Name	Mass (kg)	Cumulative Mass (kg)	ReO ₂ (wt%)
Pre Test	Baseline AZ-101 Feed, No Rhenium	2/28/11	DNM-G-147A	-	-	-
			DNM-G-147B	1.12	1.12	0.004
			DNM-G-149A	2.10	3.22	0.005
			DNM-G-149B	-	-	-
			DNM-G-151A	-	-	-
1		DNM-G-151B	4.36	7.58	0.004	
		DNM-G-151C	-	-	-	
		DNM-G-151D	3.22	10.80	0.004	
		3/01/11	ENM-G-9A	-	-	-
			ENM-G-9B	3.46	14.26	0.004
			ENM-G-9C	-	-	-
			ENM-G-9D	2.04	16.30	0.002
			ENM-G-10A	1.62	17.92	0.003
			ENM-G-10B	2.24	20.16	0.003
			ENM-G-10C	-	-	-
			ENM-G-13A	2.84	23.00	0.003
			ENM-G-13B	-	-	-
			ENM-G-13C	2.50	25.50	0.003
			ENM-G-13D	-	-	-
			ENM-G-13E	2.44	27.94	0.004
2	3/02/11		ENM-G-15A	1.88	29.82	0.007
		ENM-G-16A	2.00	31.82	0.010	
		ENM-G-16B	1.82	33.64	0.014	
		ENM-G-16C	2.10	35.74	0.013	
		ENM-G-16D	1.86	37.60	0.013	
		ENM-G-17A	1.82	39.42	0.014	
		ENM-G-17B	1.66	41.08	0.014	
		ENM-G-17C	1.98	43.06	0.014	
3	3/03/11	ENM-G-21A	2.58	45.64	0.014	
		ENM-G-26A	2.28	47.92	0.014	
		ENM-G-26B	2.38	50.30	0.014	
		ENM-G-29A	2.26	52.56	0.014	
		ENM-G-29B	1.86	54.42	0.014	
		ENM-G-29C	2.42	56.84	0.014	
		ENM-G-29D	1.92	58.76	0.014	
		ENM-G-29E	1.98	60.74	0.014	
		ENM-G-31A	2.42	63.16	0.014	
		ENM-G-31B	1.86	65.02	0.015	
4	Unheated Goethite	ENM-G-36A	2.52	67.54	0.018	
		ENM-G-36B	2.02	69.56	0.017	
		ENM-G-37A	2.34	71.90	0.017	
		ENM-G-37B	1.94	73.84	0.017	

- Empty data field

Table 4.1. Listing of Glasses, Masses, Cumulative Masses, and Rhenium Concentrations from DM10 Tests (continued).

Test	Iron/Rhenium Source	Date	Name	Mass (kg)	Cumulative Mass (kg)	ReO ₂ (wt%)
4	Unheated Goethite	3/04/11	ENM-G-37C	1.96	75.80	0.016
			ENM-G-41A	1.76	77.56	0.017
			ENM-G-41B	1.78	79.34	0.015
			ENM-G-41C	2.30	81.64	0.015
			ENM-G-41D	2.14	83.78	0.017
			ENM-G-42A	2.84	86.62	0.017
5	Baseline AZ-101 Feed, Perrhenic Acid		ENM-G-44A	2.74	89.36	0.017
			ENM-G-44B	2.02	91.38	0.019
			ENM-G-45A	2.08	93.46	0.018
			ENM-G-45B	1.92	95.38	0.019
			ENM-G-49A	1.98	97.36	0.018
			ENM-G-49B	1.92	99.28	0.017
			ENM-G-49C	1.44	100.72	0.018

Table 4.2. XRF Analyzed Compositions for Glass Discharged During DM10 Tests (wt%).

Test	Target	PreTest		1							
Mass (kg)		1.12	3.22	7.58	10.80	14.26	16.30	17.92	20.16	23.00	25.50
Sample		DNM-G-147B	DNM-G-149A	DNM-G-151B	DNM-G-151D	ENM-G-9B	ENM-G-9D	ENM-G-10A	ENM-G-10B	ENM-G-13A	ENM-G-13C
Al ₂ O ₃	7.81	2.88	2.71	4.32	5.14	5.87	6.05	6.34	6.49	6.56	6.90
B ₂ O ₃ *	10.50	15.64**	14.67	13.19	12.45	11.88	11.63	11.46	11.26	11.08	10.95
CaO	0.44	0.84	0.84	1.07	1.13	1.26	1.26	1.30	1.30	1.36	1.36
CdO	0.69	0.16	0.15	0.51	0.59	0.73	0.56	0.70	0.72	0.80	0.79
Ce ₂ O ₃	0.25	0.04	0.02	0.09	0.14	0.17	0.17	0.18	0.21	0.20	0.23
Cr ₂ O ₃	0.15	0.43	0.43	0.40	0.35	0.32	0.29	0.28	0.28	0.28	0.26
Cs ₂ O	0.16	0.03	0.02	0.07	0.08	0.11	0.07	0.09	0.10	0.11	0.11
Fe ₂ O ₃	11.98	3.53	2.90	7.23	8.62	10.65	10.49	11.21	11.49	12.07	12.19
K ₂ O	§	0.75	0.77	0.68	0.63	0.59	0.60	0.60	0.58	0.60	0.61
La ₂ O ₃	0.28	0.02	0.01	0.06	0.08	0.10	0.11	0.10	0.11	0.12	0.12
Li ₂ O*	3.75	6.99**	6.38	5.45	4.98	4.62	4.46	4.35	4.23	4.11	4.03
MgO	§	0.28	0.30	0.25	0.25	0.18	0.20	0.22	0.23	0.24	0.20
MnO	0.29	0.16	0.12	0.42	0.52	0.66	0.65	0.70	0.71	0.75	0.75
Na ₂ O	11.87	5.38	5.01	7.19	8.36	9.06	9.59	9.34	10.23	10.07	10.57
Nd ₂ O ₃	0.21	0.04	0.03	0.10	0.12	0.16	0.17	0.17	0.18	0.20	0.19
NiO	0.53	0.40	0.37	0.52	0.53	0.57	0.53	0.55	0.56	0.56	0.55
P ₂ O ₅	0.43	0.17	0.15	0.36	0.46	0.58	0.59	0.61	0.62	0.63	0.64
ReO ₂	§	0.004	0.005	0.004	0.004	0.004	0.002	0.003	0.003	0.003	0.003
SiO ₂	44.70	59.87	62.89	54.22	51.34	47.46	48.01	46.81	45.54	44.78	44.15
SnO ₂	0.21	0.04	0.04	0.12	0.15	0.17	0.14	0.18	0.17	0.20	0.19
SO ₃	0.12	0.11	0.10	0.16	0.16	0.18	0.17	0.18	0.19	0.20	0.16
TiO ₂	§	0.19	0.21	0.15	0.12	0.09	0.08	0.08	0.07	0.07	0.06
ZnO	2.00	0.89	0.83	1.26	1.35	1.57	1.48	1.57	1.60	1.70	1.68
ZrO ₂	3.64	1.14	1.05	2.17	2.44	2.99	2.69	2.98	3.10	3.31	3.29
Sum	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00

* Target values calculated based on simple well stirred tank model using the DCP-AES analyzed results for the first discharged glass during Test 1.

§ - Not a target constituent

NC – Not calculated

** DCP-AES analyzed results.

**Table 4.2. XRF Analyzed Compositions for Glass Discharged During DM10 Tests (wt%),
(Continued).**

Test	1			2							
Mass (kg)	Target	27.94		Target	29.82	31.82	33.64	35.74	37.60	39.42	41.08
Sample		ENM-G-13E	% Dev		ENM-G-15A	ENM-G-16A	ENM-G-16B	ENM-G-16C	ENM-G-16D	ENM-G-17A	ENM-G-17B
Al ₂ O ₃	7.81	6.93	-11.30	7.81	6.95	6.96	6.97	7.12	7.21	7.10	7.26
B ₂ O ₃ *	10.50	10.85	3.35	10.49	10.79	10.73	10.69	10.66	10.63	10.61	10.59
CaO	0.44	1.36	NC	0.44	1.36	1.38	1.42	1.38	1.38	1.41	1.38
CdO	0.69	0.79	NC	0.69	0.86	0.81	0.83	0.76	0.83	0.85	0.78
Ce ₂ O ₃	0.25	0.21	NC	0.25	0.23	0.24	0.24	0.24	0.23	0.24	0.24
Cr ₂ O ₃	0.15	0.24	NC	0.15	0.24	0.24	0.24	0.23	0.21	0.21	0.21
Cs ₂ O	0.16	0.11	NC	0.16	0.11	0.10	0.11	0.09	0.11	0.11	0.09
Fe ₂ O ₃	11.98	12.23	2.11	11.97	12.44	12.83	13.21	12.74	12.48	12.82	12.64
K ₂ O	§	0.56	NC	§	0.52	0.55	0.55	0.55	0.56	0.56	0.54
La ₂ O ₃	0.28	0.12	NC	0.28	0.11	0.14	0.14	0.13	0.14	0.14	0.15
Li ₂ O*	3.75	3.97	5.91	3.75	3.93	3.90	3.87	3.85	3.83	3.82	3.81
MgO	§	0.19	NC	§	0.23	0.19	0.20	0.17	0.19	0.18	0.18
MnO	0.29	0.75	NC	0.29	0.77	0.80	0.83	0.81	0.79	0.82	0.80
Na ₂ O	11.87	11.06	-6.84	11.86	10.97	10.94	10.60	11.30	11.51	11.37	11.30
Nd ₂ O ₃	0.21	0.20	NC	0.21	0.21	0.21	0.22	0.22	0.21	0.21	0.21
NiO	0.53	0.53	NC	0.53	0.55	0.57	0.57	0.55	0.53	0.53	0.53
P ₂ O ₅	0.43	0.68	NC	0.43	0.69	0.69	0.70	0.70	0.70	0.71	0.73
ReO ₂	§	0.004	NC	0.05	0.007	0.010	0.014	0.013	0.013	0.014	0.014
SiO ₂	44.70	43.86	-1.87	44.67	43.57	43.09	42.85	43.07	43.01	42.66	43.15
SnO ₂	0.21	0.19	NC	0.21	0.22	0.20	0.20	0.18	0.20	0.21	0.20
SO ₃	0.12	0.16	NC	0.12	0.16	0.16	0.16	0.15	0.15	0.16	0.15
TiO ₂	§	0.05	NC	§	0.05	0.05	0.05	0.05	0.05	0.05	0.05
ZnO	2.00	1.68	-15.89	2.00	1.68	1.75	1.82	1.73	1.69	1.73	1.69
ZrO ₂	3.64	3.27	-10.03	3.64	3.36	3.45	3.51	3.30	3.34	3.47	3.32
Sum	100.00	100.00	NC	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00

* Target values calculated based on simple well stirred tank model using the DCP-AES analyzed results for the first discharged glass during Test 1.

