

# **Redox Control for Hanford HLW Feeds, VSL-12R2530-1, Rev. 0**

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

**Office of River Protection**

**P.O. Box 450  
Richland, Washington 99352**

# Redox Control for Hanford HLW Feeds, VSL-12R2530-1, Rev. 0

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## **Final Report**

### **Redox Control for Hanford HLW Feeds**

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*Redox Control for Hanford HLW Feeds*  
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This report describes the results of testing specified by the above Test Plan. The work was performed in compliance with the quality assurance requirements specified in the Test Plan. Results required by the Test Plan are reported. The test results and this report have been reviewed for correctness, technical adequacy, completeness, and accuracy.

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## TABLE OF CONTENTS

LIST OF TABLES.....	4
LIST OF FIGURES.....	5
LIST OF ABBREVIATIONS.....	7
<b>SECTION 1.0 INTRODUCTION .....</b>	<b>8</b>
1.1    TEST OBJECTIVES.....	9
1.2    QUALITY ASSURANCE .....	10
1.3    MELTER SYSTEM DESCRIPTION .....	10
1.3.1 <i>Feed System</i> .....	10
1.3.2 <i>Melter System</i> .....	11
1.3.3 <i>Off-Gas System</i> .....	11
1.4    EXPERIMENTAL AND ANALYTICAL METHODS.....	12
1.4.1 <i>Feed</i> .....	12
1.4.2 <i>Compositional Analysis</i> .....	12
1.4.3 <i>Mössbauer Spectroscopy</i> .....	13
1.4.4 <i>Melter Exhaust Sampling and Analysis</i> .....	13
<b>SECTION 2.0 WASTE SIMULANT AND GLASS FORMULATIONS.....</b>	<b>14</b>
2.1    C-106/AY-102 WASTE SIMULANT .....	14
2.2    C-106/AY-102 GLASS AND FEED FORMULATION.....	15
2.3    OXALATE AND NITRATE CONCENTRATIONS .....	17
2.4    ANALYSIS OF MELTER FEED .....	18
<b>SECTION 3.0 MELTER OPERATIONS.....</b>	<b>19</b>
3.1    MELTER OPERATIONS DATA.....	20
<b>SECTION 4.0 GLASS PRODUCTS .....</b>	<b>22</b>
4.1    DISCHARGE GLASSES.....	22
4.2    GLASS POOL SAMPLES.....	23
4.2.1 <i>Redox State</i> .....	23
4.2.2 <i>Suction Samples</i> .....	25
<b>SECTION 5.0 MONITORED OFF-GAS EMISSIONS .....</b>	<b>26</b>
5.1    PARTICULATE SAMPLING .....	26
5.2    GASES MONITORED BY FTIR.....	27
<b>SECTION 6.0 MASS BALANCE FOR VOLATILES IN GLASS AND EMISSIONS .....</b>	<b>28</b>
<b>SECTION 7.0 SUMMARY AND CONCLUSIONS .....</b>	<b>30</b>
7.1    RECOMMENDATIONS FOR FUTURE WORK .....	32
<b>SECTION 8.0 REFERENCES .....</b>	<b>34</b>

## List of Tables

	<u>Page No.</u>
Table 2.1 Compositional Summary of Different Waste Streams and Blended Solids for the C-106/AY-102 HLW Simulant.	T-1
Table 2.2 Compositional Summary (Oxide Basis) of the C-106/AY-102 Base Simulant, Spikes, and the Target HLW Simulant.	T-2
Table 2.3 Compositional Summary (oxide basis) of the C-106/AY-102 HLW Simulant, Glass Former Additives, Target Test Glass, and the Reference Glass (HLW98-86).	T-3
Table 2.4 Composition of Melter Feed to Produce 1 Metric Ton of Target Glass from C-106/AY-102 HLW Simulant (20 wt% suspended solids).	T-4
Table 2.5 Nitrate, Nitrite, and Organic Carbon Content in Feed Used in Previous Melter Tests with HLW C-106/AY-102 Waste Compared to WTP Contract Limits.	T-5
Table 2.6 Analysis of As-Received Feed.	T-6
Table 2.7 Melter Feed Recipes for DM10 Tests.	T-7
Table 3.1 Summary of DM10 Melter Tests.	T-8
Table 4.1 Listing of Glasses Discharged During DM10 Tests.	T-11
Table 4.2 XRF Analyzed Compositions of Glass Discharged During DM10 Tests (wt%).	T-14
Table 4.3 List of Glass Pool Samples with Sampling Times, Measured Iron Oxidation State, Measured Rhenium Oxide Concentrations and Observations of Secondary Phases.	T-20
Table 4.4 XRF Analyzed Compositions of Glass Pool Samples from DM10 Tests (wt%).	T-21
Table 4.5 XRF Analyzed Composition of Suction Samples (wt%).	T-23
Table 5.1 Results from DM10 Off-Gas Emission Samples.	T-24
Table 5.2 Concentrations [ppmv] of Selected Species in Off-Gas Measured by FTIR Spectroscopy During the DM10 Tests.	T-28
Table 6.1 Mass Balance for Volatile Species (%)	T-30
Table 6.2 Nitrogen Oxides and Carbon Monoxide Mass Balance.	T-31

## 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 3.1.a Feed and bubbling rates for DM10 Test 1 N.	F-3
Figure 3.1.b Feed and bubbling rates for DM10 Tests 2 N, 3 N, 9 N, and 1 OX.	F-4
Figure 3.1.c Feed and bubbling rates for DM10 Tests 2 OX, 3 OX, and 5 OX.	F-5
Figure 3.2.a Glass temperatures for DM10 Test 1 N.	F-6
Figure 3.2.b Glass temperatures for DM10 Tests 2 N, 3 N, 9N, and 1 OX.	F-7
Figure 3.2.c Glass temperatures for DM10 Tests 2 OX, 3 OX, and 5 OX.	F-8
Figure 3.3.a Plenum temperatures for DM10 Test 1 N.	F-9
Figure 3.3.b Plenum temperatures for DM10 Tests 2 N, 3 N, 9 N, and 1 OX.	F-10
Figure 3.3.c Plenum temperatures for DM10 Tests 2 OX, 3 OX, and 5 OX.	F-11
Figure 3.4.a Electrode power for DM10 Test 1 N.	F-12
Figure 3.4.b Electrode power for DM10 Test 2 N, 3 N, 9 N, and 1 OX.	F-13
Figure 3.4.c Electrode power for DM10 Test 2 OX, 3 OX, and 5 OX.	F-14
Figure 3.5.a Glass pool resistance during DM10 Test 1 N.	F-15
Figure 3.5.b Glass pool resistance during DM10 Tests 2 N, 3 N, 9 N, and 1 OX.	F-16
Figure 3.5.c Glass pool resistance during DM10 Tests 2 OX, 3 OX, and 5 OX.	F-17
Figure 3.6 Test Average glass production rate and resistance versus feed nitrate content.	F-18
Figure 3.7 Test Average glass production rate versus glass pool resistance.	F-19
Figure 4.1 DM10 product and target glass soda and silica concentrations determined by XRF.	F-20
Figure 4.2 DM10 product and target glass aluminum and iron oxide concentrations determined by XRF.	F-21
Figure 4.3 DM10 product and target glass zinc and zirconium oxide concentrations determined by XRF.	F-22
Figure 4.4 DM10 product and target glass magnesium and manganese oxide concentrations determined by XRF.	F-23
Figure 4.5 DM10 product and target glass lead and phosphorous oxide concentrations determined by XRF.	F-24
Figure 4.6 DM10 product and target glass chlorine and iodine concentrations determined by XRF.	F-25
Figure 4.7 DM10 product and target glass chromium and nickel oxide concentrations determined by XRF.	F-26
Figure 4.8 DM10 product and target glass arsenic and cadmium oxide concentrations determined by XRF.	F-27
Figure 4.9 DM10 product and target glass selenium oxide concentrations determined by XRF.	F-28
Figure 4.10 DM10 product glass, target glass, and feed sulfur oxide concentrations determined by XRF.	F-29
Figure 4.11 DM10 product and target glass rhenium oxide concentrations determined by XRF.	F-30

Figure 4.12	Measured iron oxidation state in glass pool samples versus the difference in carbon and NO <sub>x</sub> contents in the feed.	F-31
Figure 4.13	Measured iron oxidation state versus feed carbon/ nitrate mole ratio from this work and from previous tests.	F-32
Figure 4.14	Measured iron oxidation state in glass from this work and from previous tests versus the variable $D_r$ defined in Section 4.2.1.	F-33
Figure 4.15	Secondary phase with metallic appearance observed on top of suction sample taken after Test 5 OX.	F-34
Figure 5.1.a	Water measured in off-gas system exhaust, Test 1 N.	F-35
Figure 5.1.b	Water measured in off-gas system exhaust, Tests 2 N, 3 N, 9 N, and 1 OX.	F-36
Figure 5.1.c	Water measured in off-gas system exhaust, Tests 2 OX, 3 OX, and 5 OX.	F-37
Figure 5.2.a	Nitrogen oxides measured in off-gas system exhaust, Test 1 N.	F-38
Figure 5.2.b	Nitrogen oxides measured in off-gas system exhaust, Tests 2 N, 3 N, 9 N, and 1 OX.	F-39
Figure 5.2.c	Nitrogen oxides measured in off-gas system exhaust, Tests 2 OX, 3 OX, and 5 OX.	F-40
Figure 5.3.a	Carbon monoxide measured in off-gas system exhaust, Tests 2 N, 3 N, 9 N, and 1 OX.	F-41
Figure 5.3.b	Carbon monoxide measured in off-gas system exhaust, Tests 2 OX, 3 OX, and 5 OX.	F-42
Figure 6.1	Volatile retention in glass as a function of feed nitrate concentration.	F-43
Figure 6.2	Volatiles in melter exhaust as a function of feed nitrate concentration.	F-44
Figure 6.3	Volatile retention in glass versus the difference in carbon and NO <sub>x</sub> contents in the feed.	F-45
Figure 6.4	Volatiles in melter exhaust versus the difference in carbon and NO <sub>x</sub> contents in the feed.	F-46

### List of Abbreviations

ASME	American Society of Mechanical Engineers
BBI	Best Basis Inventory
CEM	Continuous Emissions Monitoring
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
FTIR	Fourier Transform Infrared Spectroscopy
HEPA	High-Efficiency Particulate Air Filter
HLW	High Level Waste
IC	Ion Chromatography
ICP	Inductively Coupled Plasma
LAW	Low Activity Waste
MACT	Maximum Achievable Control Technology
NIST	National Institute of Standards and Technology
NQA	Nuclear Quality Assurance
ORP	Office of River Protection
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
SEM	Scanning Electron Microscopy
TFCOUP	Tank Farm Contractor Operation and Utilization Plan
TOC	Total Organic Carbon
VSL	Vitreous State Laboratory
WTP	Hanford Tank Waste Treatment and Immobilization Plant
XRF	X-Ray Fluorescence

## **SECTION 1.0 INTRODUCTION**

With high nitrate feeds, the addition of reductants is necessary in order to control melt foaming. While the control strategy for Hanford Tank Waste Treatment and Immobilization Plant (WTP) Low Activity Waste (LAW) feeds is well developed, considerably less testing has been performed for WTP High Level Waste (HLW) feeds because of their lower projected nitrate contents. Sugar, which was used for this purpose at West Valley, has been selected as the baseline reductant for the WTP LAW vitrification system. The amount of sugar required increases with the amount of nitrates present in the feed and decreases with the amount of waste organics present in the feed, which themselves act as reductants. Excessive additions of reductants can be deleterious, leading to over-reduction of the melt and formation of sulfides and molten metals. Melter tests conducted with monitoring of the iron redox state in the glass have shown that carbon additions greater than the targeted amount can lead to overly reducing conditions in the glass melt [1-4]. Consequently, the oxidants and reductants in the feed must be suitably balanced. The basis for achieving this balance was developed by the Vitreous State Laboratory (VSL) of the Catholic University of America (CUA) and EnergySolutions for the vitrification of high-sodium-nitrate feeds at Savannah River's M-Area and has been successfully applied to the processing of a wide variety of simulated WTP LAW feeds over many years [1-25]. The empirically determined amount required to successfully control melt foaming without significantly reducing the glass melt was found to correspond to a ratio of 0.75 moles of organic carbon per mole of nitrate and nitrite. This approach has been employed as the baseline condition for all WTP LAW melter testing. More recently, adjustments to the algorithm were evaluated to account for differences in the reducing power of waste organics in comparison to sugar [26, 27], particularly for LAW streams that are high in organics [3], and address the differences between nitrate and nitrite [27]. This testing also demonstrated higher retention in the glass product of volatiles such as technetium and iodine at optimized concentrations of reductants in melter feeds [26, 27].

The baseline WTP HLW glass and melter testing work was done based on BNI flow sheet models that indicated low levels of waste oxidants (nitrates) and reductants (organics). Those projections lead to zero additions of sugar in the melter feed. As a result, no work was done to support the development of a redox control algorithm for HLW waste feeds. The limited testing with sugar additions conducted on the DuraMelter 1200 (DM1200) HLW Pilot Melter with HLW AZ-101 and C-106/AY-102 waste streams demonstrated sensitivity to small additions of sugar with respect to the iron oxidation state, cold cap behavior, and iodine retention in the glass product, as well as noble metal volatility and settling [28]. Another negative consequence of the reduced melt pool observed in those tests was the formation of corrosive metal sulfides. Other tests on the DM1200 with nitrated HLW AZ-101 feeds have shown the potential to control foaming and oxidation state in the glass with additions of sugar to the feed according to the baseline algorithm for LAW feeds [29]. Testing was also conducted on the DM1200 with HLW C-106/AY-102 wastes at bounding concentrations of regulated metals, halogens, nitrite, nitrate,

and oxalate to collect regulatory data [30]. These tests highlighted the facts that oxalate behaves very differently as a reductant than sugar and that the algorithm used for calculating sugar additions to LAW feeds for foaming and redox control is not universally applicable to HLW waste streams. Collectively, the limited amount of melter testing conducted with WTP HLW feeds with significant amounts of nitrates and waste organic compounds such as oxalate provide insufficient data to define the potential effects of these oxidants and reductants in HLW feeds. Since oxalate forms several insoluble salts, it is one of the few organics that can report to the HLW stream in significant amounts. Consequently, there is a need to test the impacts of variations in the amounts of nitrate and oxalate over the full range of potential flow sheet changes that are likely to be encountered during WTP operations, and to develop and test the strategy to control HLW glass redox over the expected range of WTP processing conditions.

This report provides results for a series of tests that were performed on the DM10 melter system with simulated C-106/AY-102 HLW in response to a Scope of Work provided by the Department of Energy's (DOE) Office of River Protection (ORP) [31]. The tests, which are described in the Test Plan for this work [32], employed simulated HLW feeds containing variable amounts of nitrates and waste organic compounds corresponding to maximum concentrations projected for Hanford HLW streams in order to determine their effects on glass production rate, processing characteristics, glass redox conditions, melt pool foaming, and the tendency to form secondary phases. Such melter tests provide information on key process factors such as feed processing behavior, dynamic effects during processing, processing rates, off-gas amounts and compositions, foaming control, etc., that cannot be reliably obtained from crucible melts.

## 1.1 Test Objectives

The principal objectives of this work were to investigate the effects of processing simulated Hanford HLW at the estimated maximum concentrations of nitrates and oxalates and to identify strategies to mitigate any processing issues resulting from high concentrations of nitrates and oxalates. A C-106/AY-102 HLW simulant and glass composition that had been processed previously on a continuously fed melter [28, 30, 33] was selected for these tests.

Specific objectives of these tests were to:

- Determine the effects of variable waste nitrate concentrations on the DM10 melter processing of a Hanford C-101/AY-102 HLW high-iron stream, including foaming, cold cap characteristics, glass redox, processing rate, off-gas characteristics, and formation of secondary phases.
- Determine the effects of variable waste oxalate concentrations on the DM10 melter processing of a Hanford C-101/AY-102 HLW high-iron stream, including foaming, cold cap characteristics, glass redox, processing rate, off-gas characteristics, and formation of secondary phases.

- Develop potential feed control strategies to mitigate potential negative consequences of processing high concentrations of waste nitrates and oxalates in a Hanford C-101/AY-102 HLW high-iron waste stream on the DM10 melter (e.g., foaming, poor cold cap characteristics, unacceptable glass redox, low processing rate, formation of secondary phases, etc.).
- Determine retention of volatiles such as rhenium, iodine, sulfur and halogens in the DM10 glass product at various waste concentrations of oxalate and nitrate.
- Characterize the chemical composition of each discharge glass and measure the iron oxidation state for glass from the end of each melter test for each feed composition.
- Monitor off-gas constituents (N<sub>2</sub>O, NO, NO<sub>2</sub>, NH<sub>3</sub>, CO<sub>2</sub>, CO, SO<sub>2</sub>) by Fourier Transform Infrared Spectroscopy (FTIR).
- Characterize the melter emissions (particulate, aerosol, and gaseous) to permit material mass balance across the melter for each oxalate and nitrate concentration.