§ - Not a target constituent

NC – Not calculated

**Table 4.2. XRF Analyzed Compositions for Glass Discharged During DM10 Tests (wt%),
(Continued).**

Test	2			3							
Mass (kg)	Target	43.06		Target	45.64	47.92	50.30	52.56	54.42	56.84	58.76
Sample		ENM-G-17C	% Dev.		ENM-G-21A	ENM-G-26A	ENM-G-26B	ENM-G-29A	ENM-G-29B	ENM-G-29C	ENM-G-29D
Al ₂ O ₃	7.81	7.16	-8.33	7.81	7.39	7.31	7.43	7.52	7.54	7.71	7.78
B ₂ O ₃ *	10.49	10.57	0.74	10.49	10.55	10.54	10.53	10.52	10.52	10.51	10.51
CaO	0.44	1.39	NC	0.44	1.22	1.15	1.02	0.97	0.84	0.78	0.76
CdO	0.69	0.86	NC	0.69	0.79	0.83	0.80	0.71	0.69	0.65	0.67
Ce ₂ O ₃	0.25	0.25	NC	0.25	0.24	0.27	0.27	0.25	0.27	0.28	0.27
Cr ₂ O ₃	0.15	0.22	NC	0.15	0.21	0.22	0.22	0.20	0.21	0.20	0.20
Cs ₂ O	0.16	0.11	NC	0.16	0.12	0.13	0.16	0.14	0.15	0.15	0.16
Fe ₂ O ₃	11.97	12.97	8.32	11.97	12.29	12.60	12.55	12.24	12.16	11.85	11.94
K ₂ O	§	0.55	NC	§	0.43	0.39	0.30	0.28	0.21	0.18	0.15
La ₂ O ₃	0.28	0.14	NC	0.28	0.12	0.13	0.13	0.13	0.16	0.16	0.16
Li ₂ O*	3.75	3.80	1.31	3.75	3.79	3.78	3.77	3.77	3.76	3.76	3.76
MgO	§	0.14	NC	§	0.14	0.14	0.10	0.08	<0.01	0.06	<0.01
MnO	0.29	0.82	NC	0.29	0.71	0.69	0.60	0.55	0.50	0.45	0.43
Na ₂ O	11.86	11.11	-6.37	11.86	12.11	11.69	11.59	11.99	12.71	12.09	11.97
Nd ₂ O ₃	0.21	0.22	NC	0.21	0.21	0.21	0.20	0.19	0.19	0.18	0.18
NiO	0.53	0.55	NC	0.53	0.48	0.56	0.55	0.50	0.55	0.52	0.53
P ₂ O ₅	0.43	0.71	NC	0.43	0.68	0.64	0.61	0.57	0.54	0.54	0.52
ReO ₂	0.05	0.014	NC	0.043	0.014	0.014	0.014	0.014	0.014	0.014	0.014
SiO ₂	44.67	42.72	-4.37	44.68	43.11	43.09	43.45	43.81	43.46	44.54	44.47
SnO ₂	0.21	0.21	NC	0.21	0.21	0.21	0.22	0.20	0.19	0.19	0.20
SO ₃	0.12	0.16	NC	0.12	0.12	0.10	0.09	0.08	0.07	0.07	0.07
TiO ₂	§	0.05	NC	§	0.03	0.03	<0.01	<0.01	<0.01	<0.01	<0.01
ZnO	2.00	1.77	-11.38	2.00	1.71	1.81	1.87	1.88	1.91	1.85	1.90
ZrO ₂	3.64	3.50	-3.72	3.64	3.30	3.46	3.52	3.38	3.36	3.25	3.34
Sum	100.00	100.00	NC	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00

* Target values calculated based on simple well stirred tank model using the DCP-AES analyzed results for the first discharged glass during Test 1.

§ - Not a target constituent

NC – Not calculated

**Table 4.2. XRF Analyzed Compositions for Glass Discharged During DM10 Tests (wt%),
(Continued).**

Test	3						4						
Mass (kg)	Target	60.74	63.16	65.02	67.54		Target	69.56	71.90	73.84	75.80	77.56	79.34
Sample		ENM-G-29E	ENM-G-31A	ENM-G-31B	ENM-G-36A	% Dev.		ENM-G-36B	ENM-G-37A	ENM-G-37B	ENM-G-37C	ENM-G-41A	ENM-G-41B
Al ₂ O ₃	7.81	7.61	7.63	7.68	7.72	-1.06	7.81	7.67	7.67	7.69	7.47	7.46	7.48
B ₂ O ₃ *	10.49	10.51	10.50	10.50	10.50	0.06	10.49	10.50	10.50	10.50	10.50	10.50	10.50
CaO	0.44	0.73	0.70	0.65	0.65	NC	0.44	0.64	0.60	0.60	0.62	0.58	0.59
CdO	0.69	0.73	0.66	0.65	0.68	NC	0.69	0.68	0.60	0.60	0.66	0.61	0.66
Ce ₂ O ₃	0.25	0.27	0.28	0.28	0.28	NC	0.25	0.30	0.27	0.28	0.28	0.28	0.27
Cr ₂ O ₃	0.15	0.20	0.21	0.20	0.19	NC	0.15	0.20	0.19	0.19	0.20	0.19	0.19
Cs ₂ O	0.16	0.18	0.16	0.16	0.18	NC	0.16	0.17	0.15	0.15	0.18	0.17	0.18
Fe ₂ O ₃	11.97	12.11	12.03	11.70	11.74	-1.96	11.97	12.03	11.69	12.05	12.33	12.14	12.15
K ₂ O	§	0.14	0.12	0.09	0.09	NC	§	0.08	0.07	0.07	0.05	0.06	0.05
La ₂ O ₃	0.28	0.18	0.18	0.18	0.20	NC	0.28	0.20	0.20	0.20	0.21	0.20	0.20
Li ₂ O*	3.75	3.76	3.75	3.75	3.75	0.11	3.75	3.75	3.75	3.75	3.75	3.75	3.75
MgO	§	<0.01	<0.01	<0.01	<0.01	NC	§	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
MnO	0.29	0.43	0.40	0.38	0.37	NC	0.29	0.36	0.34	0.34	0.34	0.33	0.33
Na ₂ O	11.86	12.26	12.63	13.16	13.00	9.59	11.86	12.68	13.46	12.29	12.31	12.97	12.81
Nd ₂ O ₃	0.21	0.19	0.18	0.18	0.18	NC	0.21	0.17	0.17	0.17	0.18	0.18	0.18
NiO	0.53	0.55	0.55	0.52	0.53	NC	0.53	0.54	0.51	0.53	0.53	0.51	0.51
P ₂ O ₅	0.43	0.51	0.51	0.49	0.48	NC	0.43	0.49	0.47	0.50	0.48	0.47	0.48
ReO ₂	0.043	0.014	0.014	0.015	0.018	NC	0.042	0.017	0.017	0.017	0.016	0.017	0.015
SiO ₂	44.68	43.93	43.85	43.99	43.89	-1.75	44.68	43.89	44.06	44.67	44.32	44.16	44.17
SnO ₂	0.21	0.22	0.21	0.19	0.21	NC	0.21	0.21	0.18	0.18	0.21	0.19	0.20
SO ₃	0.12	0.07	0.07	0.07	0.07	NC	0.12	0.07	0.07	0.06	0.07	0.07	0.06
TiO ₂	§	<0.01	<0.01	<0.01	<0.01	NC	§	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
ZnO	2.00	1.95	1.95	1.89	1.92	-3.79	2.00	1.96	1.87	1.93	1.95	1.94	1.93
ZrO ₂	3.64	3.46	3.39	3.24	3.35	-8.04	3.64	3.40	3.15	3.22	3.34	3.22	3.28
Sum	100.00	100.00	100.00	100.00	100.00	NC	100.00	100.00	100.00	100.00	100.00	100.00	100.00

* Target values calculated based on simple well stirred tank model using the DCP-AES analyzed results for the first discharged glass during Test 1.

§ - Not a target constituent

NC – Not calculated

**Table 4.2. XRF Analyzed Compositions for Glass Discharged During DM10 Tests (wt%),
(Continued).**

Test	4					5					
Mass (kg)	Target	81.64	83.78	86.62		Target	89.36	91.38	93.46	95.38	97.36
Sample		ENM-G-41C	ENM-G-41D	ENM-G-42A	% Dev.		ENM-G-44A	ENM-G-44B	ENM-G-45A	ENM-G-45B	ENM-G-49A
Al ₂ O ₃	7.81	7.41	7.42	7.37	-5.64	7.81	7.28	7.23	7.25	7.20	7.09
B ₂ O ₃ *	10.49	10.50	10.50	10.50	0.01	10.49	10.49	10.49	10.49	10.49	10.49
CaO	0.44	0.59	0.57	0.58	NC	0.44	0.65	0.82	0.89	0.98	1.09
CdO	0.69	0.65	0.64	0.58	NC	0.69	0.63	0.73	0.76	0.72	0.81
Ce ₂ O ₃	0.25	0.28	0.29	0.30	NC	0.25	0.28	0.29	0.29	0.26	0.28
Cr ₂ O ₃	0.15	0.19	0.18	0.19	NC	0.15	0.19	0.21	0.22	0.23	0.23
Cs ₂ O	0.16	0.18	0.18	0.15	NC	0.16	0.16	0.16	0.14	0.12	0.13
Fe ₂ O ₃	11.97	12.33	12.06	12.53	4.62	11.97	12.52	12.99	13.07	12.82	13.31
K ₂ O	§	<0.01	<0.01	<0.01	NC	§	0.08	0.17	0.22	0.29	0.33
La ₂ O ₃	0.28	0.20	0.20	0.22	NC	0.28	0.20	0.17	0.16	0.12	0.13
Li ₂ O*	3.75	3.75	3.75	3.75	0.02	3.75	3.75	3.75	3.75	3.75	3.75
MgO	§	<0.01	<0.01	<0.01	NC	§	<0.01	<0.01	0.07	0.09	0.13
MnO	0.29	0.33	0.32	0.33	NC	0.29	0.37	0.48	0.54	0.58	0.65
Na ₂ O	11.86	12.70	13.01	12.76	7.53	11.86	12.75	12.07	11.85	12.40	11.67
Nd ₂ O ₃	0.21	0.17	0.18	0.18	NC	0.21	0.18	0.20	0.20	0.20	0.21
NiO	0.53	0.53	0.48	0.53	NC	0.53	0.53	0.56	0.57	0.55	0.57
P ₂ O ₅	0.43	0.47	0.48	0.46	NC	0.43	0.49	0.53	0.56	0.59	0.61
ReO ₂	0.042	0.015	0.017	0.017	NC	0.05	0.017	0.019	0.018	0.019	0.018
SiO ₂	44.68	44.14	44.31	44.18	-1.10	44.67	43.83	43.27	43.06	42.95	42.52
SnO ₂	0.21	0.20	0.20	0.18	NC	0.21	0.20	0.21	0.22	0.19	0.21
SO ₃	0.12	0.07	0.06	0.07	NC	0.12	0.08	0.12	0.14	0.16	0.18
TiO ₂	§	<0.01	<0.01	<0.01	NC	§	<0.01	<0.01	<0.01	<0.01	0.02
ZnO	2.00	1.96	1.90	1.97	-1.24	2.00	1.95	1.98	1.96	1.88	1.94
ZrO ₂	3.64	3.32	3.25	3.17	-12.96	3.64	3.35	3.54	3.57	3.41	3.62
Sum	100.00	100.00	100.00	100.00	NC	100.00	100.00	100.00	100.00	100.00	100.00

* Target values calculated based on simple well stirred tank model using the DCP-AES analyzed results for the first discharged glass during Test 1.

§ - Not a target constituent

NC – Not calculated.

**Table 4.2. XRF Analyzed Compositions for Glass Discharged During DM10 Tests (wt%)
(continued).**

Test	5			
Mass (kg)	Target	99.28	100.72	
Sample		ENM-G-49B	ENM-G-49C	% Dev.
Al ₂ O ₃	7.81	7.32	7.22	-7.45
B ₂ O ₃ *	10.49	10.49	10.49	0.00
CaO	0.44	1.06	1.14	NC
CdO	0.69	0.78	0.84	NC
Ce ₂ O ₃	0.25	0.25	0.27	NC
Cr ₂ O ₃	0.15	0.20	0.22	NC
Cs ₂ O	0.16	0.13	0.14	NC
Fe ₂ O ₃	11.97	12.53	12.94	8.04
K ₂ O	§	0.34	0.38	NC
La ₂ O ₃	0.28	0.13	0.12	NC
Li ₂ O*	3.75	3.75	3.75	0.00
MgO	§	0.09	0.15	NC
MnO	0.29	0.63	0.68	NC
Na ₂ O	11.86	11.93	11.85	-0.08
Nd ₂ O ₃	0.21	0.20	0.21	NC
NiO	0.53	0.48	0.56	NC
P ₂ O ₅	0.43	0.63	0.65	NC
ReO ₂	0.05	0.017	0.018	NC
SiO ₂	44.67	43.52	42.66	-4.50
SnO ₂	0.21	0.20	0.22	NC
SO ₃	0.12	0.16	0.18	NC
TiO ₂	§	0.02	0.02	NC
ZnO	2.00	1.79	1.82	-8.77
ZrO ₂	3.64	3.34	3.46	-4.98
Sum	100.00	100.00	100.00	NC

* Target values calculated based on simple well stirred tank model using the DCP-AES analyzed results for the first discharged glass during Test 1.

§ - Not a target constituent

NC – Not calculated

Table 4.3. List of Glass Pool Samples with Sampling Times, Measured Iron Oxidation State, Measured ReO₂ Concentrations and Observations of Secondary Phases.

Test	Date	Time	Sample I.D.	Fe ²⁺ / Fe _{tot} (%)	ReO ₂ (wt%)	Secondary Phases?	Depth of Glass Pool (inches)
Before 1	2/28/2011	9:05	DNM-D-147A	NA	0.003	No	8.00
End of 1	3/1/2011	19:35	ENM-D-15A	< 1.0	0.004	No	6.50
End of 2	3/2/2011	20:30	ENM-D-21A	< 1.0	0.014	No	6.75
End of 3	3/3/2011	17:20	ENM-D-36A	1.9	0.017	No	9.50
End of 4	3/4/2011	10:31	ENM-D-42A	2.3	0.018	No	8.00
End of 5	3/4/2011	21:00	ENM-D-49A	< 1.0	0.019	No	8.25

NA – Not analyzed

Table 4.4. Comparison of XRF Analyzed Compositions for Glass Pool and Discharge Glass Samples Taken from the End of DM10 Tests (wt%).

Test	1		2		3		4		5	
Type	Pool	Discharge	Pool	Discharge	Pool	Discharge	Pool	Discharge	Pool	Discharge
Name	ENM-D-15A	ENM-G-13E	ENM-D-21A	ENM-G-17C	ENM-D-36A	ENM-G-36A	ENM-D-42A	ENM-G-42A	ENM-G-49A	ENM-G-49C
Al ₂ O ₃	6.89	6.93	7.05	7.16	7.73	7.72	7.32	7.37	7.09	7.22
B ₂ O ₃ *	10.85	10.85	10.57	10.57	10.50	10.50	10.50	10.50	10.49	10.49
CaO	1.37	1.36	1.43	1.39	0.67	0.65	0.57	0.58	1.09	1.14
CdO	0.83	0.79	0.85	0.86	0.69	0.68	0.62	0.58	0.81	0.84
Ce ₂ O ₃	0.23	0.21	0.26	0.25	0.30	0.28	0.29	0.30	0.28	0.27
Cr ₂ O ₃	0.25	0.24	0.23	0.22	0.21	0.19	0.20	0.19	0.23	0.22
Cs ₂ O	0.12	0.11	0.10	0.11	0.18	0.18	0.16	0.15	0.13	0.14
Fe ₂ O ₃	12.80	12.23	13.49	12.97	12.02	11.74	12.72	12.53	13.31	12.94
K ₂ O	0.55	0.56	0.55	0.55	0.09	0.09	<0.01	<0.01	0.33	0.38
La ₂ O ₃	0.12	0.12	0.14	0.14	0.18	0.20	0.22	0.22	0.13	0.12
Li ₂ O*	3.97	3.97	3.80	3.80	3.75	3.75	3.75	3.75	3.75	3.75
MgO	0.20	0.19	0.15	0.14	<0.01	<0.01	§	<0.01	0.13	0.15
MnO	0.78	0.75	0.85	0.82	0.38	0.37	0.33	0.33	0.65	0.68
Na ₂ O	10.82	11.06	10.85	11.11	12.17	13.00	12.26	12.76	11.67	11.85
Nd ₂ O ₃	0.21	0.20	0.22	0.22	0.18	0.18	0.18	0.18	0.21	0.21
NiO	0.59	0.53	0.60	0.55	0.55	0.53	0.56	0.53	0.57	0.56
P ₂ O ₅	0.67	0.68	0.71	0.71	0.51	0.48	0.47	0.46	0.61	0.65
ReO ₂	0.004	0.004	0.014	0.014	0.017	0.018	0.018	0.017	0.019	0.018
SiO ₂	43.17	43.86	42.34	42.72	44.17	43.89	44.15	44.18	42.52	42.66
SnO ₂	0.21	0.19	0.20	0.21	0.21	0.21	0.19	0.18	0.21	0.22
SO ₃	0.16	0.16	0.15	0.16	0.07	0.07	0.08	0.07	0.18	0.18
TiO ₂	0.06	0.05	0.05	0.05	<0.01	<0.01	<0.01	<0.01	0.02	0.02
ZnO	1.76	1.68	1.83	1.77	1.98	1.92	2.03	1.97	1.94	1.82
ZrO ₂	3.39	3.27	3.57	3.50	3.44	3.35	3.38	3.17	3.62	3.46
Sum	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00

§ - Not a target constituent

* - Target values.

Table 5.1. Results from DM10 Test 1 Off-Gas Emission Samples.

		Feed [#] (mg/min)	3/1/11 11:52 – 12:52 5.46% Moisture, 101% Isokinetic		3/1/11 14:34 – 15:34 5.49% Moisture, 98.2% Isokinetic		3/1/11 16:39 – 17:39 6.15% Moisture, 99.7% Isokinetic	
			Output (mg/min)	% Emitted	Output (mg/min)	% Emitted	Output (mg/min)	% Emitted
Particulate	Total ^{\$}	18322	97.96	0.53	87.92	0.48	136.75	0.75
	Al	616	2.11	0.34	1.92	0.31	3.11	0.51
	B	486	6.04	1.24	5.60	1.15	8.36	1.72
	Ca	46.9	0.65	1.40	0.54	1.15	0.95	2.03
	Cd	92	1.53	1.66	1.60	1.73	2.35	2.55
	Cr	15.3	0.25	1.64	0.25	1.62	0.32	2.10
	Cs	22.5	< 0.10	< 0.44	< 0.10	< 0.44	0.17	0.74
	Fe	1248	7.15	0.57	6.77	0.54	10.70	0.86
	Li	260	1.15	0.44	1.14	0.44	1.62	0.63
	Mn	33.5	< 0.10	< 0.30	< 0.10	< 0.30	< 0.10	< 0.30
	Na	1312	8.89	0.68	8.46	0.64	12.67	0.97
	Nd	62.6	< 0.10	< 0.16	< 0.10	< 0.16	< 0.10	< 0.16
	Ni	62.1	0.32	0.52	0.25	0.41	0.44	0.71
	P	28.0	0.17	0.59	< 0.10	< 0.36	0.11	0.40
	Re	0	< 0.10	NC	< 0.10	NC	< 0.10	NC
	S*	7.2	1.93	27.0	1.76	24.6	2.67	37.3
	Sn	24.6	< 0.10	< 0.41	< 0.10	< 0.41	< 0.10	< 0.41
	Si	3114	13.14	0.42	11.34	0.36	18.90	0.61
	Zn	239	1.15	0.48	1.11	0.46	1.73	0.72
	Zr	402	0.88	0.22	0.70	0.18	1.28	0.32
Gas	B	486	2.21	0.46	2.20	0.45	2.61	0.54
	S	7.2	8.66	121	7.59	106	9.11	127

^{\$} - From gravimetric analysis of filters and particulate nitric acid rinses

[#] - Calculated from target composition and test average glass production rate

* - Calculated from analysis of filter particulate by water dissolution

NC – Not Calculated

Table 5.2. Results from DM10 Test 2 Off-Gas Emission Samples.

		Feed [#] (mg/min)	3/2/11 11:11 – 12:11 4.56% Moisture, 101% Isokinetic		3/2/11 14:22 – 15:22 4.38% Moisture, 101% Isokinetic		3/2/11 16:10 – 17:10 4.50% Moisture, 98.2% Isokinetic	
			Output (mg/min)	% Emitted	Output (mg/min)	% Emitted	Output (mg/min)	% Emitted
Particulate	Total ^{\$}	14369	91.48	0.64	62.18	0.43	108.03	0.75
	Al	485	2.10	0.43	1.30	0.27	2.52	0.52
	B	383	5.23	1.37	3.10	0.81	5.35	1.40
	Ca	36.9	0.57	1.54	0.32	0.86	0.69	1.88
	Cd	72.6	1.22	1.68	0.82	1.13	1.46	2.02
	Cr	12.1	0.14	1.20	0.12	1.01	0.16	1.30
	Cs	17.7	< 0.10	< 0.56	< 0.10	< 0.56	< 0.10	< 0.56
	Fe	984	7.17	0.73	4.23	0.43	8.47	0.86
	Li	205	1.01	0.49	0.62	0.30	1.20	0.58
	Mn	26.4	< 0.10	< 0.38	< 0.10	< 0.38	< 0.10	< 0.38
	Na	1034	8.28	0.80	5.37	0.52	9.40	0.91
	Nd	49.3	< 0.10	< 0.20	< 0.10	< 0.20	< 0.10	< 0.20
	Ni	48.9	0.32	0.66	0.19	0.39	0.38	0.77
	P	22.0	< 0.10	< 0.45	< 0.10	< 0.45	2.55	11.6
	Re	5.01	2.87	57.31	2.95	58.97	2.94	58.71
	S*	5.65	1.22	21.7	1.09	19.4	1.45	25.7
	Sn	19.4	< 0.10	< 0.51	< 0.10	< 0.51	< 0.10	< 0.51
	Si	2454	13.60	0.55	9.82	0.40	16.17	0.66
	Zn	189	1.20	0.63	0.65	0.34	1.38	0.73
	Zr	316	0.79	0.25	0.48	0.15	0.98	0.31
Gas	B	383	1.28	0.33	1.78	0.47	1.83	0.48
	S	5.65	4.24	75.1	5.08	90.0	6.64	118

^{\$} - From gravimetric analysis of filters and particulate nitric acid rinses

[#] - Calculated from target composition and test average glass production rate

* - Calculated from analysis of filter particulate by water dissolution

Table 5.3. Results from DM10 Test 3 Off-Gas Emission Samples.