## 1.2 Quality Assurance

Testing was performed according to the existing quality assurance (QA) 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) Nuclear Quality Assurance (NQA)-1, 2004; and DOE Order 414.1 C, Quality Assurance. The requirements of DOE/RW-0333P are not applicable to this work. The program is supplemented by a Quality Assurance Project Plan (QAPP) for ORP work [34] 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 [35].

## 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 Inconel 690 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  $0.02 \text{ m}^2$  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 high efficiency particulate air (HEPA) filters. The temperature of the cyclonic filters is maintained above  $150^\circ\text{C}$  while the HEPA filters are held above  $100^\circ\text{C}$  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.

### 1.3.4 DM10 System Sampling Points

A variety of sampling points are available on the DM10 system. The sampling points used in this work are as follows:

- *Melter Feed:* Samples of the melter feed taken from the parent feed batch to provide confirmation of the feed composition.
- *Glass Product:* Samples of the glass product taken from glass that is air-lift discharged into steel cans.
- *Glass Pool:* Glass samples taken directly from the glass pool ("dip" samples).

- *Melt Pool Floor*: Glass samples taken directly from the melt pool floor using a vacuum pump and ceramic tubes ("suction" samples).
- *Off-gas 1*: Isokinetic sampling of melter exhaust conducted at a point located immediately downstream of the film cooler.
- *Off-gas 2*: A sampling point located down stream of the HEPA filter was used for continuous emissions monitoring (CEM) by Fourier transform infra-red spectroscopy (FTIR) of a wide variety of gaseous species, including NO, NO<sub>2</sub>, N<sub>2</sub>O, CO, and SO<sub>2</sub>.

## 1.4 Experimental and Analytical Methods

The measurements and analyses that were performed in this work are detailed in controlled VSL technical procedures [35], 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 Feed

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 direct current plasma - atomic emissions spectroscopy (DCP-AES) analysis of solutions generated by microwave aided acid dissolution.

### 1.4.2 Compositional Analysis

Each glass sample was powdered and sieved to produce -200 mesh material for analysis by X-ray fluorescence spectroscopy (XRF). The analysis was performed on a PANalytical Axios max-Advanced XRF spectrometer. The spectrometer was calibrated over a range of glass compositions using standard reference materials traceable to National Institute of Standard and Technology (NIST), as well as waste glasses including the Argonne National Laboratory-Low Activity Waste Reference Material (ANL-LRM), the Defense Waste Processing Facility-Environmental Assessment (DWPF-EA) Glass, and Hanford WTP glasses. An additional analysis was also conducted using the XRF in order to quantify the very low levels of rhenium more precisely. This method involved analyzing a specific rhenium spectral line and the associated background for extended periods of time. Analysis by XRF provides data for most glass components of interest except lithium and boron, which were determined by total acid dissolution of ground glass samples in HF/HNO<sub>3</sub> 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  $\text{Fe}^{2+}$  and  $\text{Fe}^{3+}$  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  $^{57}\text{Co}$  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  $\text{Fe}^{2+}$  and  $\text{Fe}^{3+}$  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  $\text{Fe}^{2+}$  to  $\text{Fe}_{\text{total}}$  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 channel. 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  $\text{Fe}^{2+}$  and  $\text{Fe}^{3+}$  peaks.

### 1.4.4 Melter Exhaust Sampling and Analysis

The melter exhaust was sampled for metals and 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 C-106/AY-102 Waste Simulant

The HLW simulant used for these tests is based on the C-106/AY-102 composition, which has been extensively tested previously [28, 30, 33, 36], with minor adjustments to accommodate a prescribed set of spikes used in previous HLW MACT tests [30]. This section describes the derivation of the C-106/AY-102 HLW simulant and the modifications made to arrive at the HLW simulant for the present tests.

Formulation of the C-106/AY-102 waste simulant is based on the BNI Test Specification [37], which made use of inventory data from the TFCOUP [38], calculated data from Steady-State ACM Flow sheet modeling, and analytical data on Cs- and Tc-removal eluates from LAW pretreatment<sup>1</sup>. In addition, products from Sr/TRU removal for pretreatment of LAW were also included in the waste blend. Table 2.1 summarizes the compositions of the different waste streams and the blended waste.

The composition of the C-106/AY-102 Envelope D solids (Stream FRP02) is based on the inventory data found in Revision 3A of the Tank Farm Contractor Operation and Utilization Plan (TFCOUP) [38]. As seen in Table 2.1, in addition to updated information, Revision 3A of the TFCOUP also provides information on minor components that were not included in earlier revisions [39] and the Best Basis Inventory (BBI) database (e.g., cadmium). The use of other data sources (e.g., HLW Feed Staging Plan [40]) to supplement the TFCOUP, as was done in previous tests, was therefore not necessary. The ACM model calculates the composition of the recycle stream (PWD01), which is then blended with the Envelope D solids based on the expected daily processing rates (i.e., 3.79E+04 lb/day for Envelope D solids and 1.31E+03 lb/day for the recycle stream on a dry solid basis). The resulting material is concentrated and pretreated through caustic leaching/water washing and ultra-filtration to produce the pretreated HLW solids. The separation factors due to caustic leaching and ultra-filtration are given in Table 2.1. Note that some of the separation factors are larger than unity (many of which were ignored in derivation of the waste composition, which is used as-provided [37] in the present work) and that the ACM model predicts mass increases for Fe and Zr after ultra-filtration (75 lb/day and 68 lb/day, respectively) [37].

To complete the C-106/AY-102 simulant formulation, the pretreated HLW solids are blended with wastes from LAW pretreatment. Similar to the blending scenario used in Part B1 tests [41], Sr/TRU removal products from pretreatment of Envelope C wastes have been added, although the amounts of Sr and Mn (449 lb/day and 499 lb/day, respectively) blended are

<sup>1</sup> It is recognized that technetium removal in pretreatment is no longer part of the WTP flow sheet. However, the impact of this stream is very small. The same simulant composition was used for the present work in order to permit direct comparison to the results from the earlier tests.

considerably less than those used in earlier tests, which results in lower concentrations of SrO and MnO in the current test glass (e.g., 0.92 wt% vs. 7.35 wt% for SrO) [41]. Analytical data on eluates from Cs- and Tc-removal on an Envelope B sample (AN-102) [42] provide the compositional bases for the respective ACM-model feed streams CNP12 and TEP12, although that was not the case for the Sr/TRU stream. The blending proportions are determined by the projected daily processing rate of sodium in the eluates (i.e., 2.02E+01 lb/day for Cs-removal and 9.14E-01 lb/day for Tc-removal). It can be seen in Table 2.1 that waste blending primarily leads to increase of manganese, strontium, sodium, chloride, and nitrate in the HLW simulant.

The calculated composition of the blended HLW solids (HLP09b), which is shown in Table 2.1, lists a total of 55 components. A few of the components, however, were left out of the blended solid composition, as provided by the WTP Project, because of missing separation factors, low concentrations and other unspecified reasons (e.g., Be, Co and Mo) [37]. In addition, similar to the approach taken in previous melter tests, radionuclides, noble metals (including silver), and minor components (< 0.02 wt% in glass on an oxide basis) are omitted from the C-106/AY-102 simulant formulation. Cesium and iodine were spiked in the C-106/AY-102 simulant for analytical purposes for some of the previous tests [28, 33] with this waste; iodine is retained and the cesium spike is replaced by rhenium for the current tests since that component is considerably more sensitive to redox changes. The resulting HLW simulant formulation for the C-106/AY-102 waste, which is given in Table 2.2, consists of 33 components, 29 of which are non-volatile.

Modifications to the base formulation of the C-106/AY-102 simulant primarily involve addition of the components to be spiked, at their respective levels, and then renormalizing the remaining components. A total of thirteen constituents were spiked and they are listed in Table 2.2, together with their respective spike levels as the total amount targeted in the glass product [43]. Ten of the spikes are non-volatile and are expected to be retained in glass; they total 1.391 wt% (with respect to glass). Since the spike levels are defined with respect to the target glass composition, derivation of the HLW simulant for the present tests starts with the reference glass developed for the C-106/AY-102 base simulant, which is discussed in the next section. The ten non-volatile spike components are fixed in the reference glass (and simulant) composition while the other components are re-scaled to make up 98.609 wt% of the glass composition; sulfate is included in the feed at a level corresponding to 0.15 wt% of SO<sub>3</sub> in glass if volatilization is ignored. Similarly, iodine as iodide and rhenium as perrhenic acid are included in the feed at levels corresponding to 0.1 and 0.05 wt% of I and Re<sub>2</sub>O<sub>7</sub> in glass, respectively, if volatilization is ignored. The new glass composition can then be used to arrive at the HLW simulant composition by removal of the glass forming additives. The resultant HLW simulant composition is given in Table 2.2. Finally, the volatile carbonate, which is not a spike component, is targeted in the HLW simulant at the same concentration as in the base simulant.

## 2.2 C-106/AY-102 Glass and Feed Formulation

After definition of the composition for the C-106/AY-102 HLW base simulant, glass formulations were developed and tested at VSL to support previous melter tests. The glass

composition selected as the basis for those tests, HLW98-86, is presented in Table 2.3. The same glass formulation, with modifications to incorporate the spikes (see above) [30], was used for the present tests. On an oxide basis, HLW98-86 has a total waste loading of 27.75 wt%, of which 25.13 wt% is Envelope D waste. These can be compared with the respective values of 51.00 wt% and 39.42 wt% for HLW98-34, the C-106/AY-102 reference glass used in Part B1 [41]. The difference is primarily due to the presence of much more Na<sub>2</sub>O in the Part B1 simulant (20.61 wt% vs. 2.11 wt% for the current simulant). The current target glass (HLW98-86) is also different from HLW98-34 in that it meets the contract minimum component limit by incorporating 12.56 wt% of Fe<sub>2</sub>O<sub>3</sub>, instead of > 21 wt% of (Al<sub>2</sub>O<sub>3</sub>+Fe<sub>2</sub>O<sub>3</sub>+ZrO<sub>2</sub>) [41].

Crucible melts of HLW98-86 and related formulations have been prepared and tested to determine that the target glass meets the necessary processing requirements. Heat treatment of HLW98-86 at 950°C for over 70 hours results in a homogeneous dark brown glass that is free of secondary phases. The viscosity and electrical conductivity measured for HLW98-86AG, which has the same composition as HLW98-86 except with Ag<sub>2</sub>O excluded, are 44 P and 0.36 S/cm, respectively, at 1150°C. Finally, the normalized PCT leach rates of HLW98-86 are (in g/(m<sup>2</sup>-day)) 0.058, 0.047, 0.046 and 0.028, respectively for B, Li, Na and Si; these values can be compared with those for the reference glass, Defense Waste Processing Facility – Environmental Assessment (DWPF-EA): 1.17, 0.71, 0.80 and 0.27. The target glass formulation for previous DM1200 tests with the C-106/AY-102 simulant [28, 33] differs slightly from HLW98-86, with the removal of silver and the addition of small amounts of cesium and iodine.

The glass formulation used for the MACT HLW tests [30] is very similar to the target formulation, as shown in Table 2.3. The increase in chromium may increase the crystal content at the reference heat treatment temperature (950°C) and the TCLP leachate concentrations of some of the components are likely to be higher as a result of adding the MACT spikes. However, other properties, such as viscosity, electrical conductivity, and PCT response are likely to be unaffected.

The additional constituents required to form the target test glass from the MACT HLW simulant are aluminum, boron, lithium, sodium, silicon, and zinc. They were added in the same proportions as those used in HLW98-86 (with the exception of lithium, which was added at 2.993 wt%, with respect to glass (oxide basis), compared to 3.01 wt% in HLW98-86). The raw materials that are the sources for the glass-forming additives were selected based on previous testing and per direction of the WTP Project. Table 2.4 lists the starting materials and amounts required to produce the target C-106/AY-102 simulant and melter feed. Note that all of the TOC is assumed to be oxalate. Additionally, the recipe in Table 2.4 corresponds to only 2.18 (g/100 g waste oxide) of carbonate present in the simulant, instead of the targeted 4.65 (g/100 g waste oxide). This discrepancy in carbonate does not impact the tests since much greater amounts are present in the glass forming additives. The undissolved solids content in the *simulant* is assumed to be 20 wt%, which is equivalent to 21.49 wt% total solids, based on the data from AZ-102 testing [44]. Compared with the previously tested C-106/AY-102 HLW simulants [28, 33], notably more water is required to produce a slurry with 21.49 wt% total solids for the MACT HLW simulant because of the increased amounts of volatile components (i.e., oxalate and nitrate). This in turn has the effect of reducing the glass yields for the current testing. The

theoretical glass yield of the resulting MACT feed is 312 g of glass/kg of feed (or about 400-460 g/l of feed, depending on feed density, measured as 426 g/l in previous tests [30]).

### 2.3 Oxalate and Nitrate Concentrations

Simulated WTP HLW feeds with only a limited range of nitrate and oxalate concentrations have been subjected to melter testing in previous work. A list of the melter tests conducted, the nitrate and oxalate concentrations used, and the WTP contract limits for HLW streams is provided in Table 2.5. Initial projections of nitrate and organic carbon concentrations were very low and the form of organic carbon was not measured and assumed to be oxalate [28, 33, 36]. Subsequently, WTP estimated the maximum possible concentration of nitrate at 30 g per kg of glass produced [45]. WTP later supplemented this estimate with projections of maxima for other waste constituents including nitrite and TOC for HLW feeds to be processed to collect regulatory data [43]. Tests performed with all these feeds did not result in any processing problems such as foaming or an overly reduced glass. However, the WTP contract maximum concentrations are considerably higher than these values: 36 and 11 grams per 100 grams waste oxide for waste concentrations of nitrogen oxides (nitrate + nitrite) and total organic carbon [46], which corresponds to 99.9 grams and 30.53 grams of nitrogen oxides and total organic carbon, respectively, per kilogram of glass for the C-106/AY-102 HLW98-96 glass composition. Action limits for carbon in HLW that are higher than the WTP contract are given as 10 wt% TOC [47] which corresponds to 225.7 grams total organic carbon for the C-106/AY-102 HLW98-96 glass composition.

Nitrate and TOC concentrations per unit mass of glass would be correspondingly higher for higher waste loading formulations, such as for the HLW04-09 glass formulation for C-106/AY-102 waste previously processed on the DM1200 [36]. For further comparison, LAW feeds recently processed on the melters at VSL had nitrate concentrations between 8 and 195 g per kg glass and total nitrogen oxide concentrations between 31 and 257 g per kg glass [26, 27].

In view of this information, the feeds used in the current tests were designed to span the WTP waste envelope maximum value of 99.9 g nitrogen oxide per kg glass and to significantly exceed the values tested previously; thus the feeds for the present HLW tests were targeted at 60, 90, and 120 g nitrogen oxide per kg glass, which also covers the lower end of the range for LAW feeds.

No tests with HLW feeds have been conducted with higher feed TOC concentrations in the absence of nitrate, which is likely to present the worst case with respect to glass redox. Therefore the initial test in this work was conducted at previous estimates of oxalate maximum concentrations without the inclusion of nitrate in the feed. The carbon concentration was then increased to 50 and 75 g per kg glass. Even though some work was done previously with sugar additions to HLW feeds [28], it was possible to test significantly higher ratios of total carbon to nitrogen oxides in the present work than was previously tested with sugar because oxalate is a less effective reductant than sugar.

## 2.4 Analysis of Melter Feed

Melter feed was produced by NOAH Technologies Corporation, the supplier of simulant and feed samples used in previous testing on the DM100 and DM1200 melter systems. Feed was received without oxalic and nitric acids to enable the subsequent adjustment of these constituents for each test at VSL. As-received feed was sampled and analyzed to determine physical properties and confirm chemical composition. Table 2.6 provides measured feed properties and a comparison of the analyzed chemical composition to the target. The measured glass conversion ratio and feed water content were used to calculate the amount of additives required to achieve the target feed solids content for feed used in the melter tests. Resulting feed recipes for each test targeting various nitrate and oxalate concentrations are provided in Table 2.7.

The XRF and DCP analyses confirm the composition of the as-received feed. The results generally 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 aluminum oxide in the feed produced by NOAH had deviations of greater than 10% from target. The absolute deviation for aluminum was less than 0.75 wt%, and was not adjusted because it was not expected to affect the objectives of the current tests since the aluminum concentration remained consistent throughout all of the tests. Boron and lithium concentrations measured by DCP were within three and eight percent of the target for feed produced from NOAH, 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, La, Ni, P, Sb, S, Ti, and Zr were observed in the feed at levels slightly above their respective targets. Similarly, several elements that were not target constituents (Ba, Bi, Ce, K, Sn) were identified at low concentrations in feed made by NOAH. The volatile trace element sulfur was measured at concentrations higher than the target concentration, suggesting that sulfur was present as a contaminant in the glass forming additives or chemicals used to make the simulant. Overall, however, given their low target concentrations, these surpluses are not expected to have any significant effect on glass properties or volatile retention. Halide and rhenium concentrations are below target in feed samples due to volatilization during crucible melting, as expected.