		Feed [#] (mg/min)	3/3/11 11:57 – 12:57 9.98% Moisture, 104% Isokinetic		3/3/11 14:33 – 15:33 9.34% Moisture, 102% Isokinetic		3/3/11 15:51 – 16:51 7.38% Moisture, 109% Isokinetic	
			Output (mg/min)	% Emitted	Output (mg/min)	% Emitted	Output (mg/min)	% Emitted
Particulate	Total ^{\$}	27238	68.22	0.25	51.29	0.19	57.60	0.21
	Al	897	0.33	0.04	0.31	0.03	0.34	0.04
	B	707	3.08	0.44	1.62	0.23	1.35	0.19
	Ca	68.28	< 0.10	< 0.15	0.15	0.22	0.21	0.31
	Cd	134	2.35	1.75	1.60	1.19	0.95	0.71
	Cr	22.28	0.35	1.59	0.26	1.17	0.15	0.70
	Cs	32.77	0.28	0.85	0.39	1.19	< 0.10	< 0.31
	Fe	1819	0.54	0.03	0.36	0.02	0.30	0.02
	Li	378	0.97	0.26	0.64	0.17	0.32	0.08
	Mn	48.76	< 0.10	< 0.21	< 0.10	< 0.21	< 0.10	< 0.21
	Na	1912	9.15	0.48	6.52	0.34	4.60	0.24
	Nd	91.2	< 0.10	< 0.11	< 0.10	< 0.11	< 0.10	< 0.11
	Ni	90.4	< 0.10	< 0.11	< 0.10	< 0.11	< 0.10	< 0.11
	P	40.76	< 0.10	< 0.25	< 0.10	< 0.25	< 0.10	< 0.25
	Re	8.15	7.73	94.81	7.00	85.89	4.47	54.78
	S*	10.44	1.82	17.40	1.06	10.16	1.37	13.16
	Sn	35.91	< 0.10	< 0.28	< 0.10	< 0.28	< 0.10	< 0.28
	Si	4537	4.90	0.11	5.15	0.11	6.46	0.14
	Zn	349	0.15	0.04	0.12	0.03	0.12	0.04
	Zr	585	0.10	0.02	< 0.10	< 0.02	< 0.10	< 0.02
Gas	B	707	9.64	1.36	8.61	1.22	6.18	0.87
	S	10.44	6.56	62.8	6.80	65.2	5.04	48.3

^{\$} - From gravimetric analysis of filters and particulate nitric acid rinses

[#] - Calculated from target composition and test average glass production rate

* - Calculated from analysis of filter particulate by water dissolution

Table 5.4. Results from DM10 Test 4 Off-Gas Emission Samples.

		Feed [#] (mg/min)	3/4/11 05:19 – 06:19 9.40% Moisture, 99.5% Isokinetic		3/4/11 06:37 – 07:37 9.54% Moisture, 98.6% Isokinetic		3/4/11 07:59 – 08:59 9.10% Moisture, 98.8% Isokinetic	
			Output (mg/min)	% Emitted	Output (mg/min)	% Emitted	Output (mg/min)	% Emitted
Particulate	Total ^{\$}	25270	83.01	0.33	76.07	0.30	61.33	0.24
	Al	851	0.60	0.07	0.54	0.06	0.43	0.05
	B	671	4.88	0.73	4.28	0.64	2.60	0.39
	Ca	64.7	0.32	0.50	0.20	0.31	0.25	0.39
	Cd	127	2.61	2.05	2.25	1.77	1.59	1.25
	Cr	21.1	0.35	1.65	0.33	1.54	0.26	1.25
	Cs	31.1	0.33	1.07	0.36	1.16	0.29	0.94
	Fe	1724	1.53	0.09	1.39	0.08	0.93	0.05
	Li	359	1.16	0.32	1.09	0.30	0.79	0.22
	Mn	46.2	< 0.10	< 0.22	< 0.10	< 0.22	< 0.10	< 0.22
	Na	1813	11.34	0.63	10.24	0.56	7.78	0.43
	Nd	86.5	< 0.10	< 0.12	< 0.10	< 0.12	< 0.10	< 0.12
	Ni	85.7	< 0.10	< 0.12	< 0.10	< 0.12	< 0.10	< 0.12
	P	38.6	< 0.10	< 0.26	0.21	0.54	< 0.10	< 0.26
	Re	7.73	7.19	93.00	7.47	96.67	6.70	86.66
	S*	9.90	2.85	28.76	2.46	24.88	1.89	19.12
	Sn	34.1	< 0.10	< 0.29	< 0.10	< 0.29	< 0.10	< 0.29
	Si	4302	6.47	0.15	5.93	0.14	6.04	0.14
	Zn	331	0.33	0.10	0.29	0.09	0.18	0.05
	Zr	555	0.21	0.04	0.20	0.04	0.15	0.03
Gas	B	671	8.60	1.28	9.03	1.35	8.31	1.24
	S	9.90	3.22	32.5	5.23	52.9	3.23	32.6

^{\$} - From gravimetric analysis of filters and particulate nitric acid rinses

[#] - Calculated from target composition and test average glass production rate

* - Calculated from analysis of filter particulate by water dissolution

Table 5.5. Results from DM10 Test 5 Off-Gas Emission Samples.

		Feed [#] (mg/min)	3/4/11 16:51 – 17:51 8.69% Moisture, 101% Isokinetic	
			Output (mg/min)	% Emitted
Particulate	Total ^S	29295	206.55	0.71
	Al	974	4.48	0.46
	B	768	11.50	1.50
	Ca	74.2	1.18	1.59
	Cd	146	2.70	1.85
	Cr	24.2	0.55	2.26
	Cs	35.6	< 0.10	< 0.28
	Fe	1976	17.98	0.91
	Li	411	2.56	0.62
	Mn	53.0	< 0.10	< 0.19
	Na	2077	20.12	0.97
	Nd	99.0	< 0.10	< 0.10
	Ni	98.2	0.64	0.65
	P	44.3	0.25	0.57
	Re	10.1	7.66	76.16
	S*	11.3	4.01	35.4
	Sn	39.0	0.16	0.41
	Si	4928	22.81	0.46
	Zn	379	2.91	0.77
	Zr	635	1.44	0.23
Gas	B	768	3.60	0.47
	S	11.3	8.22	72.5

^S - From gravimetric analysis of filters and particulate nitric acid rinses

[#] - Calculated from target composition and test average glass production rate

* - Calculated from analysis of filter particulate by water dissolution

Table 5.6. Average Emissions Rates and Carryover for DM10 Tests.

		Test 1		Test 2		Test 3		Test 4		Test 5	
		Output (mg/min)	% Emitted	Output (mg/min)	% Emitted	Output (mg/min)	% Emitted	Output (mg/min)	% Emitted	Output (mg/min)	% Emitted
Particulate	Total ^s	107.54	0.59	87.23	0.61	59.04	0.22	73.47	0.29	206.55	0.71
	Al	2.38	0.39	1.97	0.41	0.33	0.04	0.52	0.06	4.48	0.46
	B	6.67	1.37	4.56	1.19	2.02	0.29	3.92	0.58	11.50	1.50
	Ca	0.71	1.53	0.53	1.43	0.18	0.26	0.26	0.40	1.18	1.59
	Cd	1.82	1.98	1.17	1.61	1.63	1.22	2.15	1.69	2.70	1.85
	Cr	0.27	1.78	0.14	1.17	0.26	1.15	0.31	1.48	0.55	2.26
	Cs	0.17	0.74	< 0.10	< 0.53	0.33	1.02	0.33	1.05	< 0.10	< 0.28
	Fe	8.21	0.66	6.62	0.67	0.40	0.02	1.28	0.07	17.98	0.91
	Li	1.31	0.50	0.94	0.46	0.64	0.17	1.01	0.28	2.56	0.62
	Mn	< 0.10	< 0.21	< 0.10	< 0.36	< 0.10	< 0.21	< 0.10	< 0.21	< 0.10	< 0.19
	Na	10.00	0.76	7.69	0.74	6.76	0.35	9.79	0.54	20.12	0.97
	Nd	< 0.10	< 0.11	< 0.10	< 0.19	< 0.10	< 0.11	< 0.10	< 0.11	< 0.10	< 0.10
	Ni	0.34	0.54	0.30	0.61	< 0.10	< 0.11	< 0.10	< 0.11	0.64	0.65
	P	0.14	0.50	2.55	11.58	< 0.10	< 0.25	0.21	0.54	0.25	0.57
	Re	< 0.10	NC	2.92	58.33	6.40	78.49	7.12	92.11	7.66	76.16
	S*	2.12	29.61	1.26	22.25	1.42	13.57	2.40	24.25	4.01	35.35
	Sn	< 0.10	< 0.29	< 0.10	< 0.49	< 0.10	< 0.29	< 0.10	< 0.28	0.16	0.41
	Si	14.46	0.46	13.20	0.54	5.50	0.12	6.15	0.14	22.81	0.46
	Zn	1.33	0.56	1.08	0.57	0.13	0.04	0.27	0.08	2.91	0.77
	Zr	0.96	0.24	0.75	0.24	0.10	0.02	0.19	0.03	1.44	0.23
Gas	B	2.34	0.48	1.63	0.43	8.14	1.15	8.65	1.29	3.60	0.47
	S	8.45	118	5.32	94.2	6.13	58.8	3.89	39.3	8.22	72.5

^s - From gravimetric analysis of filters and particulate nitric acid rinses

* - Calculated from analysis of filter particulate by water dissolution

Table 6.1. Mass Distribution of Rhenium Based on Alternative Feed Analyses and Glass Background Corrections.

	Test	2	3	4	5
	Feed Origin	NOAH + Perrhenic Acid	VSL+ Heated Goethite Slurry	VSL+ Unheated Goethite Slurry	NOAH + Perrhenic Acid
Glass	Target ReO ₂ wt% (Recipe amount – Tests 2 and 5, PNNL Feed Analysis – Tests 3 and 4)	0.05	0.043	0.042	0.05
	Target ReO ₂ wt% (Recipe amount – Tests 2 and 5, VSL Feed Analysis – Tests 3 and 4)	0.05	0.05375	0.05712	0.05
	Measured ReO ₂ wt%	0.014	0.015	0.017	0.018
	Background Corrected ReO ₂ wt% (- 0.0035 wt% measured in Test 1)	0.0105	0.0115	0.0135	0.0145
Feed: Recipe amount - Tests 2 and 5, PNNL Feed Analysis – Tests 3 and 4 Glass: Background corrected	% Feed Rhenium in Glass	21	27	32	29
	% Feed Rhenium in Exhaust	58	78 (92)*	92	76
	% Total Rhenium Recovery	79	105 (119)	124	105
Feed: Recipe amount - Tests 2 and 5, PNNL Feed Analysis – Tests 3 and 4 Glass: No Background correction	% Feed Rhenium in Glass	NA	35	40	NA
	% Feed Rhenium in Exhaust	NA	78 (92)	92	NA
	% Total Rhenium Recovery	NA	113 (127)	132	NA
Feed: Recipe amount - Tests 2 and 5, VSL Feed Analysis – Tests 3 and 4 Glass: Background corrected	% Feed Rhenium in Glass	21	21	24	29
	% Feed Rhenium in Exhaust	58	62 (74)	68	76
	% Total Rhenium Recovery	79	84 (95)	91	105
Feed: Recipe amount - Tests 2 and 5, VSL Feed Analysis – Tests 3 and 4 Glass: No Background correction	% Feed Rhenium in Glass	NA	28	30	NA
	% Feed Rhenium in Exhaust	NA	62 (74)	68	NA
	% Total Rhenium Recovery	NA	90 (102)	97	NA

*- Values based on an average of two of the emission values without the low outlier are shown in parentheses.

NA – Not applicable.

Note: The recommended mass balance is shown by the bold entries shaded in blue.