## SECTION 3.0 MELTER OPERATIONS

Melter tests were conducted with HLW C-106/AY-102 waste simulants containing various amounts of nitrate and oxalate on the DM10 between 4/19/12 and 5/3/12. These tests produced over 165 kg of glass from over half a metric ton of feed. The nominally 24 hour in duration tests were distinguished by differences in feed composition as follows:

- Test 1 N: 60 g nitrate/kg glass; no processing problems were observed, therefore the nitrate concentration was increased in the following test.
- Test 2 N: 90 g nitrate/kg glass; no processing problems were observed therefore the nitrate concentration was increased in the following test.
- Test 3 N: 120 g nitrate/kg glass.
- Test 9 N: 120 g nitrate/kg glass + sugar (18.56 g C/kg glass).
- Test 1 OX: Oxalate (25 g C/kg glass); no processing problems were observed, therefore the oxalate concentration was increased in the following test.
- Test 2 OX: Oxalate (50 g C/kg glass)
- Test 3 OX: Oxalate (50 g C/kg glass) + 30 g nitrate/kg glass
- Test 5 OX: Oxalate (75 g C/kg glass) + 30 g nitrate/kg glass

Attempts were made to replicate the melter configuration and operating conditions used for previous melter tests with HLW simulants [28, 29, 33, 36, 44, 48-57]. 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 and the feed rate was adjusted to provide the desired complete cold cap. Power was supplied to the electrodes to maintain a glass temperature of 1150°C throughout the tests. All tests targeted the same glass composition (HLW98-86 with spiked minor constituents used in regulatory tests [30], as described in Section 2) with the difference in feed composition in each test being the amount of added nitrate and carbon as either oxalate or sugar. This approach permitted the direct comparison of each nitrate and carbon concentration with respect to feed processing characteristics such as foaming, glass production rate, glass oxidation state, and volatile retention in the glass product at constant operating conditions.

The feed processed easily throughout the tests without clogs and resulting disruptions to melter operations. The cold cap was observed through a view port on top of the melter over the duration of the tests. No significant foaming was visible during the tests; however, there were significant changes in processing rate, as discussed below. Changes in feed rate were made in each test in response to observations of the cold cap: increases in feed rate when openings in the cold cap were increasing in extent and decreases in feed rate when openings were decreasing in extent or not visible, indicating that feed was piling up on the cold cap surface faster than it was being incorporated into the glass pool.

### 3.1 Melter Operations Data

Production rates, run conditions, and measured melter parameters for the eight 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.a – 3.5.c. The test average production rates varied by a factor of almost two, ranging from about 700 kg/m<sup>2</sup>/day at higher nitrate feed contents to 1300 kg/m<sup>2</sup>/day in tests without nitrate and added carbon. Glass production rates plotted in Figures 3.1.a - 3.1.c illustrate the lowest production rates in Tests 2 N and 3 N while processing feed rich in nitrate; an increase in production rate while transitioning from Test 3 N to 9 N as sugar is added to the high nitrate feed; the highest production rate in Tests 1 OX and 2 OX while processing feed without nitrates but which contains oxalate; decreasing production rate while transitioning from Test 2 OX to 3 OX as nitrate is added to the feed; and increasing production rate while transitioning from Test 3 OX to 5 OX as the oxalate concentration in the feed is increased. The relationship between feed nitrate content and production rate is further illustrated in Figure 3.6. Generally, the glass production rate decreases with increasing feed nitrate content and tests with the same nitrate content have higher production rates at higher feed organic carbon content.

The measured glass resistance also displayed in Figure 3.6 shows the opposite trend with feed nitrate content. The glass production rate is strongly correlated with the glass resistance and varies approximately linearly, as shown in Figure 3.7; on average, the glass production rate decreases by about 0.1 kg/hr with an increase of 0.01 ohms in melt pool resistance for the same feed with different nitrate and oxalate contents. Since the glass composition and melt pool temperature are essentially fixed during these tests, the most likely explanation for the observed variation in the melt pool resistance is foaming within the glass pool, which is evidently not visible from the cold cap observation made through the melter view port. Such foaming would introduce gas inclusions within the melt pool which would increase the electrical resistance that is measured between the electrodes. Typically, the generation of gas bubbles in the melt is a result of either redox changes or gas solubility changes due to local temperature fluctuations, or both. Foaming with highly oxidizing feeds is well-known and indeed is the reason for addition of reductants such as sugar in many high-nitrate flow sheets (e.g., West Valley, M-Area, WTP LAW). It is also likely that if gas bubbles are present between the electrodes, as is clearly indicated by the resistance, then the rising bubbles would accumulate under the cold-cap, which would hinder heat transfer and reduce the glass production rate, as was observed; it is quite possible that such bubbles under the cold cap would not be visible from the view port. In addition, the entrained gas and foamy melt pool would also tend to retard heat transport to the cold cap, which would decrease the melt rate. This effect of nitrates on both melt rate and measured melt pool resistance can be partially mitigated by the addition of organic carbon to the feed.

Glass temperatures (2 and 4 inches from the melt pool floor) averaged within 10°C of the target glass temperatures throughout all the tests. The glass temperature 4 inches from the melt floor averaged about 10°C less than the temperature 2 inches from the melt floor and 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. Electrode temperatures were 60 to 90°C lower than the

temperature of the glass pool. The discharge temperature was maintained above 1050°C throughout the tests to facilitate glass pouring. Test average plenum temperature measurements were between 496 and 560°C in the thermowell and about 30 to 60°C cooler at the exposed thermocouple. This difference is opposite to that in many previous tests, indicating that the exposed thermocouple was partially shielded or that the thermowell was closer to a bubbling outlet.

The gas temperature at the film cooler, which averaged between 248-284°C, is dependent on the plenum temperature, the amount of added film cooler air, and the temperature of the added film cooler air. The glass pool resistance mostly ranged between 0.16 and 0.21 ohms and increased with feed nitrate content. Test average bubbling rates equaled the target of 1.0 lpm for all eight tests. A vacuum of about 1 inch of water was maintained on the melter throughout the tests. Power supplied to the melter increased from near 4 kW to 6 kW with increasing feed rates. The trend is reversed when the power is normalized to glass production rate, with the lowest production rates having the highest power usage per unit glass produced (Tests 1 N, 2 N, 3 N: 6.0 – 7.6 kWhr/kg glass) and the highest production rates having the lowest power usage per unit glass produced (Tests 1 OX, 2 OX: 5.1 – 5.3 kWhr/kg glass); this is a consequence of the power that is required to idle the melter at constant temperature at zero glass production rate.

## SECTION 4.0 GLASS PRODUCTS

Over one hundred and sixty 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 by removing sufficient glass from the top of each can for total inorganic analysis. Product glass masses, sample names, and discharge dates 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, sieved, 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 analysis (see Section 2.4). Of the oxides with a target concentration of one percent or greater, the average XRF values for aluminum, manganese, and zinc oxides had deviations of greater than 10% from target values. Deviations from target for the average melter glass analysis were larger than measured in the as-received feed for manganese and zinc as a result of the initial glass pool having much less manganese and zinc than in the target composition. Zinc and manganese deviations from target for the glass sampled at the end of testing were less than half an absolute weight percent and therefore are not expected to have any significant effect on glass properties. As observed in the feed samples, the glass analyses indicated that aluminum was somewhat above target in the feed but as noted in Section 2.4, it was not adjusted because it was not expected to affect the objectives of the current tests since the aluminum concentration remained consistent throughout all of the tests. Minor constituents such as calcium, chromium, nickel, phosphorous, sulfur, titanium, and zirconium were over represented in the glass product at about the same frequency and magnitude as in the feed samples (see Section 2.4). The volatile trace elements rhenium, chlorine, and iodine were measured at concentrations lower than their respective targets, as expected.

The discharge glass compositions over the course of testing are illustrated in Figures 4.1-4.11. Most oxides approximate their respective target or analyzed feed values and varied little during testing after three melt pool turnovers had been completed for each composition. At the beginning of testing, oxides of As, Cd, La, Li, Mg, Mn, Nd, Sb, Si, and Zn increase in concentration at the expense of B, Ca, Fe, Na, Ni, P, Pb, S, and Zr as the glass pool transitions to the spiked target HLW C-106/AY-102 composition. Major oxides such as those of silicon, sodium, and iron reach steady state concentrations after 20 kg glass production and vary little over the remainder of the tests. Aluminum and phosphorus increase in concentration over the

two day idling periods between Tests 1 N and 2 N and 1 OX and 2 OX (20 and 100 kg glass production) while decreases due to volatilization were observed for chlorine, rhenium, and selenium during the same period. As expected, halogen, rhenium and selenium concentrations were well below target concentrations as a result of volatilization from the cold cap and glass pool. In Figures 4.8 and 4.10, the measured feed concentrations are displayed in addition to the target concentrations for sulfur, cadmium, and arsenic; the analyte concentrations measured in the glass more closely approximate the measured feed concentrations, particularly considering the known volatility of these elements. The target and measured rhenium concentrations in the glass are plotted in Figure 4.11. The rhenium retention in glass averaged around 30% for these tests but, in contrast to observations with LAW feeds that showed increased rhenium retention in the glass under more reducing conditions, little, if any, increase in rhenium retention was observed under more reducing conditions in the present tests.

## 4.2 Glass Pool Samples

Glass pool dip and suction 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, determine the iron oxidation state in the glass pool, and to determine the melt level to quantify the amount of glass in the melt pool. A list of all dip and suction 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 taken at the end of each of the eight tests. Secondary sulfate was observed on the initial dip sample prior to testing; the melt pool was bubbled then re-sampled to verify that the secondary phase was removed prior to the start of testing. The analysis of the glass pool samples corroborates the composition of the discharge glasses, as shown in Table 4.4. The differences between the rhenium concentrations measured in the glass pool samples and those measured in the respective discharge glass samples were all less than 0.006 wt% oxide.

### 4.2.1 Redox State

The redox state of the glass was assessed through measurements of the iron oxidation state in glass samples using Mossbauer spectroscopy. No reduced iron was detected in glass from the end of tests processing feed containing greater than 30 g nitrate per kg glass (Tests 1 N, 2 N, 3 N, and 9 N), which is consistent with the oxidizing nature of nitrate and the relatively low concentration of reductants. Divalent iron concentrations increased with increased amounts of feed carbon, as shown in Figure 4.12. The concentrations of divalent iron from previous tests conducted with nitrate, oxalate, sugar, and the HLW HLW98-96 composition (see Table 2.5) are displayed together with the results from the present work in Figure 4.13 over the range of feed carbon to nitrate ratios tested. Higher amounts of reduced iron were found in tests with sugar in place of oxalate confirming the previous observations that sugar is a more effective reductant than oxalate [3, 26, 27]; consequently, the algorithm used to determine sugar additions to high nitrate feeds (such as WTP LAW feeds) would need to be modified for oxalate. At low nitrate concentrations, lower concentrations of reduced iron were found at higher carbon to nitrate ratios

than for high nitrate feeds at lower carbon to nitrate ratios; this suggest that the algorithm used to determine sugar additions to high nitrate feeds (such as WTP LAW feeds) may need to be modified for low nitrate feeds.

The specific redox reactions that occur during feed processing in a melter, and their stoichiometry, are not known nor easily determined. Therefore, the present effort focused on the development of an empirical correlation between glass melt redox and feed chemistry that can be used to control HLW processing at the WTP; this type of approach is the baseline for the WTP LAW vitrification system and has been used to successfully control the production of about 11 million pounds of glass at the EnergySolutions LAW Pilot Melter and the Savannah River M-Area vitrification facilities. In addition, from previous [6] and current test results, it is clear that carbon from sugar is a more effective reductant than carbon from other sources such as oxalic acid. Thus, in an initial effort to develop an improved correlation of the data shown in Figure 4.13 the data were plotted against a new variable defined as:

$$D_r = N - a (C_s + b C_o),$$

where  $N$  is the number of moles of nitrate per kilogram of glass,  $C_s$  is the number of moles of carbon from sugar per kilogram of glass,  $C_o$  is the number of moles of carbon from oxalate per kilogram of glass, and  $a$  and  $b$  are constants; for the present purpose, the small amounts of nitrites that are present are counted as nitrate. The motivation for this variable is the idea that nitrates and organic carbon will first react in the cold cap and any excess will then react with the feed and glass component to reduce or oxidize the resulting glass melt. Thus, the constant  $a$  is an effective stoichiometric factor characterizing the reaction between nitrate and organic carbon in the cold cap while  $b$  is a factor that captures the relative reducing power of carbon from sugar versus carbon from oxalate. The extent of oxidation or reduction of the melt should then vary with the amount of un-reacted nitrate or organic carbon that is available to reduce or oxidize the glass melt per kilogram of glass, which is represented by the variable  $D_r$ . It would be expected that the redox state of the glass would become more oxidizing as  $D_r$  becomes more positive and more reducing as  $D_r$  becomes more negative. Figure 4.14 shows the same data as in Figure 4.13 now plotted against  $D_r$ , where the constants  $a$  and  $b$  have been set at values that give a reasonably smooth trend, which gives  $a = 1.0$  and  $b = 0.22$ . The value of  $b$  is consistent with what one might estimate directly from the data shown in Figure 4.13 while the value of  $a$  would suggest a one-to-one reaction of nitrates and organic carbon in the cold cap. This value is close to but somewhat greater than the value of 0.75 that is used in the WTP LAW algorithm for sugar additions and which is the empirically determined amount required to successfully control LAW melt foaming without significantly reducing the glass melt.

The trend line in Figure 4.14 is given by

$$\text{Fe}^{2+}/\text{Fe total} = A \exp(B D_r) + 0.01,$$

where  $A = 0.01613$ , and  $B = -2.2595$ . It might be expected that the trend line shown in Figure 4.14 would shift with changes in the amount of iron in the glass composition<sup>2</sup> but that was constant for all of the data used in the present analysis; however, that effect should be investigated in any further development of this approach.

The correlation described above can be used to determine the amount of sugar or nitrate that should be added to the melter feed to control melt foaming and avoid overly reducing conditions in the melt pool, as discussed in Section 7.

#### 4.2.2 Suction Samples

Samples from the bottom of the melt pool were taken after Tests 3 OX and 5 OX to assess whether there was any evidence of segregation of material to the bottom of the melter under reducing conditions. The glass extracted from the bottom of the melter with ceramic tubes was visually inspected for secondary phases and analyzed by XRF for oxide composition, Mossbauer spectroscopy for iron oxidation state, and Scanning Electron Microscopy coupled with Energy Dispersive X-ray spectroscopy (SEM-EDS) for secondary phases. No secondary phases were observed in the suction sample after Test 3 OX. A metallic film was observed along the sides of suction tube in the samples after Test 5 OX, as shown in Figure 4.15. The material was at the top of the suction tube, indicating that it resided on the melter floor. SEM-EDS analysis showed that secondary phase was nickel copper selenide. Compositional analysis of the glass from both suction samples is provided in Table 4.5 and indicates the composition to be very similar to dip and discharge glasses from the same tests. Exceptions are elevated concentrations of selenium, copper, and nickel in the sample taken after Test 5 OX, indicative of incorporation of the macroscopic secondary phase. The measured divalent iron concentration in the suctioned glass is about ten relative percent below the amount measured in the bulk glass. SEM-EDS analysis of the glass showed 0.1 volume percent secondary phases in the form of chromite.

<sup>2</sup> Or, more specifically, the total number of moles per kilogram of glass of reducible species that are able to appreciably shift their valance state over the range of oxygen fugacities of interest, which for most practical purposes for HLW compositions is typically limited to iron and possibly manganese.

## SECTION 5.0 MONITORED OFF-GAS EMISSIONS

### 5.1 Particulate Sampling

The melter exhaust was sampled for metals and 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. Melter emission fluxes are compared to feed fluxes in Table 5.1. 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  $\mu\text{m}$  heated filter. All eight samples are within the 90 – 110% limits for isokinetic sampling.

Particulate emissions from the melter constituted 0.33 to 0.94 percent of feed solids during tests. The level of carryover for these tests is mostly within the range measured previously for HLW C106/AY-102 simulants processed on the DM100 [53] (0.61 - 0.81 percent) and on the DM1200 (0.54 - 1.8 percent [28]; 0.67 percent [33]; and 0.75 percent [36]). The carryover of solids was generally higher during tests processing feed with nitrates (0.47 - 0.94 percent) than tests without nitrates (0.33 - 0.56 percent), which is consistent with previous DM1200 tests processing C106/AY-102 simulant at two different nitrate contents [28]. No trend for elemental particulate emissions was evident with respect to feed nitrate or carbon content. The feed element emitted at the highest rate was iodine with up to all feed iodine being present in the melter exhaust, mostly as a gas. Rhenium, selenium, chlorine, fluorine, and sulfur also exhibited significant carryover with 20 to 70 percent of the respective amounts in the feed present in the exhaust. Other elements exhibiting volatile behavior include chromium, cadmium, and boron with 0.5 to 11 percent of feed measured in the exhaust. 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; larger scale melter tests typically provide more reliable data in this respect. Boron, halogens, selenium, 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.

## 5.2 Gases Monitored by FTIR

Melter emissions were monitored in each test for a variety of gaseous components, most notably CO and nitrogen species, by Fourier Transform Infra-Red Spectroscopy (FTIR). The off-gas system temperature is maintained well above 100°C beyond the sampling port downstream of the HEPA filter to prevent analyte loss due to condensation prior to monitoring. A summary of average concentrations and concentration ranges monitored during each test is provided in Table 5.2. The concentrations of water, nitrogen oxides, and carbon monoxide, which are the species most affected by changes in feed composition, are plotted in Figures 5.1.a - 5.3.b. The analytes listed in Table 5.2 are those that were thought likely to be observed during the test based on previous work; no other species were detected in the off-gas stream by FTIR. Monitored emissions varied with the feed nitrate, oxalate, and sugar concentrations in each test, and the feed rate resulting from those feed concentrations. The percent moisture in the exhaust was the highest in tests with highest feeds rates, averaging 2.9 and 2.6 percent in Tests 1 OX and 2 OX, and lowest in tests with the lowest feeds rates, averaging 1.8 and 1.5 percent in Tests 2 N and 3 N. Nitrogen oxide concentrations increased with increasing feed nitrate concentrations and decreased with increasing feed carbon concentrations, as expected. Similarly, carbon monoxide and carbon dioxide concentration increased with increasing feed carbon content. The most abundant nitrogen species monitored was NO, which is consistent with previous tests in which nitrates and nitrites were present in the feed. Ammonia is a known byproduct of nitrate decomposition in the presence of sugar and, as expected, it was detected only during Test 9 N, which was the only test with sugar and nitrates present in the feed. Consistent with the low feed chlorine and fluorine concentrations, the amounts observed using the Method 5-type sampling discussed above, and the air dilution in between the two sampling points, no HF or HCl were observed by FTIR. The relatively large variations in emissions that are evident in the FTIR data over the course of each test segment are due in part to changes in the melt pool cold cap.

## SECTION 6.0

### MASS BALANCE FOR VOLATILES IN GLASS AND EMISSIONS

Results from glass and melter exhaust analysis were compared to the feed analysis or target compositions to complete a mass balance around the melter for each feed composition. The calculated mass balance for spiked toxic metals, halogens, sulfur, and rhenium as a surrogate for technetium for each test is provided in Table 6.1. The analyzed concentrations from feed sample analysis were used except for halogens, selenium, and rhenium, which significantly volatilize during crucible melting used in the analytical process.

Excellent mass balance closure was achieved for the toxic metals As, Cd, Cu, Pb, and Sb with all recoveries falling between 80 and 120% and most falling between 90 and 110%. A good mass balance was also obtained for chromium through the majority of the tests although excesses were observed for all tests due presumably to chromium leached from melter components. A good mass balance for halogens, sulfur, rhenium, and selenium was obtained only for a limited number of tests due primarily to uncertainty in the feed concentrations. Total sulfur recoveries were between 113 and 146 percent using the analyzed feed sulfur content, which was higher than the target concentration. However, the actual feed sulfur concentration was undoubtedly higher than the analyzed concentration due to losses during crucible melting. Total recoveries for chlorine, iodine, rhenium, and selenium were all below 100% except for an iodine and a chlorine outlier. Because of the low concentrations of these species, the absolute amounts responsible for the lack of closure are only 0.04 wt% Cl, 0.02 wt% I, 0.01 wt% Re<sub>2</sub>O<sub>7</sub>, 0.08 wt% SeO<sub>2</sub>, and 0.04 wt% SO<sub>3</sub> on a glass basis, which are probably associated with batching errors and, in the case of sulfur and chlorine, trace level contamination of feed. Deviations of this magnitude or greater have been measured for halogens and sulfur in several as received LAW feeds [58].

The distribution of volatile components between the glass and the melter exhaust varied widely over the various elements tested. On average, over 90% of the As, Cd, Cr, Cu, Pb, and Sb that was fed into the melter was retained in the glass product while less than a third of the feed Re, I, and Se was found in the glass. The average retentions in glass for the most volatile constituents when normalized to total recovery were Cl – 70%, I – 23%, Re – 37%, Se – 23%, and S – 65%. These levels of retention are consistent with previous tests for most of these elements. The rhenium retention is at the low end of a wide range of LAW feed and glass compositions [27] and higher than the 21-30% measured while processing a high iron HLW through the DM10 [59]. Together, these results suggest that rhenium retention may be lower for HLW feeds than for LAW feeds, with implications for potentially similar behavior for technetium. Iodine retention ranged from no retention in glass while processing feed without any organic carbon to over forty percent retention while processing feed with higher amounts of organic carbon; increased iodine retention under more reducing conditions was observed in previous tests [26-28]. Other than iodine, no clear trend of elemental retention in the glass product with respect to changes in the feed chemistry could be discerned. The amount of the four most volatile elements measured in glass and exhaust as a function of feed nitrate and carbon

contents are depicted in Figures 6.1 – 6.4. Note that selenium, sulfur, and rhenium retention in glass vary within ten to twenty percent ranges and do not vary systematically with feed nitrate content or the relative amounts of nitrate and carbon in the feed. The amounts measured in the exhaust show a similar lack of any trend; however, the data span a wider range due to the greater variability inherent in exhaust sampling.

A nitrogen and partial carbon mass balance around the melter is summarized in Table 6.2. The nitrogen oxide and carbon fluxes into the melter were defined by the waste nitrate, nitrite, and carbon concentrations and the average melter feed rate for each test. The feed rates are compared to emission rates, which are calculated from the average measured NO, NO<sub>2</sub>, and CO concentrations and the average exhaust flow rate at the point of measurement. In the first three tests in which no carbon was added to the feed, all the feed nitrogen oxides were emitted as NO and NO<sub>2</sub>. As carbon is added to the feed in the form of sugar, about seventy percent of the feed nitrogen oxides were emitted as nitrogen oxides, with the balance of the nitrogen reduced to diatomic nitrogen. This result approximates previous tests processing LAW wastes with similar sugar to nitrogen oxide ratios in the feed, which showed forty to sixty percent emitted as nitrogen oxides [1-13, 58]. In tests without nitrate added to the feed, the amounts of NO and NO<sub>2</sub> monitored in the exhaust far exceeds the small amount of nitrite in the feed indicating trace level contamination of nitrogen oxides in the feed. Approximately half of feed nitrate is emitted as nitrogen oxides while processing feed with nitrate and oxalate. The carbon to nitrate ratio in these tests is an order of magnitude higher than in tests with sugar as a carbon source but resulted in the same level of reduction of nitrates to diatomic nitrogen, further illustrating that oxalate is a much less effective reductant than sugar. Carbon monoxide emissions as a percent of feed carbon increased with increasing carbon content in the feed and decreased with the addition of nitrate to the feed.

## SECTION 7.0 SUMMARY AND CONCLUSIONS

A series of melter tests were conducted on the DM10 using a HLW C106/AY-102 composition to evaluate the effect of variable nitrate and oxalate waste concentrations on feed and glass processing, glass oxidation state, and volatile retention in the glass product. The tests employed melter feed spiked with toxic metals and radioactive surrogates and used four different nitrate and oxalate concentrations. In each of the eight tests, the bubbling rate was set at 1 lpm and the feed rate was adjusted to provide the desired complete cold cap and target plenum temperature of 550 – 650°C for each of the four feed types. 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 were made throughout the tests. In addition, particulate loading and composition as well as acid gas concentrations were determined, which permitted the calculation of 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. Glass samples were taken from the melt pool to measure the oxidation state of the glass melt and to detect secondary phases on the melt pool floor.

The melter tests resulted in the production of over 165 kg of glass from over half a metric ton of feed. Testing demonstrated that the HLW simulant could be processed with nitrate and oxalate concentrations far exceeding the current WTP Contract Maximum values [46]. However, increases in feed concentrations of nitrate and oxalate had significant impacts on the processing characteristics and processing rate. The principal results are summarized as follows:

- The feed rate and glass production rate decreased by over 40% with the addition of nitrates to the feed. The decrease in production rate was accompanied by an increase in melt pool resistance indicating that foaming was occurring in the melt pool even though no visual evidence of foaming was apparent on the melt pool surface. Addition of sugar to the high nitrate HLW feed partially mitigated the foaming and the resulting production rates were then only 15% below that achieved with feeds that do not contain nitrates.
- Processing feed containing 50 g oxalate per kg glass resulted in a glass product with greater than 10%  $\text{Fe}^{2+}$ /Total Fe, which effectively defines the limiting oxalate concentration for processing this waste composition. Although this oxalate concentration exceeds the WTP Contract Maximum, it is less than a quarter of the Action Limit [47].
- With nitrate additions to the feed, feed containing 50 g oxalate per kg glass was processed while maintaining a divalent iron concentration in the product glass of less than 10%.

- Processing feed containing 75 g oxalate and 30 g nitrate per kg glass resulted in a glass product with about 10%  $\text{Fe}^{2+}$ /Total Fe and small amounts of a secondary phase on the melt pool floor. The secondary phase was identified as nickel copper selenide; continued deposition of this material on the melter floor could compromise performance and limit melter life.
- The tests demonstrated that oxalate is a much less effective reductant than sugar. Therefore the algorithms used to determine the addition of sugar to high nitrate LAW feeds to prevent foaming would have to be modified for wastes that are high in oxalate.
- The data from the present tests and previous tests were analyzed to develop a correlation that could be used for redox control for HLW vitrification, which determines the required additions of sugar or nitrate to the melter feed in a similar manner to that employed for the LAW flow sheet.
- Solids and elemental carryover from the melter was largely unaffected by feed nitrate and oxalate concentrations. Conversely iodine was retained in the glass only under reducing conditions.

Together these results provide the basis for a preliminary strategy for redox control and management of variations in nitrate and organic carbon content of the HLW feed to the WTP vitrification systems. Excessive variations in these constituents can lead to overly oxidizing or overly reducing conditions in the melt pool, respectively. Overly oxidizing conditions can lead to foaming in the melt pool and significant decreases in the glass production rate. Overly reducing conditions in the melt pool can lead to deposition of reduced phases such as metals and metalloids (such as sulfides and selenides); these phases are typically much denser than the glass, and can be highly electrically conductive, low melting, corrosive, and very fluid. As a result, they have the potential to compromise melter operations and melter lifetime. The results of the present work suggest the following preliminary strategy to mitigate these risks:

- (1) In the absence of significant nitrate in the melter feed, the results from the present tests indicate that oxalate concentrations should be limited to a maximum of 50 g oxalate per kg of glass in order to prevent the melt pool redox state from exceeding about 10 %  $\text{Fe}^{2+}$ /Total Fe. More reducing conditions have the potential to cause the deposition of reduced phases such as the nickel copper selenide identified in the present work. In view of the limited test data presently available, it would seem reasonable to add a safety margin to this limit, decreasing it to perhaps 45 g oxalate per kg glass.
  - For melter feeds that would exceed this limit either: (i) the waste loading should be decreased to remain within this limit; or (ii) nitric acid should be added to the melter feed in the amount determined in (3) below to counterbalance the reducing effects of the oxalate.

- (2) In the absence of significant oxalate in the melter feed, the results from the present tests indicate that melt pool foaming will increase and glass production rates will decrease as the nitrate content in the feed increases. The effects are very significant (greater than 40% decrease in glass production rate) at 60 g nitrate per kg of glass, and probably also at 30 g nitrate per kg glass. Conversely, these effects appear to be minimal at about 8 g nitrate per kg glass; additional data would be required to better define the nitrate concentration at which these effects become significant. It should also be noted that these results were obtained on the DM10 system and these effects may be scale dependent.
  - At higher levels of nitrates, the deleterious effects of high nitrates can be mitigated by the addition of sugar in the amount determined in (3) below as a counterbalancing reductant, as is the case for WTP LAW feeds.
- (3) The ranges described in (1) and (2) above are intended to provide an operating window over which no mitigating action is required, which is the present de facto approach for WTP HLW feeds. As noted above, however, additional data would be useful to define that range more precisely. Beyond the ranges described in (1) and (2) above, at present, it is recommended that a correlation of the type described in Section 4.2.1 be used to calculate the required sugar or nitric acid additions to the feed in order to prevent overly reducing or oxidizing conditions in the melt pool. The sugar or nitrate addition to the melter feed should be such as to bring the value of  $D_r$ , as defined in Section 4.2.1, into the range of about zero to +0.2. The results from that approach will be close to what would be obtained from the algorithm that is used for LAW feeds, with the primary difference coming from the allowance for the fact that organic carbon from oxalate is observed to be significantly less effective as a reducing agent than organic carbon from sugar.

## 7.1 Recommendations for Future Work

The results of the testing presented herein have provided the basis for an initial strategy for redox control for HLW feeds that have high nitrate and/or organic carbon contents. Further work that is recommended in order to refine this strategy is outlined below.

- *Scale-Up Testing:* Since the effects of melt pool foaming can vary with melter scale, larger scale testing should be performed to confirm the results from the present work, which were obtained on the DM10 system. Such tests could be performed on the DM100 melter system with more limited testing on the DM1200 system.
- *Maximum Nitrate:* Testing should be performed to better define the maximum nitrate concentration beyond which sugar additions are required.

- *Oxalate as a Reductant:* Testing should be performed to better define the relationship between organic carbon from oxalate as compared to organic carbon from sugar. This is one of the factors that is required to determine the amounts of nitrate or sugar that needs to be added for redox control. Although oxalate is expected to be the principal source of organic carbon in the HLW feeds, similar testing should be performed for other organic species if they are expected to be present in significant concentrations.
- *Effects of Nitrite versus Nitrate:* Testing should be performed to determine the relative effects of nitrite versus nitrite, which are treated as equivalent in both the baseline LAW redox control algorithm and the present HLW approach. It is likely that nitrite is a less effective oxidizing agent.
- *Refinement of Redox Control Algorithm:* While the redox control algorithm described above provides a reasonable starting point, additional data would be useful to refine and extend this approach.
- *Other WTP HLW Feed Types:* The present testing was based on a single high iron HLW composition from the Hanford tanks. Subsequent work should extend these results to address the full range of HLW feeds expected to be processed at the WTP. Particular attention should be given to the effects of variation in the concentrations of key reducible species such as iron and manganese.