Figure 1.1. DM10 melter and feed tank; off-gas system is in the background to the left.

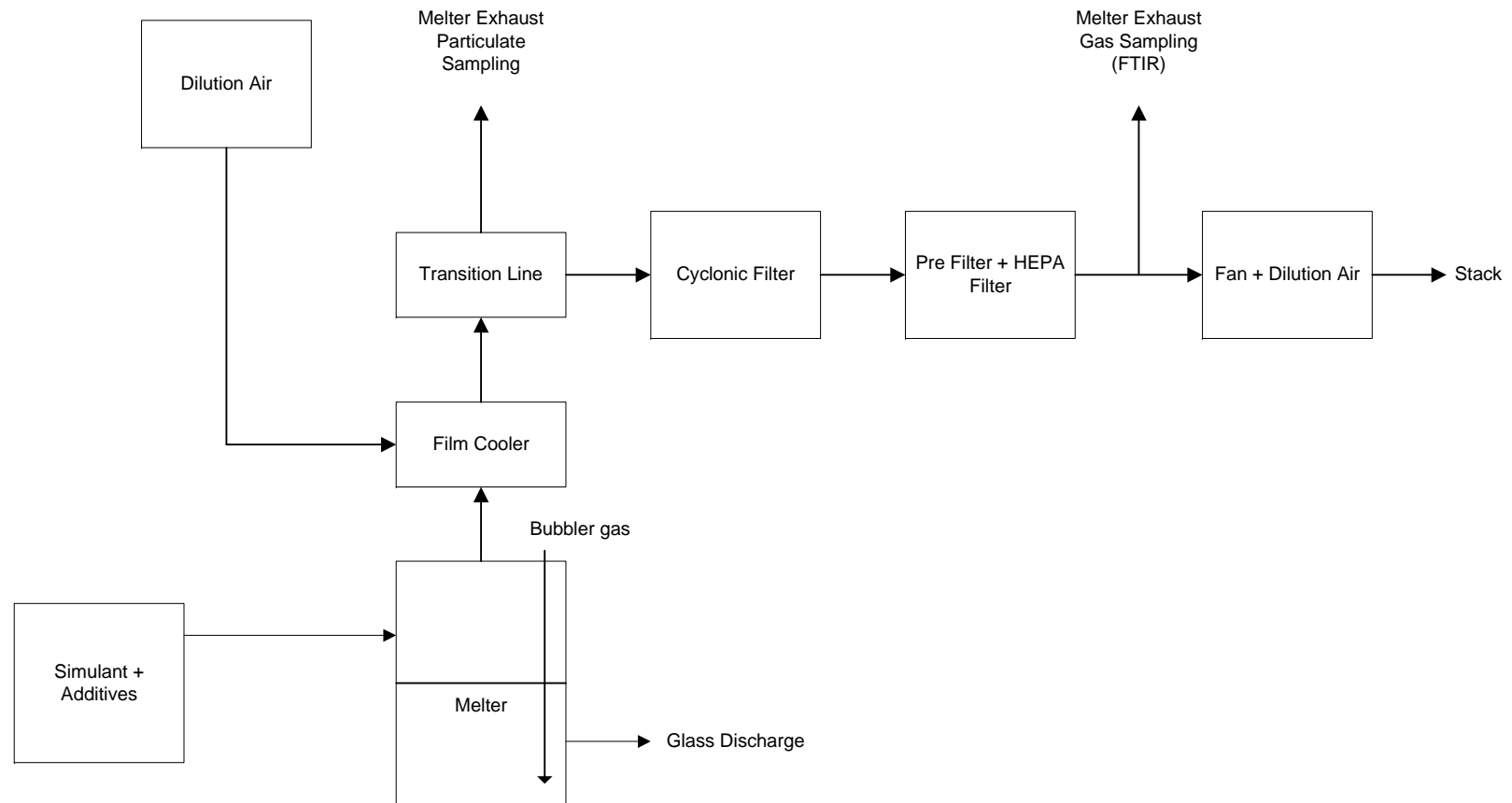


Figure 1.2. Schematic diagram of DM 10 vitrification system.



Figure 2.1. Feed composed of heated goethite slurry prior to mixing in additional water.



Figure 2.2. Feed composed of heated goethite slurry prior to mixing in additional water.

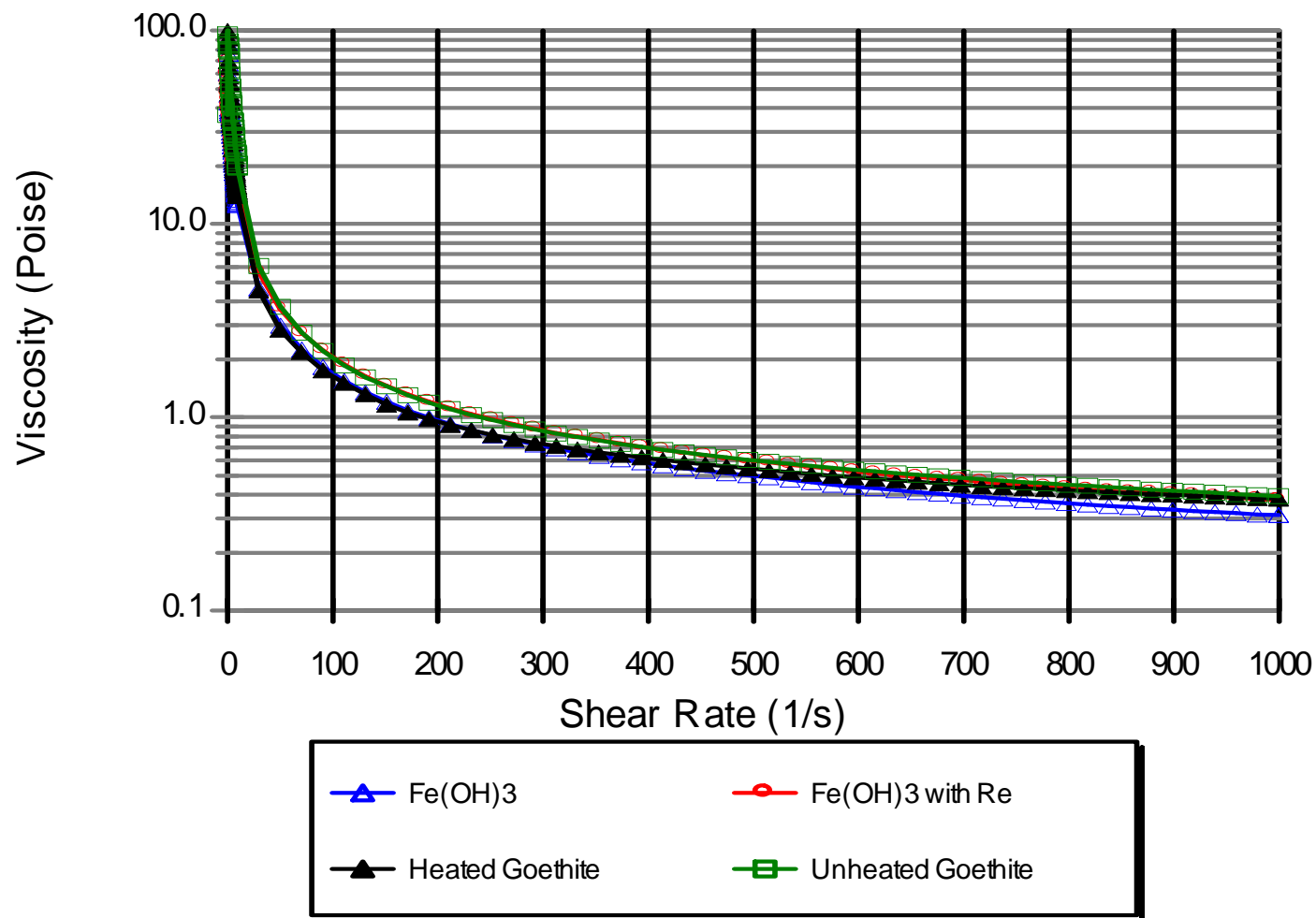


Figure 2.3. Viscosity versus shear rate for each feed with each iron and rhenium source.

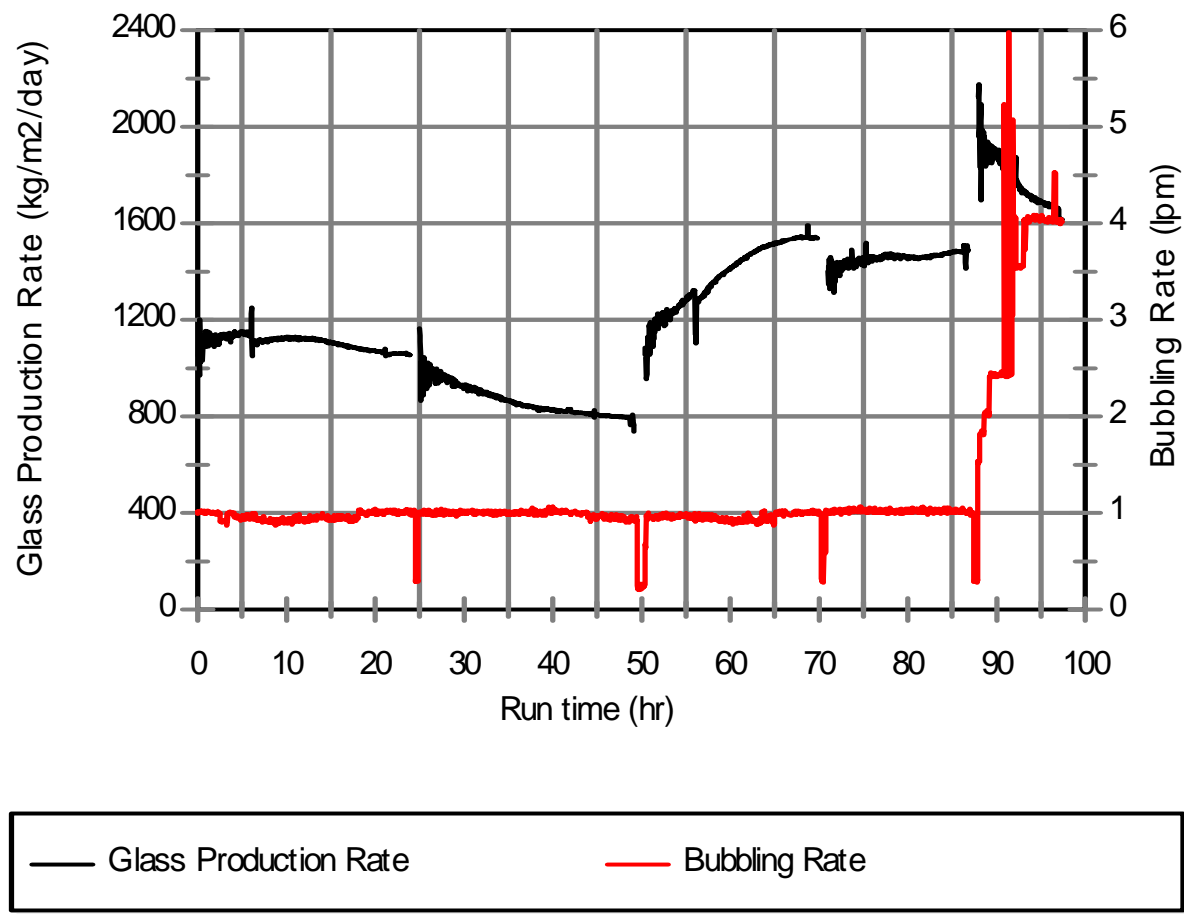


Figure 3.1. Glass production (cumulative) and bubbling rates for DM10 tests with goethite and rhenium.

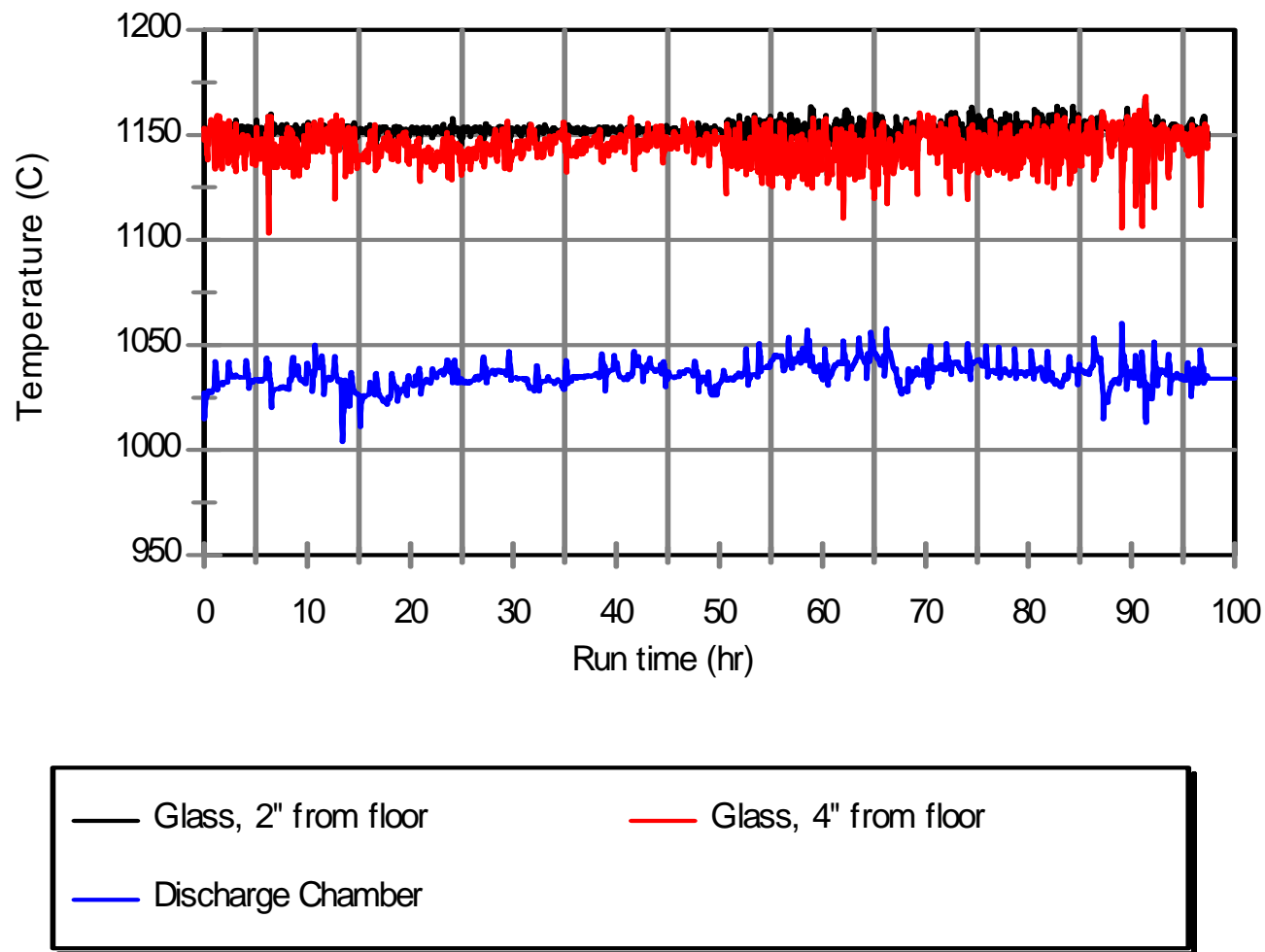


Figure 3.2. Glass temperatures for DM10 tests with goethite and rhenium.

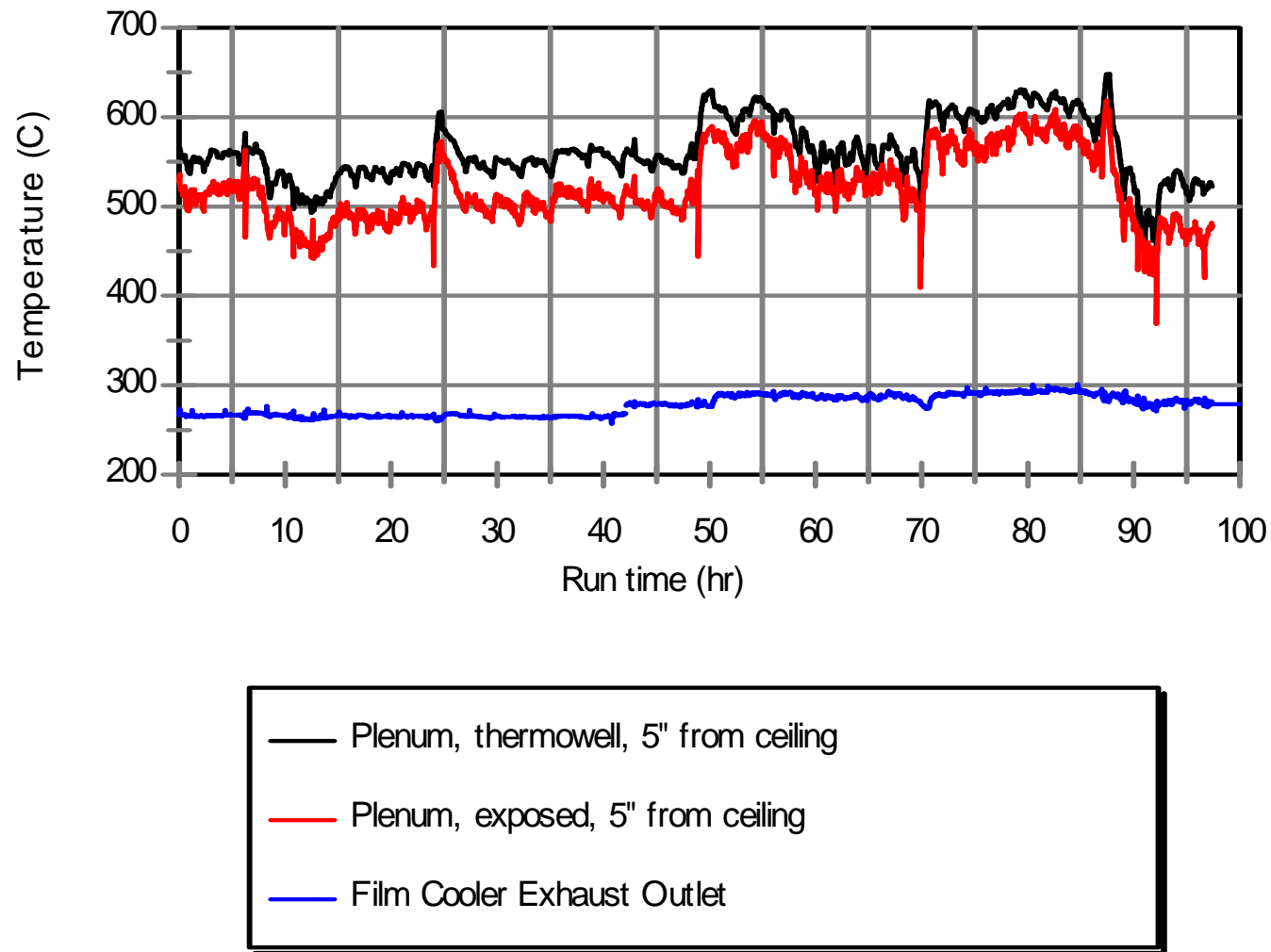


Figure 3.3. Plenum temperatures for DM10 tests with goethite and rhenium.

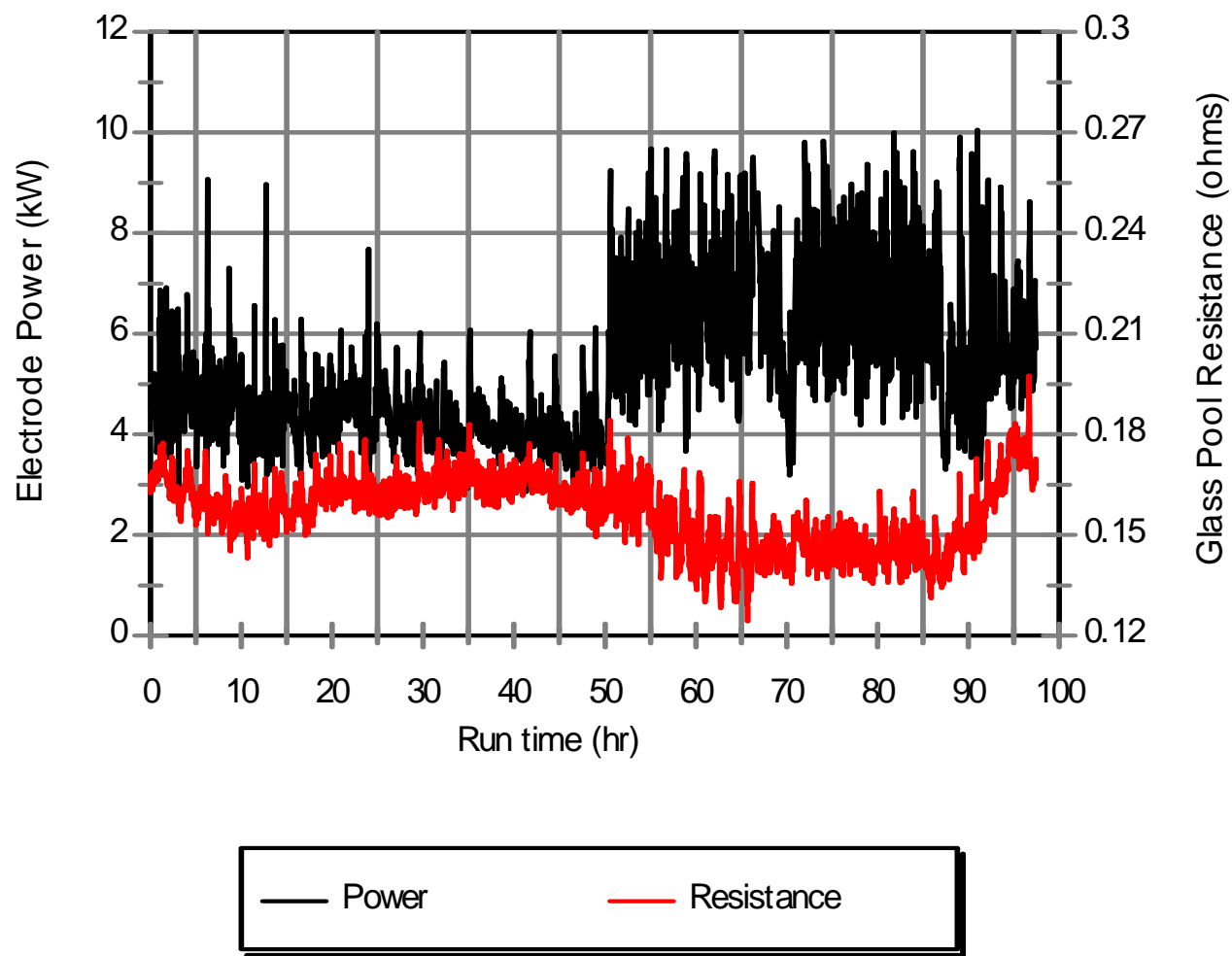


Figure 3.4. Electrode power and glass pool resistance for DM10 tests with goethite and rhenium.