**SECTION 8.0**  
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**Table 2.1. Compositional Summary of Different Waste Streams and Blended Solids for the C-106/AY-102 HLW Simulant.**

Waste Component	C-106/AY-102 Solids	Recycle Stream	Separation Factor	Sr/TRU Product	Cs-Eluate	Tc-Eluate	Blended Solids
	FRP02	PWD01	-	-	CNP12	TEP12	HLP09b
	(lb/day)	(lb/day)	(fraction remained)	(lb/day)	(lb/day)	(lb/day)	(lb/day)
Ag	9.20E+01	5.49E-21	4.885 <sup>*</sup>	-	-	-	9.20E+01
Al	3.19E+03	2.17E+00	0.395	-	5.13E+00	7.54E-02	1.27E+03
As	9.77E+01	1.32E-01	1.825 <sup>*</sup>	-	-	-	9.78E+01
B	1.83E+01	2.88E+00	2.759 <sup>*</sup>	-	7.27E+00	-	2.84E+01
Ba	6.59E+01	2.69E-04	0.054	-	6.24E-03	2.10E-03	3.55E+00
Be	4.89E+00	0.00E+00	1.000	-	-	-	4.89E+00
Bi	1.71E+00	2.58E-04	5.303 <sup>*</sup>	-	-	-	1.71E+00
Ca	4.01E+02	9.03E-02	0.360	-	9.31E-01	2.22E-02	1.45E+02
Cd	1.07E+01	1.57E-04	0.028	-	1.19E-02	2.05E-03	3.10E-01
Ce	5.08E+01	5.90E+00	0.041	-	-	-	2.33E+00
Cl	3.83E+01	2.13E+00	0.064	-	5.94E+01	1.14E+01	7.34E+01
Co	2.05E+01	0.00E+00	1.000	-	-	5.59E-03	2.05E+01
Carbonate	4.73E+03	2.41E+00	0.185	-	-	-	8.74E+02
Cr	1.27E+02	2.01E-01	0.281	-	1.38E-01	5.45E-03	3.58E+01
Cs	7.84E-01	0.00E+00	0.186	-	6.33E-02	3.35E-07	2.09E-01
Cu	2.34E+01	6.86E-33	200.513 <sup>*</sup>	-	3.75E-01	3.89E-03	2.38E+01
F	1.30E+01	7.49E-01	0.037	-	-	-	5.07E-01
Fe	5.87E+03	1.49E+00	1.897 <sup>*</sup>	-	9.57E-02	5.63E-03	5.95E+03
Hg	2.56E+01	2.09E-05	4.438 <sup>*</sup>	-	-	-	2.56E+01
K	2.09E+01	9.11E-01	0.134	-	9.77E-01	2.03E-02	3.91E+00
La	1.39E+02	1.98E-02	2.753 <sup>*</sup>	-	-	2.00E-02	1.39E+02
Li	0.00E+00	7.57E-01	2.848	-	-	5.65E-03	2.16E+00
Mg	2.21E+02	4.89E-06	2.154	-	1.50E-01	4.17E-03	4.76E+02
Mn	1.26E+03	9.01E-02	1.000	4.49E+02	8.20E-03	7.73E-04	1.71E+03
Mo	3.94E+00	0.00E+00	1.000	-	-	2.07E-03	3.94E+00
Na	4.28E+03	3.65E+02	0.059	-	2.02E+01	9.14E-01	2.93E+02
Nd	8.71E+01	0.00E+00	1.000	-	-	-	8.71E+01
Ni	2.20E+02	1.10E-01	0.411	-	5.85E-01	6.68E-03	9.13E+01
Nitrite	4.47E+01	5.06E-01	0.050	-	-	-	2.28E+00
Nitrate	2.93E+01	8.67E+02	0.037	-	1.14E+02	-	1.47E+02
Hydroxide	8.33E+03	3.16E+01	0.114	-	-	-	9.56E+02
Hydroxide(Bound)	5.34E+03	0.00E+00	0.076	-	-	-	4.06E+02
Pb	2.56E+02	2.27E-02	0.353	-	0.00E+00	2.11E-02	9.04E+01
Pd	0.00E+00	2.15E-09	5.392	-	-	-	1.16E-08
Phosphate	1.15E+03	1.66E-02	0.074	-	-	-	8.53E+01
Pr	0.00E+00	0.00E+00	1.000	-	-	-	0.00E+00
Rb	0.00E+00	0.00E+00	1.000	-	-	-	0.00E+00
Rh	0.00E+00	0.00E+00	1.000	-	-	-	0.00E+00
Ru	0.00E+00	0.00E+00	1.000	-	-	-	0.00E+00
Sb	5.91E+01	0.00E+00	2.434	-	-	-	1.44E+02
Se	9.77E+01	0.00E+00	1.825	-	-	-	1.78E+02
Si	6.36E+02	6.02E+00	4.398 <sup>*</sup>	-	2.13E+00	5.69E-02	6.44E+02
Sulfate	3.48E+01	5.45E-01	0.034	-	-	-	1.20E+00
Sr	2.52E+01	0.00E+00	0.985	4.99E+02	-	1.05E-03	5.24E+02
Ta	0.00E+00	0.00E+00	-	-	-	-	0.00E+00
Te	5.83E+00	0.00E+00	-	-	-	-	0.00E+00
Th	0.00E+00	0.00E+00	-	-	-	-	0.00E+00
Ti	1.07E+01	1.53E-03	5.306	-	-	5.02E-03	5.69E+01
Tl	1.97E+02	0.00E+00	-	-	-	-	0.00E+00
TOC	2.96E+02	0.00E+00	0.017	-	-	-	4.92E+00
U	2.18E+02	0.00E+00	-	-	2.01E-01	-	2.01E-01
V	4.89E+01	0.00E+00	-	-	-	9.14E-03	9.14E-03
Y	0.00E+00	0.00E+00	-	-	-	-	0.00E+00
Zn	1.30E+01	4.36E-01	2.843	-	4.66E-02	2.87E-03	3.81E+01
Zr	6.14E+01	3.44E-01	4.576 <sup>*</sup>	-	-	6.94E-03	1.30E+02
TOTAL	3.79E+04	1.31E+03 <sup>**</sup>	-	9.48E+02	2.12E+02	1.26E+01	1.49E+04

\* Separation Factors not Used in Calculation (see text); - Empty Data Field; \*\* Includes negligible components that are omitted.

**Table 2.2. Compositional Summary (Oxide Basis) of the C-106/AY-102 Base Simulant, Spikes, and the Target HLW Simulant.**

Wt%	C-106/AY-102 HLW Simulant	Total Spike Concentration (as wt% of glass)	Target HLW Simulant
Al <sub>2</sub> O <sub>3</sub>	12.771%	—	12.535%
As <sub>2</sub> O <sub>3</sub>	0.689%	—	0.676%
B <sub>2</sub> O <sub>3</sub>	0.488%	—	0.479%
CaO	1.086%	—	1.065%
CdO	—	0.250%	0.901%
Cl	0.392%	0.125%	0.450%
Cr <sub>2</sub> O <sub>3</sub>	0.279%	0.175%	0.630%
Cs <sub>2</sub> O	0.180%	—	—
CuO	0.159%	0.052%	0.187%
F	—	0.042%	0.151%
Fe <sub>2</sub> O <sub>3</sub>	45.351%	—	44.514%
I	0.360%	0.100%	0.360%
La <sub>2</sub> O <sub>3</sub>	0.872%	—	0.856%
Li <sub>2</sub> O	0.025%	—	0.024%
MgO	4.208%	—	4.131%
MnO	14.405%	—	14.139%
Na <sub>2</sub> O	2.107%	—	2.068%
Nd <sub>2</sub> O <sub>3</sub>	0.542%	—	0.532%
NiO	0.619%	—	0.608%
P <sub>2</sub> O <sub>5</sub>	0.340%	—	0.334%
PbO	0.520%	0.300%	1.081%
Re <sub>2</sub> O <sub>7</sub>	—	0.050%	0.180%
SO <sub>3</sub>	—	0.150%	0.540%
Sb <sub>2</sub> O <sub>3</sub>	0.918%	—	0.901%
SeO <sub>2</sub>	1.336%	0.147%	0.530%
SiO <sub>2</sub>	7.353%	—	7.218%
SrO	3.306%	—	3.245%
TiO <sub>2</sub>	0.506%	—	0.497%
ZnO	0.253%	—	0.248%
ZrO <sub>2</sub>	0.935%	—	0.917%
TOTAL	100.00%	1.391%	100.00%
<i>Volatiles</i>	(g/100 g waste oxide)	(g/100 g glass)	(g/100 g glass)
Carbonate	4.650	—	12.908
Nitrite	0.012	0.582	0.582
Nitrate	0.784	3.000	3.000
TOC	0.026	2.491	2.491

- Empty data field

**Table 2.3. Compositional Summary (oxide basis) of the C-106/AY-102 HLW Simulant, Glass Former Additives, Target Test Glass, and the Reference Glass (HLW98-96).**

Wt%	C-106/AY-102 HLW Simulant	Glass Formers (as wt% of glass)	Target HLW Glass	MACT Melter Test Target Glass [30]	HLW98-96
Ag <sub>2</sub> O	—	—	—	—	0.15%
Al <sub>2</sub> O <sub>3</sub>	12.535%	1.750%	5.229%	5.283%	5.29%
As <sub>2</sub> O <sub>3</sub>	0.676%	—	0.188%	0.191%	0.19%
B <sub>2</sub> O <sub>3</sub>	0.479%	9.250%	9.383%	9.344%	9.39%
CaO	1.065%	—	0.296%	0.301%	0.30%
CdO	0.901%	—	0.250%	0.250%	—
Cl	0.450%	—	0.125%	0.125%	0.11%
Cr <sub>2</sub> O <sub>3</sub>	0.630%	—	0.175%	0.175%	0.08%
CuO	0.187%	—	0.052%	0.052%	0.04%
F	0.151%	—	0.042%	0.042%	—
Fe <sub>2</sub> O <sub>3</sub>	44.514%	—	12.356%	12.574%	12.56%
I	0.360%	—	0.100%	—	—
La <sub>2</sub> O <sub>3</sub>	0.856%	—	0.238%	0.242%	0.24%
Li <sub>2</sub> O	0.024%	2.993%	3.000%	2.993%	3.01%
MgO	4.131%	—	1.147%	1.167%	1.17%
MnO	14.139%	—	3.925%	3.994%	3.99%
Na <sub>2</sub> O	2.068%	11.250%	11.824%	11.784%	11.84%
Nd <sub>2</sub> O <sub>3</sub>	0.532%	—	0.148%	0.150%	0.15%
NiO	0.608%	—	0.169%	0.172%	0.17%
P <sub>2</sub> O <sub>5</sub>	0.334%	—	0.093%	0.092%	0.09%
PbO	1.081%	—	0.300%	0.300%	0.14%
Re <sub>2</sub> O <sub>7</sub>	0.180%	—	0.050%	—	—
SO <sub>3</sub>	0.540%	—	0.150%	0.150%	—
Sb <sub>2</sub> O <sub>3</sub>	0.901%	—	0.250%	0.255%	0.26%
SeO <sub>2</sub>	0.530%	—	0.147%	0.147%	0.37%
SiO <sub>2</sub>	7.218%	45.000%	47.003%	46.838%	47.07%
SrO	3.245%	—	0.901%	0.917%	0.92%
TiO <sub>2</sub>	0.497%	—	0.138%	0.140%	0.14%
ZnO	0.248%	2.000%	2.069%	2.061%	2.07%
ZrO <sub>2</sub>	0.917%	—	0.255%	0.259%	0.26%
TOTAL	100.00%	72.243%	100.00%	100.00%	100.0%
Volatiles (g/100 g glass)	—	—	—	—	—
Carbonate	12.908	—	—	—	—
Nitrite	0.582	—	—	—	—
Nitrate	3.000	—	—	—	—
TOC	2.491	—	—	—	—

— Empty data field

**Table 2.4. Composition of Melter Feed to Produce 1 Metric Ton of Target Glass from C-106/AY-102 HLW Simulant (20 wt% suspended solids).**

Target Simulant		Glass-Forming Additives	
Starting Material	Target Weight (kg)*	Starting Material	Target Weight (kg)*
Al(OH) <sub>3</sub>	56.04	Kyanite (Al <sub>2</sub> SiO <sub>5</sub> )	30.65
As <sub>2</sub> O <sub>3</sub>	1.63	—	—
H <sub>3</sub> BO <sub>3</sub>	2.38	Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> .10H <sub>2</sub> O	255.91
CaCO <sub>3</sub>	2.34	—	—
CdO	2.53	—	—
NaCl	2.08	—	—
Cr <sub>2</sub> O <sub>3</sub>	1.77	—	—
CuO	0.53	—	—
CaF <sub>2</sub>	0.89	—	—
Fe(OH) <sub>3</sub> (13% Slurry)	1261.16	—	—
NaI	1.19	—	—
La(OH) <sub>3</sub> .3H <sub>2</sub> O	3.59	—	—
Li <sub>2</sub> CO <sub>3</sub>	0.17	Li <sub>2</sub> CO <sub>3</sub>	75.92
Mg(OH) <sub>2</sub>	16.93	—	—
MnO <sub>2</sub>	48.58	—	—
Na <sub>2</sub> CO <sub>3</sub>	0.83	Na <sub>2</sub> CO <sub>3</sub>	123.20
Nd <sub>2</sub> O <sub>3</sub>	1.49	—	—
Ni(OH) <sub>2</sub>	2.17	—	—
FePO <sub>4</sub> .xH <sub>2</sub> O	2.46	—	—
PbO	3.03	—	—
HReO <sub>4</sub> (70% Soultion)	1.56	—	—
CaSO <sub>4</sub> .2H <sub>2</sub> O	2.58	—	—
Sb <sub>2</sub> O <sub>3</sub>	2.53	—	—
SeO <sub>2</sub>	1.48	—	—
SiO <sub>2</sub>	20.24	SiO <sub>2</sub>	441.54
SrCO <sub>3</sub>	13.16	—	—
TiO <sub>2</sub>	1.39	—	—
ZnO	0.70	ZnO	20.20
Zr(OH) <sub>4</sub> .xH <sub>2</sub> O	5.09	—	—
NaNO <sub>2</sub>	8.95	—	—
HNO <sub>3</sub> (70% Soultion) #	43.56	—	—
H <sub>2</sub> C <sub>2</sub> O <sub>4</sub> .2H <sub>2</sub> O #	131.51	—	—
H <sub>2</sub> O	609.20	—	—
TOTAL	2253.76	TOTAL	947.42
—	—	FEED TOTAL	3201.18

\*Target weights adjusted for assay information of starting materials; - Empty data field, # nitric and oxalic acid were added at VSL to achieve the various target concentrations for the tests.

**Table 2.5. Nitrate, Nitrite, and Organic Carbon Content in Feed Used in Previous Melter Tests with HLW C-106/AY-102 Waste Compared to WTP Contract Limits.**

	Nitrate (g/kg glass)	Nitrite (g/kg glass)	Carbon from Oxalate (g/kg glass)	Carbon from Sugar (g/kg glass)	C:NOx (mole ratio)	Fe <sup>2+</sup> / Total Fe Measured in Glass
VSL-03R3800-1 [33] DM1200	6.03	0.09	0.20	0	0.17	Not Measured
VSL-05R5800-1 [36] DM1200	6.03	0.09	0.20	0	0.17	< 0.03
	7.84	0.12	0.26	0	0.17	< 0.03
VSL-04R4800-1 [28] DM1200	6.03	0.09	0.20	0	0.17	0.04
	6.03	0.09	0.20	7.63	6.57	0.07
	6.03	0.09	0.20	11.44	9.77	0.12
	6.03	0.09	0.20	13.35	11.37	0.17
	6.03	0.09	0.20	15.26	12.97	0.25
	6.03	0.09	0.20	19.07	16.16	0.24
	30	0.09	0.20	0	0.03	0.03
	30	0.09	0.20	7.63	1.34	0.04
	30	0.09	0.20	11.44	2.00	0.06
	30	0.09	0.20	15.26	2.65	0.10
	30	0.09	0.20	19.07	3.31	0.31
VSL-05R5830-1 [30] DM1200	30	5.82	24.91	0	3.40	<0.03
WTP Contract Maximum [46]	99.9 (corresponds to 36 g nitrate and nitrite per 100 g waste oxides)		30.5 (corresponds to 11 g TOC per 100 g waste oxides)		—	—
24590-WTP-RPT-MGT- 11-014 Action Limit [47]	—		225.7 (corresponds to 10 wt% TOC in waste)		—	—

- Empty data field

**Table 2.6. Analysis of As-Received Feed.**

Sample	10V-F-94C		
% Water	56.77		
pH	10.58		
Density (g/ml)	1.48		
Glass Yield	(kg/kg)	0.399	
	(g/l)	591	
Oxide	Composition		
	Target	XRF	% Dev.
Al <sub>2</sub> O <sub>3</sub>	5.23	5.95	13.77
As <sub>2</sub> O <sub>3</sub>	0.19	0.16	NC
B <sub>2</sub> O <sub>3</sub>	9.38	9.11*	-2.88
CaO	0.30	0.37	NC
CdO	0.25	0.29	NC
Cl	0.12	0.06	NC
Cr <sub>2</sub> O <sub>3</sub>	0.17	0.18	NC
CuO	0.05	0.06	NC
F	0.04	NA	NC
Fe <sub>2</sub> O <sub>3</sub>	12.36	12.31	-0.40
I	0.10	<0.003	NC
La <sub>2</sub> O <sub>3</sub>	0.24	0.30	NC
Li <sub>2</sub> O	3.00	2.78*	-7.33
MgO	1.15	1.22	6.09
MnO	3.92	3.61	-7.91
Na <sub>2</sub> O	11.82	11.89	0.59
Nd <sub>2</sub> O <sub>3</sub>	0.15	0.14	NC
NiO	0.17	0.20	NC
P <sub>2</sub> O <sub>5</sub>	0.09	0.11	NC
PbO	0.30	0.29	NC
Re <sub>2</sub> O <sub>7</sub>	0.05	0.021	NC
Sb <sub>2</sub> O <sub>3</sub>	0.25	0.30	NC
SeO <sub>2</sub>	0.15	0.03	NC
SiO <sub>2</sub>	47.00	47.11	0.23
SO <sub>3</sub>	0.15	0.22	NC
SrO	0.90	0.83	NC
TiO <sub>2</sub>	0.14	0.22	NC
ZnO	2.07	1.87	-9.66
ZrO <sub>2</sub>	0.25	0.35	NC
Sum	100.00	100.00	NC

\* - DCP-AES analyzed values.

NA - Not Analyzed.

NC - Not Calculated.