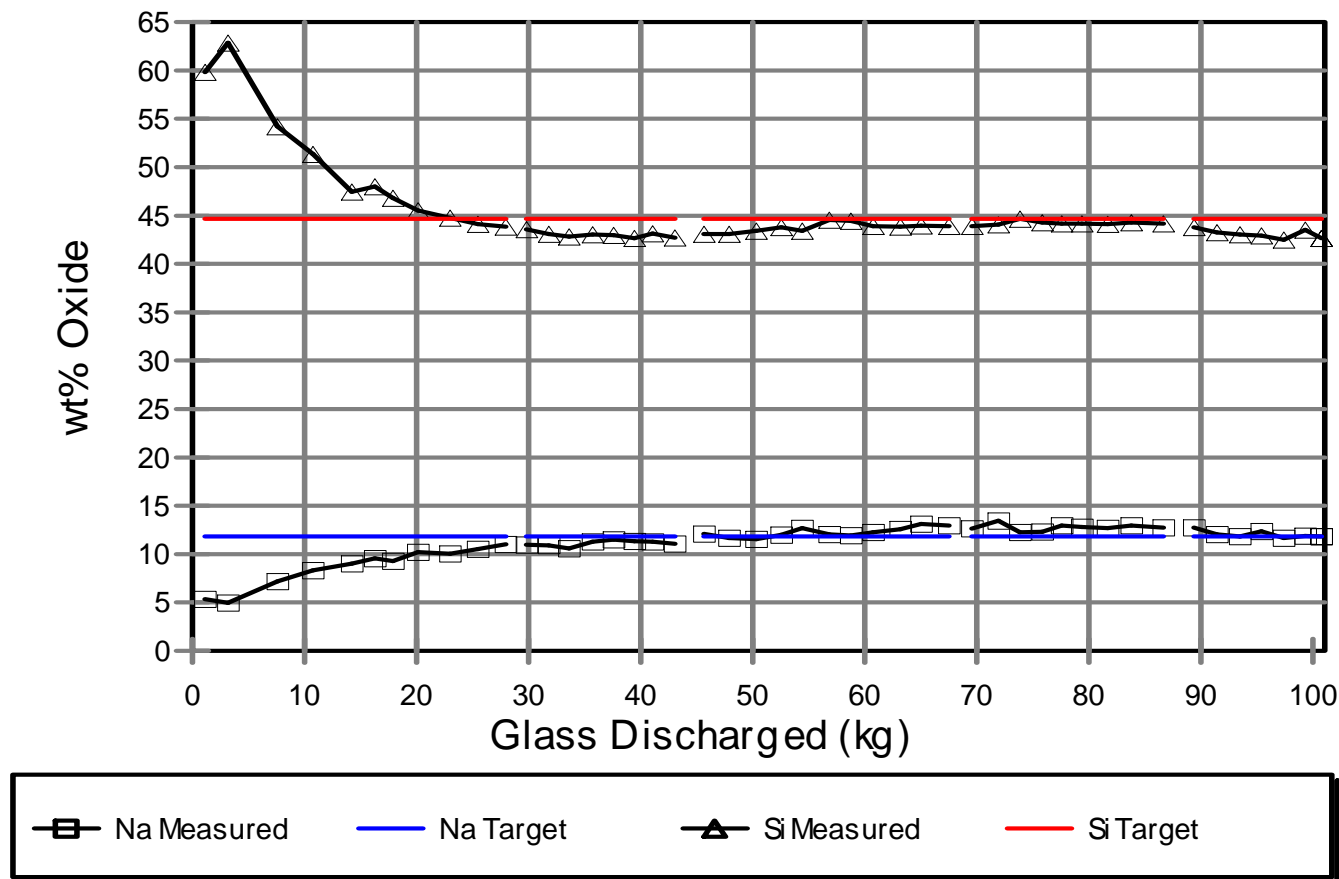


Figure 4.1. DM10 product and target glass soda and silica concentrations determined by XRF.

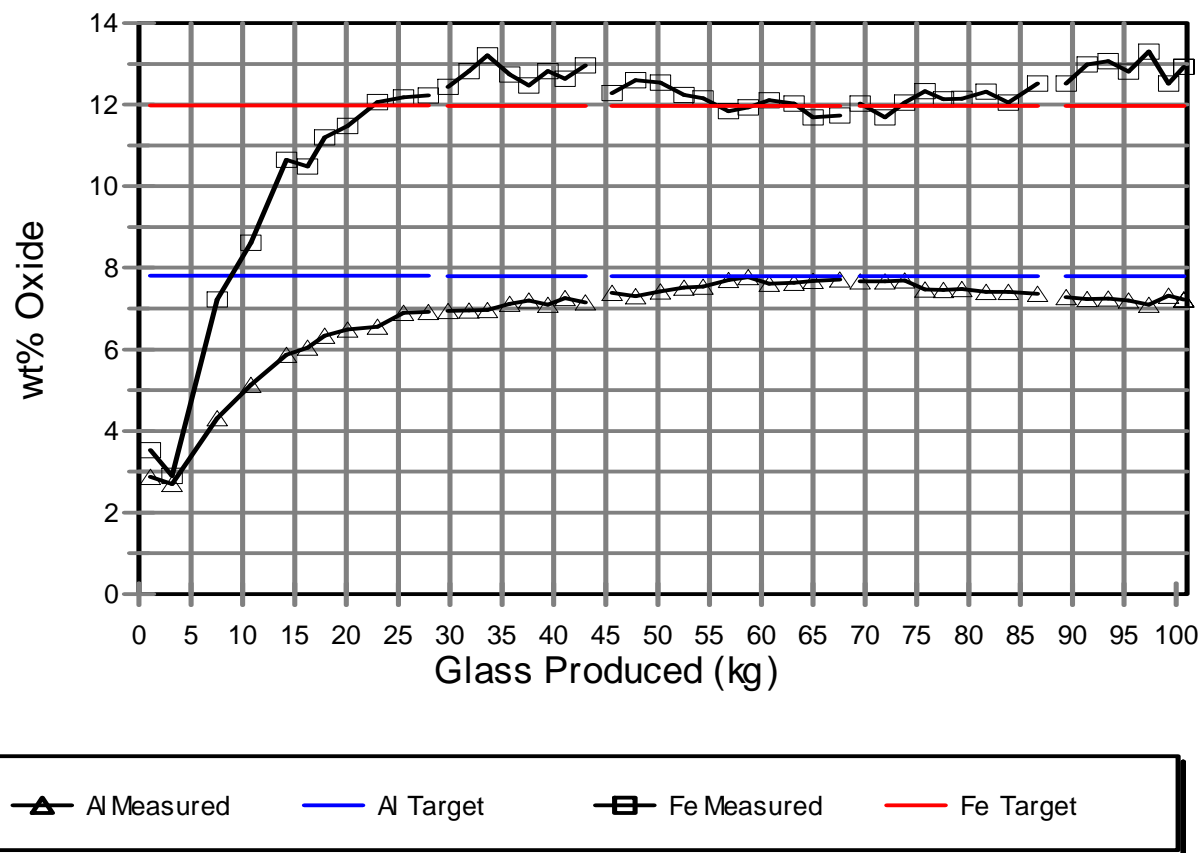


Figure 4.2. DM10 product and target glass aluminum and iron oxide concentrations determined by XRF.

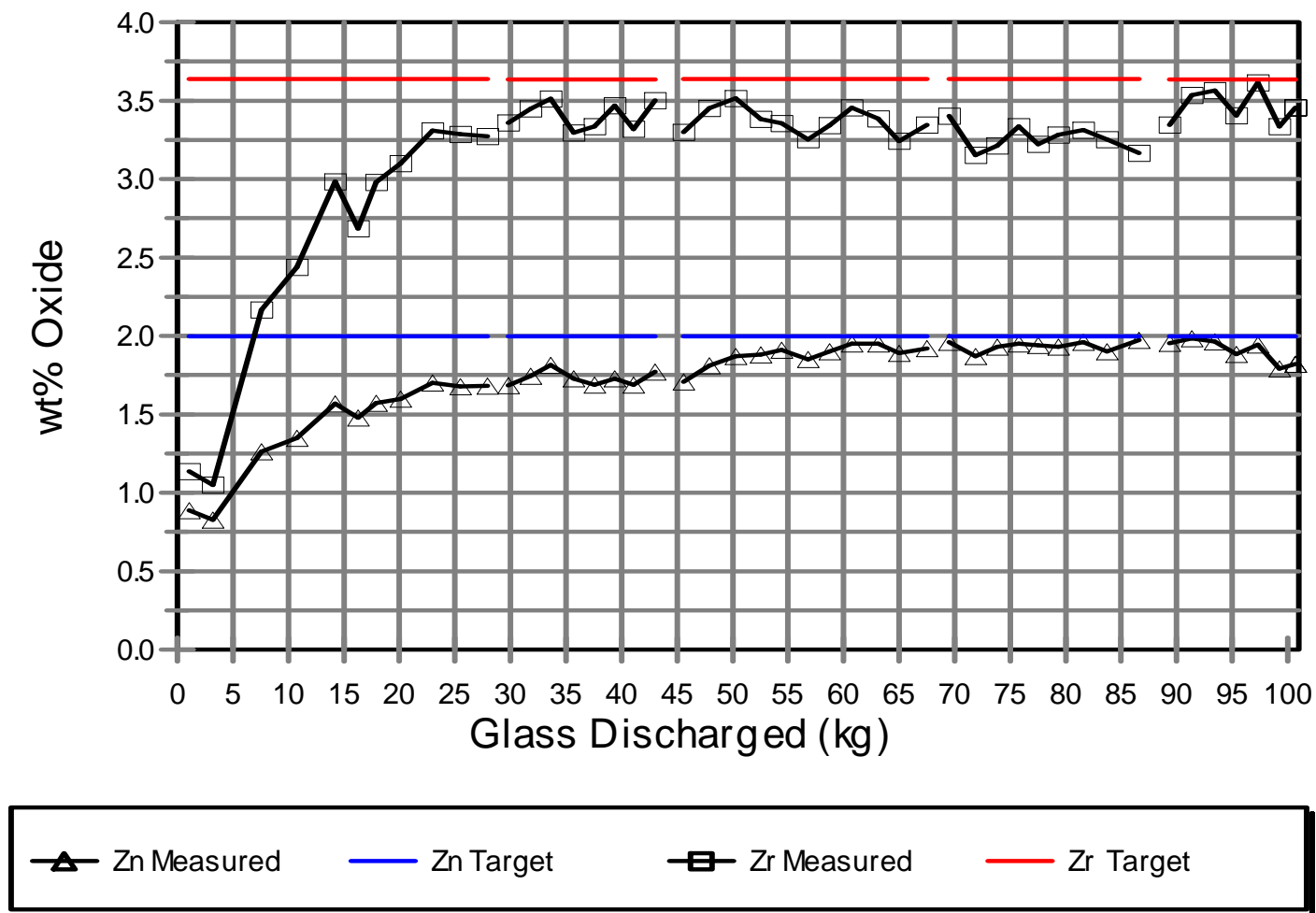


Figure 4.3. DM10 product and target glass zinc and zirconium oxide concentrations determined by XRF.

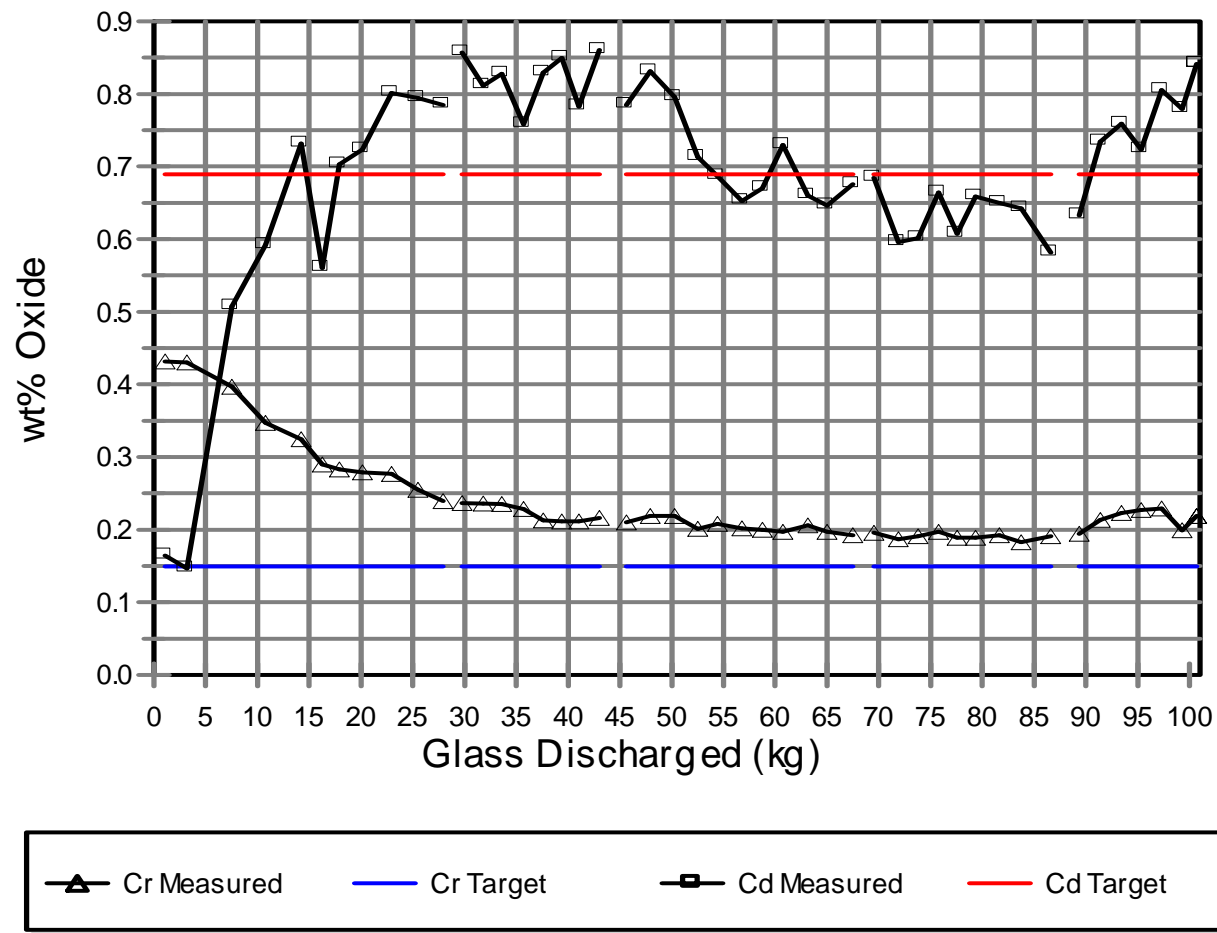


Figure 4.4. DM10 product and target glass chromium and cadmium oxide concentrations determined by XRF.

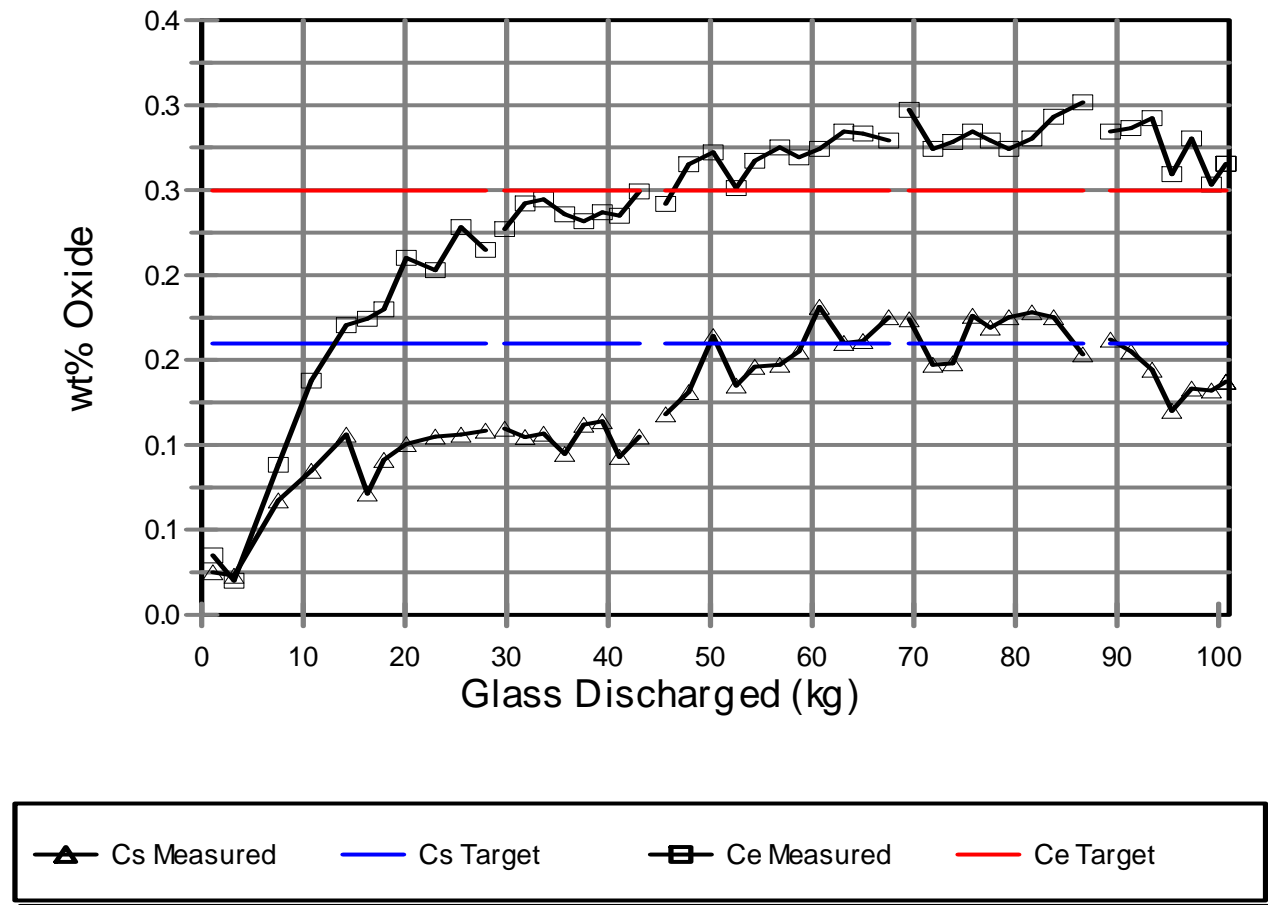


Figure 4.5. DM10 product and target glass cesium and cerium oxide concentrations determined by XRF.

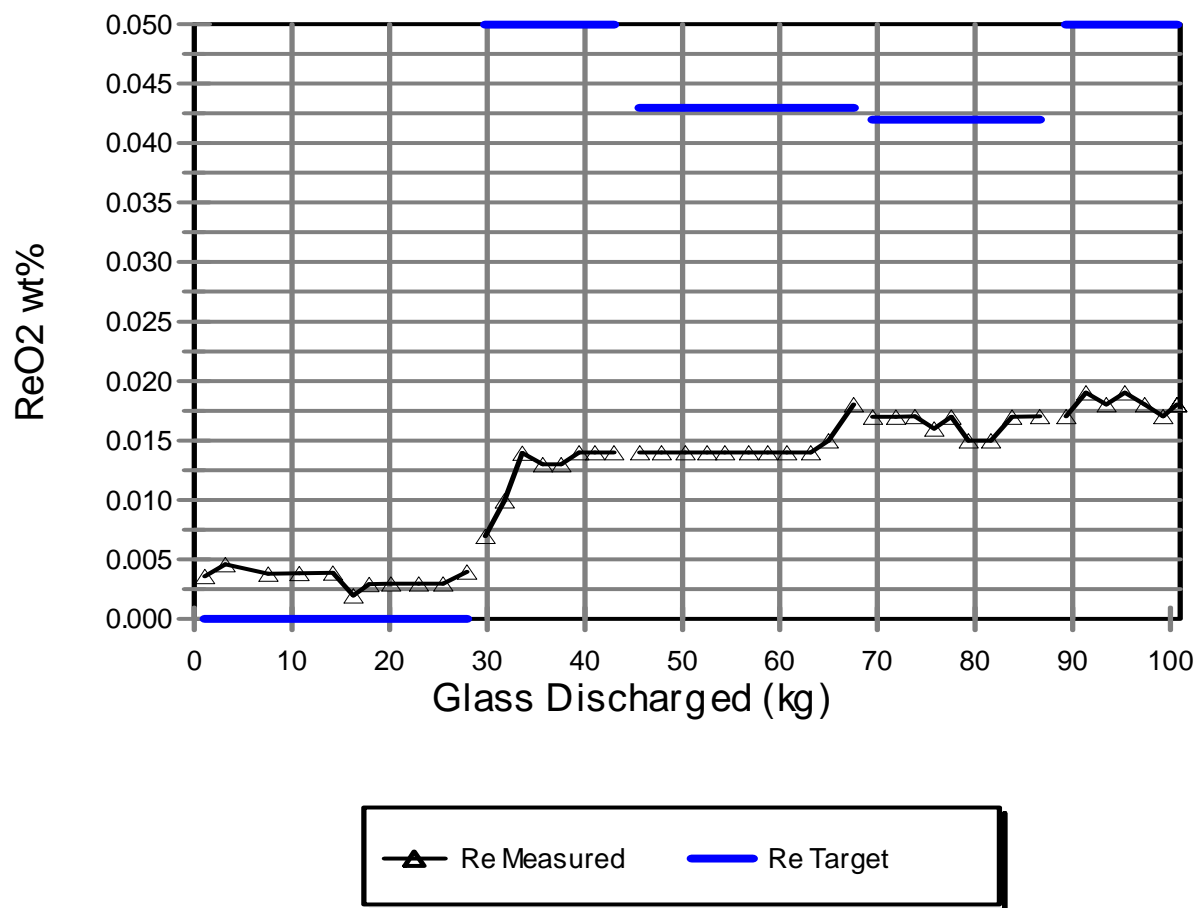


Figure 4.6. DM10 product and target glass rhenium oxide concentrations determined by XRF.
Note: target values for Tests 3 and 4 are based on the PNNL analysis of the goethite slurries.