**Table 2.7. Melter Feed Recipes for DM10 Tests.**

Test	Target		As Received Feed	Water	Concentrated Nitric Acid	Oxalic Acid Dihydrate	Sugar
	Nitrate (g/ kg glass)	Oxalate (g C/ kg glass)					
1 N	60	0	66 kg	13.5 kg	1558 ml	0	0
2 N	90	0	61 kg	11.5 kg	2160 ml	0	0
3 N	120	0	61 kg	10.5 kg	2880 ml	0	0
9 N	120	0	61 kg	9.5 kg	2880 ml	0	1038 g
1 OX	0	25	61 kg	11.5 kg	0	3092 g	0
2 OX	0	50	61 kg	8.5 kg	0	6184 g	0
3 OX	30	50	61 kg	7.5 kg	720 ml	6184 g	0
5 OX	30	75	61 kg	4.6 kg	720 ml	9276 g	0

**Table 3.1. Summary of DM10 Melter Tests.**

Test		1 N	2 N	3 N
Feed	Feeding Interval	4/19/12 20:30 – 4/20/12 23:45	4/23/12 10:00 – 4/24/12 10:00	4/24/12 10:40 – 4/25/12 10:40
	Total	27.25 hr	24 hr	24 hr
Feed	Nitrate	60 g /kg glass	90 g /kg glass	120 g /kg glass
	Sugar	0	0	0
	Oxalate	0	0	0
	C:NOx (mole ratio)	Undefined	Undefined	Undefined
	Processed (kg)	66.785	47.020	47.770
	Processing rate (kg/hr)	2.45	1.96	1.98
Glass	Produced from feed (kg)	21.04	14.67	14.90
	Discharged (kg)	19.44	14.26	16.08
	Test Average Production Rate (kg/m <sup>2</sup> /day)*	874	699	710
	Fe <sup>2+</sup> /Total Fe (%)	< 1.0	< 1.0	< 1.0
Test Average Glass Temperature (°C)	2" from floor	1154	1155	1155
	4" from floor	1146	1151	1147
Test Average Plenum Temperature (°C)	Exposed	434	488	473
	Thermowell	496	535	524
Test Average Electrode Temperature (°C)		992	972	982
Test Average Discharge Chamber Temperature (°C)		1056	1059	1058
Test Average Film Cooler Exhaust Outlet Temperature (°C)		276	274	279
Test Average Melter Pressure (inches water)		-1.0	-0.9	-0.9
Test Average Melt Pool Bubbling (lpm)		1.0	1.0	1.0
Test Average Electrical Properties	Voltage (volts)	29.7	29.4	30.9
	Current (amps)	154.0	141.7	152.9
	Power (kW)	4.6	4.2	4.7
	Glass Pool Resistance (ohms)	0.193	0.208	0.203
	kWhr/kg glass	6.0	6.8	7.6

\* - Calculated from total feed processed

NA – Not Applicable

**Table 3.1. Summary of DM10 Melter Tests (Continued).**

Test		9 N	1 OX	2 OX
Feed	Feeding Interval	4/25/12 11:15 – 4/26/12 11:00	4/26/12 11:50 – 4/27/12 11:50	4/30/12 10:00 – 5/1/12 10:00
	Total	23.75 hr	24 hr	24 hr
Feed	Nitrate	120 g /kg glass	0	0
	Sugar	18.56 g Carbon /kg glass	0	0
	Oxalate	0	25 g Carbon /kg glass	50 g Carbon /kg glass
	C:NOx (mole ratio)	0.80	1065	2130
	Processed (kg)	66.640	86.413	81.095
	Processing rate (kg/hr)	2.81	3.60	3.38
Glass	Produced from feed (kg)	20.79	26.96	25.30
	Discharged (kg)	20.90	26.52	25.29
	Test Average Production Rate (kg/m <sup>2</sup> /day)*	1001	1284	1205
	Fe <sup>2+</sup> /Total Fe (%)	< 1.0	1.6	10.0
Test Average Glass Temperature (°C)	2" from floor	1154	1152	1149
	4" from floor	1142	1142	1140
Test Average Plenum Temperature (°C)	Exposed	502	478	503
	Thermowell	548	525	546
Test Average Electrode Temperature (°C)		992	985	970
Test Average Discharge Chamber Temperature (°C)		1051	1052	1058
Test Average Film Cooler Exhaust Outlet Temperature (°C)		280	284	254
Test Average Melter Pressure (inches water)		-0.9	-0.8	-1.1
Test Average Melt Pool Bubbling (lpm)		1.0	1.0	1.0
Test Average Electrical Properties	Voltage (volts)	30.0	30.5	29.9
	Current (amps)	174.3	191.5	179.8
	Power (kW)	5.3	5.9	5.4
	Glass Pool Resistance (ohms)	0.173	0.161	0.167
	kWhr/kg glass	6.0	5.3	5.1

\* - Calculated from total feed processed

NA – Not Applicable

**Table 3.1. Summary of DM10 Melter Tests (Continued).**

Test		3 OX	5 OX
Feed	Feeding Interval	5/1/12 11:35 – 5/2/12 11:35	5/2/12 10:40 – 5/3/12 10:40
	Total	24 hr	24 hr
Feed	Nitrate	30 g /kg glass	30 g /kg glass
	Sugar	0	0
	Oxalate	50 g Carbon /kg glass	75 g Carbon /kg glass
	C:NOx (mole ratio)	8.58	12.86
	Processed (kg)	63.805	72.290
	Processing rate (kg/hr)	2.66	3.01
Glass	Produced from feed (kg)	17.21	26.97
	Discharged (kg)	18.81	25.04
	Test Average Production Rate (kg/m <sup>2</sup> /day)*	948	1074
	Fe <sup>2+</sup> /Total Fe (%)	7.1	10.7
Test Average Glass Temperature (°C)	2" from floor	1152	1151
	4" from floor	1148	1143
Test Average Plenum Temperature (°C)	Exposed	477	522
	Thermowell	513	560
Test Average Electrode Temperature (°C)		961	991
Test Average Discharge Chamber Temperature (°C)		1052	1060
Test Average Film Cooler Exhaust Outlet Temperature (°C)		248	252
Test Average Melter Pressure (inches water)		-1.1	-0.9
Test Average Melt Pool Bubbling (lpm)		1.0	1.0
Test Average Electrical Properties	Voltage (volts)	29.3	30.0
	Current (amps)	160.8	180.8
	Power (kW)	4.7	5.4
	Glass Pool Resistance (ohms)	0.182	0.166
	kWhr/kg glass	5.7	5.8

\* - Calculated from total feed processed

NA - Not Applicable

**Table 4.1. Listing of Glasses Discharged During DM10 Tests.**

Test	Date	Name	Mass (kg)	Cumulative Mass (kg)
1 N	4/19/12	10W-G-130A	3.36	3.36
		10W-G-131A		
		10W-G-131B	3.98	7.34
		10W-G-131C		
		10W-G-132A	2.96	10.30
	4/20/12	10W-G-132B		
		10W-G-132C		
		10W-G-137A	3.90	14.20
		10W-G-137B		
		10W-G-137C	2.80	17.00
2 N	4/23/12	10W-G-140A		
		10W-G-140B		
		10W-G-141A	2.44	19.44
		10W-G-141B		
		10W-G-148A	3.22	22.66
		10X-G-12A		
	4/24/12	10X-G-12B	2.72	25.38
		10X-G-12C		
		10X-G-13A	2.48	27.86
		10X-G-13B		
		10X-G-16A	2.74	30.60
3 N	4/25/12	10X-G-18A		
		10X-G-18B	3.10	33.70
		10X-G-19A		
		10X-G-23A	4.68	38.38
		10X-G-23B		
	4/26/12	10X-G-23C	3.34	41.72
		10X-G-25A		
		10X-G-25B	4.28	46.00
		10X-G-28A		
		10X-G-30A	3.78	49.78
9 N		10X-G-30B		
		10X-G-30C	1.84	51.62
4/26/12	10X-G-36A	3.48	55.10	
	10X-G-36B			
	10X-G-37A	4.52	59.62	
	10X-G-37B			
	10X-G-41A	4.44	64.06	
	10X-G-42A			
	10X-G-42B	4.24	68.30	
	10X-G-42C			

**Table 4.1. Listing of Glasses Discharged During DM10 Tests (Continued).**

Test	Date	Name	Mass (kg)	Cumulative Mass (kg)
9 N	4/26/12	10X-G-43A	2.38	70.68
4/26/12	4/26/12	10X-G-43B	3.22	73.90
		10X-G-43C	4.38	78.28
		10X-G-51A		
		10X-G-53A		
		10X-G-53B	4.10	82.38
	4/27/12	10X-G-57A		
		10X-G-57B		
		10X-G-57C		
		10X-G-57D	3.48	85.86
		10X-G-58A		
1 OX	4/27/12	10X-G-58B		
		10X-G-58C	3.74	89.60
		10X-G-58D		
		10X-G-58E		
		10X-G-60A		
	4/30/12	10X-G-60B	3.56	93.16
		10X-G-60C		
		10X-G-60D		
		10X-G-60E	2.52	95.68
		10X-G-60F		
2 OX	4/30/12	10X-G-65A	1.52	97.20
		10X-G-77A	3.44	100.64
		10X-G-77B	3.16	103.80
		10X-G-78A	2.54	106.34
		10X-G-78B		
		10X-G-78C	3.52	109.86
	5/1/12	10X-G-78D		
		10X-G-81A		
		10X-G-83A	4.04	113.90
		10X-G-83B		
3 OX	5/1/12	10X-G-83C		
		10X-G-83D	3.32	117.22
		10X-G-83E		
	5/1/12	10X-G-84A	3.68	120.90
		10X-G-84B		
	5/1/12	10X-G-89A	3.18	124.08
		10X-G-89B		

**Table 4.1. Listing of Glasses Discharged During DM10 Tests (Continued).**

Test	Date	Name	Mass (kg)	Cumulative Mass (kg)
3 OX	5/1/12	10X-G-89C	2.74	126.82
		10X-G-89D		
		10X-G-91A		
		10X-G-91B	4.14	130.96
		10X-G-91C		
		10X-G-91D		
	5/2/12	10X-G-94A	3.98	134.94
		10X-G-94B		
		10X-G-95A	3.04	137.98
		10X-G-95B		
		10X-G-95C	3.32	141.30
5 OX	5/3/12	10X-G-95D		
		10X-G-97A	4.30	145.60
		10X-G-97B		
		10X-G-97C	4.12	149.72
		10X-G-100A		
		10X-G-101A	3.50	153.22
		10X-G-101B		
		10X-G-101C	3.34	156.56
		10X-G-106A		
		10X-G-107A	4.80	161.36
		10X-G-107B		
		10X-G-107C		
		10X-G-107D	4.98	166.34
		10X-G-108A		

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

Test		1N						2N			
Mass (kg)		3.36	7.34	10.30	14.20	17.00	19.44	22.66	25.38	27.86	30.60
Constituent	Target	10W-G-131A	10W-G-132A	10W-G-132C	10W-G-137B	10W-G-140B	10W-G-141B	10X-G-12A	10X-G-12C	10X-G-13B	10X-G-18A
Al <sub>2</sub> O <sub>3</sub>		5.23	6.03	6.03	6.01	6.09	6.06	5.90	7.21	6.82	6.43
As <sub>2</sub> O <sub>3</sub>		0.19	0.03	0.08	0.10	0.11	0.13	0.15	0.14	0.16	0.14
B <sub>2</sub> O <sub>3</sub> *		9.38	13.11\$	11.89	11.24	10.64	10.34	10.13	9.92	9.79	9.70
BaO	§	0.12	0.05	0.06	0.04	0.03	0.03	0.02	0.02	<0.01	<0.01
Bi <sub>2</sub> O <sub>3</sub>	§	0.01	<0.01	<0.01	0.01	<0.01	<0.01	0.06	0.04	0.03	0.03
CaO	0.30	0.56	0.49	0.43	0.42	0.39	0.42	0.69	0.57	0.53	0.48
CdO	0.25	0.07	0.16	0.20	0.25	0.25	0.27	0.25	0.24	0.28	0.26
Ce <sub>2</sub> O <sub>3</sub>	§	0.06	0.04	0.02	<0.01	<0.01	<0.01	0.01	<0.01	<0.01	<0.01
Cl	0.12	0.02	0.05	0.05	0.06	0.05	0.05	0.02	0.03	0.05	0.05
Cr <sub>2</sub> O <sub>3</sub>	0.17	0.24	0.23	0.24	0.22	0.20	0.21	0.21	0.19	0.21	0.18
CuO	0.05	0.02	0.04	0.04	0.04	0.05	0.06	0.04	0.05	0.05	0.05
F	0.04	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Fe <sub>2</sub> O <sub>3</sub>	12.36	14.68	14.05	13.59	12.87	12.61	12.92	12.17	12.31	12.38	12.36
I	0.10	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
K <sub>2</sub> O	§	0.29	0.09	0.23	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
La <sub>2</sub> O <sub>3</sub>	0.24	0.12	0.18	0.20	0.28	0.24	0.23	0.23	0.20	0.34	0.26
Li <sub>2</sub> O*	3.00	1.84\$	2.22	2.42	2.61	2.70	2.77	2.83	2.87	2.90	2.92
MgO	1.15	0.44	0.73	0.90	1.02	1.08	1.16	1.01	1.04	1.16	1.19
MnO	3.92	2.74	3.06	3.14	3.21	3.23	3.45	3.06	3.11	3.19	3.18
Na <sub>2</sub> O	11.82	13.11	12.34	11.89	12.27	12.39	11.81	12.36	12.43	12.03	12.12
Nd <sub>2</sub> O <sub>3</sub>	0.15	<0.01	0.06	0.10	0.11	0.14	0.14	0.11	0.14	0.12	0.12
NiO	0.17	0.42	0.38	0.35	0.30	0.27	0.27	0.28	0.26	0.26	0.23
P <sub>2</sub> O <sub>5</sub>	0.09	0.50	0.36	0.28	0.20	0.19	0.17	0.23	0.20	0.18	0.16
PbO	0.30	0.45	0.40	0.37	0.32	0.29	0.31	0.31	0.29	0.29	0.29
Re <sub>2</sub> O <sub>7</sub>	0.05	<0.005	0.010	0.019	0.017	0.016	0.018	<0.005	0.011	0.012	0.012
Sb <sub>2</sub> O <sub>3</sub>	0.25	0.03	0.16	0.23	0.26	0.24	0.25	0.15	0.23	0.33	0.17
SeO <sub>2</sub>	0.15	0.01	0.02	0.02	0.02	0.02	0.02	0.01	0.01	0.01	0.02
SiO <sub>2</sub>	47.00	43.09	44.29	45.08	45.70	45.99	46.05	45.74	45.86	46.17	46.51
SnO <sub>2</sub>	§	0.07	0.04	0.03	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
SO <sub>3</sub>	0.15	0.32	0.24	0.19	0.19	0.18	0.21	0.14	0.18	0.21	0.17
SrO	0.90	0.28	0.49	0.59	0.64	0.70	0.74	0.66	0.70	0.73	0.74
TiO <sub>2</sub>	0.14	0.11	0.16	0.20	0.20	0.22	0.22	0.21	0.22	0.22	0.22
ZnO	2.07	0.39	0.98	1.23	1.44	1.55	1.64	1.47	1.60	1.65	1.72
ZrO <sub>2</sub>	0.25	0.85	0.67	0.55	0.46	0.41	0.42	0.43	0.40	0.39	0.37
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 DCP-AES analyzed results on the first discharge glass

NA - Not analyzed

§ - Not a target constituent

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

Test		2N	3N			9N				
Mass (kg)		33.70	38.38	41.72	46.00	49.78	51.62	55.10	59.62	64.06
Constituent	Target	10X-G-19A	10X-G-23B	10X-G-25A	10X-G-28A	10X-G-30B	10X-G-30C	10X-G-36B	10X-G-37B	10X-G-42A
Al <sub>2</sub> O <sub>3</sub>	5.23	6.26	6.18	6.21	6.14	6.11	6.04	5.81	5.98	5.91
As <sub>2</sub> O <sub>3</sub>	0.19	0.15	0.16	0.16	0.16	0.16	0.15	0.15	0.16	0.16
B <sub>2</sub> O <sub>3</sub> *	9.38	9.56	9.50	9.46	9.44	9.42	9.41	9.40	9.40	9.39
BaO	§	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Bi <sub>2</sub> O <sub>3</sub>	§	0.02	0.01	0.07	0.09	0.06	0.00	0.01	<0.01	<0.01
CaO	0.30	0.47	0.43	0.38	0.38	0.38	0.37	0.37	0.36	0.36
CdO	0.25	0.31	0.28	0.27	0.28	0.29	0.28	0.28	0.29	0.30
Ce <sub>2</sub> O <sub>3</sub>	§	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Cl	0.12	0.05	0.05	0.04	0.05	0.04	0.04	0.04	0.04	0.05
Cr <sub>2</sub> O <sub>3</sub>	0.17	0.20	0.18	0.21	0.22	0.20	0.20	0.21	0.22	0.23
CuO	0.05	0.06	0.05	0.05	0.05	0.07	0.06	0.06	0.05	0.07
F	0.04	NA								
Fe <sub>2</sub> O <sub>3</sub>	12.36	12.10	12.22	12.21	12.06	12.12	12.30	12.23	12.20	12.30
I	0.10	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
K <sub>2</sub> O	§	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
La <sub>2</sub> O <sub>3</sub>	0.24	0.29	0.31	0.28	0.28	0.29	0.28	0.27	0.28	0.30
Li <sub>2</sub> O*	3.00	2.94	2.96	2.97	2.98	2.99	2.99	2.99	3.00	3.00
MgO	1.15	1.21	1.19	1.18	1.16	1.21	1.21	1.29	1.26	1.19
MnO	3.92	3.28	3.30	3.33	3.35	3.38	3.43	3.54	3.53	3.69
Na <sub>2</sub> O	11.82	11.86	12.20	12.20	12.11	11.99	12.10	12.10	12.43	12.03
Nd <sub>2</sub> O <sub>3</sub>	0.15	0.12	0.11	0.15	0.14	0.13	0.13	0.14	0.13	0.13
NiO	0.17	0.21	0.22	0.23	0.23	0.23	0.23	0.24	0.23	0.24
P <sub>2</sub> O <sub>5</sub>	0.09	0.14	0.14	0.13	0.11	0.11	0.11	0.11	0.11	0.12
PbO	0.30	0.28	0.28	0.27	0.27	0.28	0.28	0.29	0.28	0.28
Re <sub>2</sub> O <sub>7</sub>	0.05	0.016	0.011	0.016	0.009	0.018	0.011	0.009	0.011	0.007
Sb <sub>2</sub> O <sub>3</sub>	0.25	0.18	0.30	0.31	0.31	0.17	0.30	0.27	0.30	0.33
SeO <sub>2</sub>	0.15	0.02	0.02	0.01	0.01	0.01	0.01	0.01	0.01	0.01
SiO <sub>2</sub>	47.00	46.99	46.62	46.57	46.89	47.04	46.68	46.76	46.44	46.48
SnO <sub>2</sub>	§	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
SO <sub>3</sub>	0.15	0.20	0.19	0.17	0.17	0.15	0.16	0.16	0.14	0.16
SrO	0.90	0.75	0.76	0.77	0.77	0.79	0.80	0.81	0.78	0.82
TiO <sub>2</sub>	0.14	0.24	0.23	0.23	0.23	0.24	0.24	0.25	0.23	0.23
ZnO	2.07	1.71	1.75	1.75	1.75	1.77	1.84	1.83	1.80	1.86
ZrO <sub>2</sub>	0.25	0.36	0.35	0.34	0.34	0.34	0.34	0.34	0.33	0.34
Sum	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 DCP-AES analyzed results on the first discharge glass

NA - Not analyzed

§ - Not a target constituent

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

Test		9N		1 OX							
Mass (kg)		68.30	70.68	73.90	78.28	82.38	85.86	89.60	93.16	95.98	
Constituent	Target	10X-G-42C	10X-G-43A	10X-G-43B	10X-G-51A	10X-G-57B	10X-G-58A	10X-G-58E	10X-G-60C	10X-G-60F	
Al <sub>2</sub> O <sub>3</sub>	5.23	5.99	5.98	5.95	6.06	5.94	6.00	5.95	5.97	5.95	
As <sub>2</sub> O <sub>3</sub>	0.19	0.15	0.16	0.14	0.14	0.15	0.15	0.15	0.15	0.15	0.16
B <sub>2</sub> O <sub>3</sub> *	9.38	9.39	9.39	9.39	9.39	9.38	9.38	9.38	9.38	9.38	9.38
BaO	§	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Bi <sub>2</sub> O <sub>3</sub>	§	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
CaO	0.30	0.36	0.33	0.36	0.34	0.35	0.36	0.36	0.36	0.35	
CdO	0.25	0.30	0.28	0.28	0.27	0.25	0.26	0.24	0.27	0.27	
Ce <sub>2</sub> O <sub>3</sub>	§	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Cl	0.12	0.05	0.05	0.05	0.05	0.07	0.06	0.07	0.06	0.06	0.06
Cr <sub>2</sub> O <sub>3</sub>	0.17	0.22	0.23	0.23	0.22	0.20	0.20	0.19	0.18	0.20	
CuO	0.05	0.06	0.06	0.06	0.07	0.05	0.06	0.06	0.06	0.05	
F	0.04	NA									
Fe <sub>2</sub> O <sub>3</sub>	12.36	12.07	12.32	12.03	12.02	12.09	12.08	11.99	12.08	11.89	
I	0.10	<0.01	<0.01	<0.01	0.04	0.03	0.03	0.03	0.04	0.03	
K <sub>2</sub> O	§	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
La <sub>2</sub> O <sub>3</sub>	0.24	0.29	0.33	0.29	0.31	0.25	0.27	0.25	0.33	0.29	
Li <sub>2</sub> O*	3.00	3.00	3.00	3.00	3.00	3.00	3.00	3.00	3.00	3.00	
MgO	1.15	1.25	1.26	1.27	1.19	1.31	1.26	1.31	1.19	1.27	
MnO	3.92	3.59	3.56	3.66	3.62	3.54	3.54	3.45	3.51	3.41	
Na <sub>2</sub> O	11.82	12.52	11.90	12.10	12.42	12.15	12.27	12.46	12.16	12.30	
Nd <sub>2</sub> O <sub>3</sub>	0.15	0.14	0.14	0.14	0.14	0.16	0.13	0.14	0.14	0.11	
NiO	0.17	0.24	0.22	0.23	0.23	0.21	0.21	0.23	0.22	0.21	
P <sub>2</sub> O <sub>5</sub>	0.09	0.10	0.11	0.11	0.12	0.11	0.11	0.10	0.11	0.12	
PbO	0.30	0.27	0.29	0.27	0.28	0.26	0.27	0.27	0.28	0.27	
Re <sub>2</sub> O <sub>7</sub>	0.05	0.010	0.017	0.015	0.021	0.018	0.017	0.018	0.015	0.016	
Sb <sub>2</sub> O <sub>3</sub>	0.25	0.28	0.30	0.28	0.30	0.23	0.16	0.27	0.33	0.28	
SeO <sub>2</sub>	0.15	0.02	0.02	0.01	0.01	0.01	0.01	0.01	0.02	0.02	
SiO <sub>2</sub>	47.00	46.36	46.70	46.81	46.42	46.90	46.75	46.65	46.83	47.04	
SnO <sub>2</sub>	§	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
SO <sub>3</sub>	0.15	0.16	0.16	0.16	0.15	0.15	0.14	0.17	0.16	0.15	
SrO	0.90	0.79	0.80	0.78	0.78	0.80	0.82	0.80	0.79	0.79	
TiO <sub>2</sub>	0.14	0.24	0.24	0.23	0.24	0.23	0.23	0.25	0.22	0.23	
ZnO	2.07	1.82	1.83	1.82	1.83	1.82	1.89	1.86	1.83	1.81	
ZrO <sub>2</sub>	0.25	0.34	0.34	0.33	0.33	0.33	0.34	0.34	0.34	0.33	
Sum	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 DCP-AES analyzed results on the first discharge glass.

NA - Not analyzed.

§ - Not a target constituent

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

Test		1 OX	2 OX							3 OX
Mass (kg)		97.50	100.94	104.10	106.64	110.16	114.20	117.52	121.20	124.38
Constituent	Target	10X-G-65A	10X-G-77A	10X-G-77B	10X-G-78B	10X-G-78D	10X-G-83C	10X-G-83E	10X-G-84B	10X-G-89B
Al <sub>2</sub> O <sub>3</sub>	5.23	6.09	6.85	6.70	6.45	6.32	6.22	6.03	6.14	6.09
As <sub>2</sub> O <sub>3</sub>	0.19	0.15	0.15	0.16	0.15	0.15	0.16	0.15	0.14	0.15
B <sub>2</sub> O <sub>3</sub> *	9.38	9.38	9.38	9.38	9.38	9.38	9.38	9.38	9.38	9.38
BaO	§	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Bi <sub>2</sub> O <sub>3</sub>	§	<0.01	0.06	0.04	0.03	0.02	0.01	0.01	0.01	0.01
CaO	0.30	0.35	0.60	0.54	0.49	0.45	0.43	0.40	0.38	0.38
CdO	0.25	0.30	0.27	0.25	0.29	0.26	0.26	0.24	0.27	0.27
Ce <sub>2</sub> O <sub>3</sub>	§	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Cl	0.12	0.07	0.03	0.05	0.05	0.06	0.07	0.08	0.06	0.07
Cr <sub>2</sub> O <sub>3</sub>	0.17	0.18	0.20	0.18	0.18	0.19	0.18	0.17	0.17	0.18
CuO	0.05	0.06	0.06	0.06	0.06	0.05	0.05	0.05	0.05	0.05
F	0.04	NA								
Fe <sub>2</sub> O <sub>3</sub>	12.36	11.93	12.13	11.94	12.19	12.16	12.31	12.43	12.03	12.34
I	0.10	0.03	0.00	0.03	0.04	0.04	0.04	0.05	0.04	0.05
K <sub>2</sub> O	§	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
La <sub>2</sub> O <sub>3</sub>	0.24	0.36	0.19	0.22	0.26	0.21	0.31	0.21	0.33	0.30
Li <sub>2</sub> O*	3.00	3.00	3.00	3.00	3.00	3.00	3.00	3.00	3.00	3.00
MgO	1.15	1.31	1.17	1.13	1.22	1.26	1.20	1.21	1.33	1.21
MnO	3.92	3.39	3.31	3.34	3.50	3.47	3.58	3.47	3.36	3.58
Na <sub>2</sub> O	11.82	12.25	11.89	12.34	11.75	11.92	11.36	11.78	12.41	11.78
Nd <sub>2</sub> O <sub>3</sub>	0.15	0.12	0.12	0.14	0.14	0.13	0.10	0.13	0.14	0.15
NiO	0.17	0.19	0.20	0.21	0.20	0.18	0.19	0.19	0.17	0.20
P <sub>2</sub> O <sub>5</sub>	0.09	0.12	0.16	0.15	0.14	0.13	0.11	0.12	0.11	0.11
PbO	0.30	0.26	0.28	0.27	0.29	0.28	0.26	0.28	0.27	0.28
Re <sub>2</sub> O <sub>7</sub>	0.05	0.019	<0.005	0.010	0.015	0.010	0.012	0.012	0.009	0.014
Sb <sub>2</sub> O <sub>3</sub>	0.25	0.20	0.28	0.17	0.31	0.28	0.26	0.26	0.29	0.30
SeO <sub>2</sub>	0.15	0.02	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.02
SiO <sub>2</sub>	47.00	46.90	46.33	46.40	46.48	46.68	47.11	46.88	46.59	46.60
SnO <sub>2</sub>	§	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
SO <sub>3</sub>	0.15	0.18	0.18	0.18	0.15	0.17	0.14	0.14	0.16	0.17
SrO	0.90	0.79	0.79	0.77	0.80	0.79	0.81	0.82	0.79	0.82
TiO <sub>2</sub>	0.14	0.23	0.23	0.24	0.23	0.23	0.21	0.23	0.23	0.23
ZnO	2.07	1.78	1.78	1.74	1.82	1.82	1.84	1.90	1.79	1.92
ZrO <sub>2</sub>	0.25	0.33	0.36	0.34	0.35	0.34	0.34	0.35	0.33	0.35
Sum	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 DCP-AES analyzed results on the first discharge glass.

NA - Not analyzed.

§ - Not a target constituent

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

Test		3 OX					5 OX			
Mass (kg)		127.12	131.26	135.24	138.28	141.60	145.90	150.02	153.52	156.86
Constituent	Target	10X-G-89D	10X-G-91C	10X-G-94B	10X-G-94B	10X-G-95D	10X-G-97B	10X-G-100A	10X-G-101B	10X-G-106A
Al <sub>2</sub> O <sub>3</sub>	5.23	6.00	5.95	6.01	5.91	6.02	5.99	6.06	6.00	5.96
As <sub>2</sub> O <sub>3</sub>	0.19	0.14	0.15	0.16	0.15	0.16	0.14	0.14	0.13	0.15
B <sub>2</sub> O <sub>3</sub> *	9.38	9.38	9.38	9.38	9.38	9.38	9.38	9.38	9.38	9.38
BaO	§	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Bi <sub>2</sub> O <sub>3</sub>	§	0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
CaO	0.30	0.36	0.37	0.39	0.36	0.33	0.36	0.35	0.33	0.35
CdO	0.25	0.27	0.27	0.28	0.28	0.26	0.25	0.26	0.25	0.26
Ce <sub>2</sub> O <sub>3</sub>	§	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Cl	0.12	0.08	0.08	0.07	0.07	0.07	0.06	0.06	0.06	0.06
Cr <sub>2</sub> O <sub>3</sub>	0.17	0.18	0.17	0.18	0.16	0.19	0.17	0.18	0.17	0.18
CuO	0.05	0.04	0.05	0.05	0.05	0.07	0.07	0.05	0.06	0.05
F	0.04	NA	NA	NA						
Fe <sub>2</sub> O <sub>3</sub>	12.36	12.10	12.23	12.12	11.97	11.96	11.73	11.91	12.19	12.13
I	0.10	0.03	0.03	0.03	0.03	0.03	0.04	0.01	0.02	0.01
K <sub>2</sub> O	§	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
La <sub>2</sub> O <sub>3</sub>	0.24	0.31	0.28	0.31	0.26	0.29	0.27	0.23	0.32	0.31
Li <sub>2</sub> O*	3.00	3.00	3.00	3.00	3.00	3.00	3.00	3.00	3.00	3.00
MgO	1.15	1.26	1.21	1.22	1.24	1.28	1.24	1.23	1.20	1.22
MnO	3.92	3.48	3.46	3.43	3.38	3.38	3.31	3.41	3.49	3.53
Na <sub>2</sub> O	11.82	12.10	12.28	11.98	12.17	12.18	12.53	12.25	12.03	12.40
Nd <sub>2</sub> O <sub>3</sub>	0.15	0.10	0.11	0.16	0.15	0.17	0.13	0.16	0.13	0.13
NiO	0.17	0.20	0.20	0.20	0.20	0.18	0.19	0.20	0.18	0.20
P <sub>2</sub> O <sub>5</sub>	0.09	0.11	0.11	0.12	0.09	0.10	0.11	0.09	0.10	0.11
PbO	0.30	0.26	0.28	0.27	0.27	0.27	0.26	0.26	0.26	0.25
Re <sub>2</sub> O <sub>7</sub>	0.05	0.020	0.021	0.024	0.023	0.015	0.014	0.014	0.011	0.018
Sb <sub>2</sub> O <sub>3</sub>	0.25	0.29	0.32	0.28	0.29	0.23	0.28	0.26	0.32	0.26
SeO <sub>2</sub>	0.15	0.02	0.03	0.02	0.02	0.02	0.02	0.01	0.01	0.01
SiO <sub>2</sub>	47.00	46.89	46.62	46.92	47.16	46.98	47.19	47.10	46.98	46.63
SnO <sub>2</sub>	§	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
SO <sub>3</sub>	0.15	0.16	0.17	0.18	0.16	0.19	0.16	0.15	0.13	0.14
SrO	0.90	0.79	0.81	0.81	0.80	0.80	0.77	0.79	0.80	0.82
TiO <sub>2</sub>	0.14	0.22	0.24	0.24	0.22	0.24	0.22	0.24	0.23	0.24
ZnO	2.07	1.84	1.84	1.83	1.85	1.85	1.80	1.84	1.82	1.84
ZrO <sub>2</sub>	0.25	0.33	0.33	0.34	0.34	0.34	0.32	0.33	0.34	0.34
Sum	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 DCP-AES analyzed results on the first discharge glass.

NA - Not analyzed.

§ - Not a target constituent

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

Test		5 OX		All	
Mass (kg)		161.66	166.64	0-167 kg	
Constituent	Target	10X-G-107B	10X-G-108A	Average	% Deviation
Al <sub>2</sub> O <sub>3</sub>	5.23	6.04	6.02	6.13	17.25
As <sub>2</sub> O <sub>3</sub>	0.19	0.14	0.14	0.14	NC
B <sub>2</sub> O <sub>3</sub> *	9.38	9.38	9.38	9.66	NC
BaO	§	<0.01	<0.01	0.01	NC
Bi <sub>2</sub> O <sub>3</sub>	§	<0.01	<0.01	0.01	NC
CaO	0.30	0.33	0.35	0.41	NC
CdO	0.25	0.25	0.23	0.26	NC
Ce <sub>2</sub> O <sub>3</sub>	§	<0.01	<0.01	<0.01	NC
Cl	0.12	0.07	0.08	0.06	NC
Cr <sub>2</sub> O <sub>3</sub>	0.17	0.16	0.15	0.20	NC
CuO	0.05	0.05	0.05	0.05	NC
F	0.04	NA	NA	NA	NC
Fe <sub>2</sub> O <sub>3</sub>	12.36	11.94	11.76	12.29	-0.56
I	0.10	0.02	0.01	0.02	NC
K <sub>2</sub> O	§	<0.01	<0.01	0.01	NC
La <sub>2</sub> O <sub>3</sub>	0.24	0.29	0.21	0.27	NC
Li <sub>2</sub> O*	3.00	3.00	3.00	2.91	NC
MgO	1.15	1.18	1.28	1.18	2.70
MnO	3.92	3.50	3.46	3.39	-13.52
Na <sub>2</sub> O	11.82	12.07	12.09	12.16	2.84
Nd <sub>2</sub> O <sub>3</sub>	0.15	0.14	0.14	0.13	NC
NiO	0.17	0.17	0.18	0.23	NC
P <sub>2</sub> O <sub>5</sub>	0.09	0.12	0.12	0.14	NC
PbO	0.30	0.25	0.25	0.29	NC
Re <sub>2</sub> O <sub>7</sub>	0.05	0.018	0.014	0.01	NC
Sb <sub>2</sub> O <sub>3</sub>	0.25	0.28	0.23	0.26	NC
SeO <sub>2</sub>	0.15	0.01	0.01	0.01	NC
SiO <sub>2</sub>	47.00	47.36	47.64	46.52	-1.03
SnO <sub>2</sub>	§	<0.01	<0.01	<0.01	NC
SO <sub>3</sub>	0.15	0.15	0.14	0.17	NC
SrO	0.90	0.77	0.78	0.76	NC
TiO <sub>2</sub>	0.14	0.23	0.22	0.23	NC
ZnO	2.07	1.78	1.74	1.72	-16.75
ZrO <sub>2</sub>	0.25	0.32	0.32	0.37	NC
Sum	100.00	100.00	100.00	100.00	NC

\* - Target values calculated based on simple well-stirred tank model using DCP-AES analyzed results on the first discharge glass.

NA - Not analyzed.

NC - Not calculated

§ - Not a target constituent

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

Test	Date	Time	Sample ID	Fe <sup>2+</sup> /Total Fe (%)	Re <sub>2</sub> O <sub>7</sub> (wt%)	Secondary Phases Observed	Depth of Glass Pool (inches)
Before 1 N	4/19/12	18:40	10W-D-127A	NA	<0.005	Yes	8.00
			10W-D-127B	NA	<0.005	No	8.00
			10W-D-127C	NA	<0.005	No	8.00
After 1 N	4/21/12	00:28	10W-D-141A	<0.1	0.015	No	7.25
Before 2 N	4/23/12	07:20	10W-D-141B	NA	<0.005	No	7.50
After 2 N	4/24/12	10:32	10W-D-19A	<0.1	0.013	No	8.50
After 3 N	4/25/12	11:02	10X-D-36A	<0.1	0.007	No	7.00
After 9 N	4/26/12	11:30	10X-D-43A	<0.1	0.011	No	7.25
After 1 OX	4/27/12	12:30	10X-D-65A	1.6	0.021	No	8.25
Before 2 OX	4/30/12	07:20	10X-D-65B	NA	0.007	No	7.50
After 2 OX	5/1/12	10:20	10X-D-84A	10.0	0.016	No	8.50
		10:37	10X-O-84A (Suction)	8.9	0.018	No	NA
After 3 OX	5/2/12	11:50	10X-D-97A	7.1	0.024	No	8.00
After 5 OX	5/3/12	12:12	10X-D-108A	10.7	0.020	No	8.00
		12:35	10X-O-108A (Suction)	10.0	0.014	Metal Film	NA

NA – Not analyzed

**Table 4.4. XRF Analyzed Compositions of Glass Pool Samples from DM10 Tests (wt%).**

Test		Before 1N	After 1N	Before 2N	After 2N	After 3N	After 9N
Constituent	Target	10W-D-127A	10W-D-141A	10W-D-141B	10X-D-19A	10X-D-36A	10W-D-43A
Al <sub>2</sub> O <sub>3</sub>	5.23	6.18	5.94	7.03	6.19	6.11	5.87
As <sub>2</sub> O <sub>3</sub>	0.19	<0.01	0.15	0.13	0.15	0.16	0.16
B <sub>2</sub> O <sub>3</sub> *	9.38	13.84	10.13	10.13	9.56	9.41	9.39
BaO	§	0.15	0.03	0.03	<0.01	<0.01	<0.01
Bi <sub>2</sub> O <sub>3</sub>	§	0.01	<0.01	0.06	0.02	<0.01	<0.01
CaO	0.30	0.59	0.38	0.69	0.45	0.38	0.35
CdO	0.25	0.03	0.26	0.24	0.29	0.27	0.29
Ce <sub>2</sub> O <sub>3</sub>	§	0.11	<0.01	<0.01	<0.01	<0.01	<0.01
Cl	0.12	0.02	0.05	0.02	0.05	0.05	0.05
Cr <sub>2</sub> O <sub>3</sub>	0.17	0.25	0.21	0.20	0.20	0.22	0.25
CuO	0.05	0.01	0.05	0.04	0.05	0.06	0.04
F	0.04	NA	NA	NA	NA	NA	NA
Fe <sub>2</sub> O <sub>3</sub>	12.36	15.10	12.63	12.49	12.36	12.26	12.19
I	0.10	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
K <sub>2</sub> O	§	0.35	<0.01	<0.01	<0.01	<0.01	<0.01
La <sub>2</sub> O <sub>3</sub>	0.24	0.10	0.27	0.26	0.28	0.33	0.28
Li <sub>2</sub> O*	3.00	1.55	2.77	2.77	2.94	2.99	3.00
MgO	1.15	0.32	1.14	1.02	1.22	1.19	1.30
MnO	3.92	2.64	3.34	3.12	3.27	3.49	3.64
Na <sub>2</sub> O	11.82	13.50	12.26	11.98	11.97	12.22	12.28
Nd <sub>2</sub> O <sub>3</sub>	0.15	<0.01	0.12	0.12	0.12	0.13	0.17
NiO	0.17	0.51	0.27	0.28	0.22	0.23	0.23
P <sub>2</sub> O <sub>5</sub>	0.09	0.59	0.17	0.25	0.15	0.14	0.12
PbO	0.30	0.49	0.29	0.32	0.29	0.28	0.28
Re <sub>2</sub> O <sub>7</sub>	0.05	<0.005	0.015	<0.005	0.013	0.007	0.011
Sb <sub>2</sub> O <sub>3</sub>	0.25	<0.01	0.30	0.23	0.30	0.33	0.30
SeO <sub>2</sub>	0.15	<0.01	0.02	<0.01	0.02	0.02	0.01
SiO <sub>2</sub>	47.00	41.83	46.01	45.69	46.63	46.38	46.49
SnO <sub>2</sub>	§	0.09	<0.01	<0.01	<0.01	<0.01	<0.01
SO <sub>3</sub>	0.15	0.45	0.18	0.18	0.18	0.20	0.15
SrO	0.90	0.17	0.73	0.66	0.76	0.77	0.78
TiO <sub>2</sub>	0.14	0.09	0.22	0.21	0.22	0.23	0.23
ZnO	2.07	0.08	1.66	1.41	1.73	1.77	1.82
ZrO <sub>2</sub>	0.25	0.97	0.40	0.45	0.36	0.36	0.32
Sum	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 DCP-AES analyzed results from previous test

§ - Not a target constituent

NA - Not analyzed by XRF

**Table 4.4. XRF Analyzed Compositions of Glass Pool Samples from DM10 Tests (wt%)  
(Continued).**

Test		After 1 OX	Before 2 OX	After 2 OX	After 3 OX	After 5 OX
Constituent	Target	10X-D-65A	10X-D-65B	10X-D-84A	10X-D-97A	10X-D-108A
Al <sub>2</sub> O <sub>3</sub>	5.23	5.93	6.85	6.03	5.97	5.96
As <sub>2</sub> O <sub>3</sub>	0.19	0.16	0.16	0.16	0.18	0.15
B <sub>2</sub> O <sub>3</sub> *	9.38	9.38	9.38	9.38	9.38	9.38
BaO	§	<0.01	<0.01	<0.01	<0.01	<0.01
Bi <sub>2</sub> O <sub>3</sub>	§	<0.01	0.05	0.01	<0.01	<0.01
CaO	0.30	0.36	0.67	0.37	0.36	0.35
CdO	0.25	0.26	0.27	0.25	0.27	0.26
Ce <sub>2</sub> O <sub>3</sub>	§	<0.01	<0.01	<0.01	<0.01	<0.01
Cl	0.12	0.06	0.03	0.07	0.08	0.06
Cr <sub>2</sub> O <sub>3</sub>	0.17	0.19	0.19	0.19	0.15	0.18
CuO	0.05	0.06	0.06	0.05	0.06	0.05
F	0.04	NA	NA	NA	NA	NA
Fe <sub>2</sub> O <sub>3</sub>	12.36	12.08	12.24	12.41	12.38	12.17
I	0.10	<0.01	<0.01	0.04	0.04	0.02
K <sub>2</sub> O	§	<0.01	<0.01	<0.01	<0.01	<0.01
La <sub>2</sub> O <sub>3</sub>	0.24	0.29	0.25	0.28	0.29	0.25
Li <sub>2</sub> O*	3.00	3.00	3.00	3.00	3.00	3.00
MgO	1.15	1.26	1.20	1.22	1.21	1.25
MnO	3.92	3.42	3.31	3.60	3.39	3.56
Na <sub>2</sub> O	11.82	12.27	11.90	11.91	11.94	12.10
Nd <sub>2</sub> O <sub>3</sub>	0.15	0.13	0.14	0.15	0.14	0.13
NiO	0.17	0.21	0.22	0.19	0.21	0.21
P <sub>2</sub> O <sub>5</sub>	0.09	0.11	0.16	0.12	0.11	0.12
PbO	0.30	0.26	0.30	0.29	0.28	0.26
Re <sub>2</sub> O <sub>7</sub>	0.05	0.021	0.007	0.016	0.024	0.020
Sb <sub>2</sub> O <sub>3</sub>	0.25	0.17	0.30	0.28	0.29	0.32
SeO <sub>2</sub>	0.15	0.02	0.00	0.01	0.02	0.01
SiO <sub>2</sub>	47.00	46.91	45.92	46.47	46.86	46.78
SnO <sub>2</sub>	§	<0.01	<0.01	<0.01	<0.01	<0.01
SO <sub>3</sub>	0.15	0.21	0.19	0.16	0.15	0.16
SrO	0.90	0.80	0.79	0.81	0.81	0.80
TiO <sub>2</sub>	0.14	0.24	0.25	0.24	0.23	0.24
ZnO	2.07	1.85	1.80	1.92	1.83	1.89
ZrO <sub>2</sub>	0.25	0.33	0.37	0.35	0.34	0.33
Sum	100.00	100.00	100.00	100.00	100.00	100.00

\* - Target values

§ - Not a target constituent

NA - Not analyzed by XRF

**Table 4.5. XRF Analyzed Composition of Suction Samples (wt%).**

Test		After 2 OX	After 5 OX
Constituent	Target	10X-O-84A	10X-O-108A
Al <sub>2</sub> O <sub>3</sub>	5.23	6.06	6.18
As <sub>2</sub> O <sub>3</sub>	0.19	0.16	0.13
B <sub>2</sub> O <sub>3</sub> *	9.38	9.38	9.38
BaO	§	<0.01	<0.01
Bi <sub>2</sub> O <sub>3</sub>	§	<0.01	<0.01
CaO	0.30	0.37	0.34
CdO	0.25	0.28	0.21
Ce <sub>2</sub> O <sub>3</sub>	§	<0.01	<0.01
Cl	0.12	0.07	0.08
Cr <sub>2</sub> O <sub>3</sub>	0.17	0.18	0.16
CuO	0.05	0.05	0.26
F	0.04	NA	NA
Fe <sub>2</sub> O <sub>3</sub>	12.36	12.41	11.07
I	0.10	0.04	0.02
K <sub>2</sub> O	§	<0.01	<0.01
La <sub>2</sub> O <sub>3</sub>	0.24	0.30	0.20
Li <sub>2</sub> O*	3.00	3.00	3.00
MgO	1.15	1.20	1.19
MnO	3.92	3.56	3.28
Na <sub>2</sub> O	11.82	12.16	12.40
Nd <sub>2</sub> O <sub>3</sub>	0.15	0.13	0.11
NiO	0.17	0.19	0.43
P <sub>2</sub> O <sub>5</sub>	0.09	0.11	0.10
PbO	0.30	0.27	0.24
Re <sub>2</sub> O <sub>7</sub>	0.05	0.018	0.014
Sb <sub>2</sub> O <sub>3</sub>	0.25	0.29	0.26
SeO <sub>2</sub>	0.15	0.01	0.47
SiO <sub>2</sub>	47.00	46.23	47.51
SnO <sub>2</sub>	§	<0.01	<0.01
SO <sub>3</sub>	0.15	0.15	0.15
SrO	0.90	0.84	0.70
TiO <sub>2</sub>	0.14	0.23	0.22
ZnO	2.07	1.93	1.63
ZrO <sub>2</sub>	0.25	0.35	0.29
Sum	100.00	100.00	100.00

\* - Target values calculated based on simple well-stirred tank model using DCP-AES analyzed results on the first discharge of the first test

§ - Not a target constituent

NA - Not analyzed by XRF

**Table 5.1. Results from DM10 Off-Gas Emission Samples.**

Particulate	Test 1N 04/20/12 15:35 – 16:35 7.0% Moisture, 92.6% Isokinetic				Test 2N 04/23/12 18:19 – 19:19 5.5 % Moisture, 97.7% Isokinetic			
	Feed <sup>#</sup> (mg/min)	Output (mg/min)	% Emitted	DF	Feed <sup>#</sup> (mg/min)	Output (mg/min)	% Emitted	DF
	Total <sup>\$</sup>	13679	128	0.94	107	11237	52.3	0.47
Al	352	1.04	0.29	339	282	0.37	0.13	764
As	18	0.92	5.10	19.6	15	0.24	1.63	61.4
B	371	2.59	0.70	143	297	0.51	0.17	582
Ca	27	0.31	1.13	88.2	22	0.15	0.68	147
Cd	29	0.42	1.47	68.1	23	0.18	0.78	129
Cl	16	2.39 <sup>*</sup>	15.0	6.7	13	0.93 <sup>¶</sup>	7.30	13.7
Cr	15	0.31	2.04	49.1	12	0.12	1.01	99.4
Cu	5.3	<0.10	<1.89	>53	4.2	<0.10	<2.36	>42
F	5.4	0.31 <sup>*</sup>	5.79	17.3	4.3	0.80 <sup>¶</sup>	18.7	5.4
Fe	1101	6.42	0.58	171	881	2.09	0.24	421
I	13	<0.10 <sup>*</sup>	<0.78	>127	10	<0.10 <sup>¶</sup>	<0.98	>102
La	26	<0.10	<0.39	>259	21	<0.10	<0.48	>207
Li	178	0.83	0.47	214	142	0.25	0.17	572
Mg	88	0.55	0.63	159	70	0.20	0.28	359
Mn	387	<0.10	<0.03	>3873	310	<0.10	<0.03	>3098
Na	1118	12.4	1.11	89.9	894	5.27	0.59	170
Ni	16.9	<0.10	<0.59	>169	14	<0.10	<0.74	>135
P	5.2	<0.10	<1.93	>52	4.1	<0.10	<2.42	>41
Pb	35.5	0.37	1.04	96.5	28	<0.10	<0.35	>284
Re	4.9	3.04	62.2	1.6	3.9	1.71	43.6	2.3
S	7.7	3.28 <sup>*</sup>	42.8	2.3	6.1	1.75 <sup>¶</sup>	28.6	3.5
Sb	27	0.24	0.89	113	21	<0.10	<0.47	>213
Se	13	6.25	46.9	2.1	11	2.37	22.2	4.5
Si	2799	5.70	0.20	491	2239	2.65	0.12	846
Sr	97	0.61	0.63	159	78	0.18	0.24	425
Ti	11	<0.10	<0.95	>105	8.4	<0.10	<1.19	>84
Zn	212	1.19	0.56	178	169	0.44	0.26	388
Zr	24	<0.10	<0.42	>241	19	<0.10	<0.52	>192
Gas	B	371	5.28	1.42	70.2	297	2.38	0.80
	Cl	16	2.11	13.2	7.6	13	0.83	6.53
	F	5.4	3.19	59.6	1.7	4.3	1.86	43.4
	I	13	11.1	87.1	1.1	10	7.99	78.4
	S	7.7	2.41	31.5	3.2	6.1	1.80	29.4
	Se	13	0.44	3.29	30.4	11	<0.10	<0.94

<sup>\$</sup> - From gravimetric analysis of filters and particulate nitric acid rinses<sup>#</sup> - Feed rate calculated from target composition and total glass production rate<sup>\*</sup> - Calculated from analysis of filter particulate by water dissolution and direct rinse analysis<sup>¶</sup> - Calculated from extrapolation of direct rinse analysis

NC - Not Calculated

**Table 5.1. Results from DM10 Off-Gas Emission Samples (Continued).**

	Test 3N 04/24/12 18:26 – 19:35 4.7% Moisture, 100% Isokinetic				Test 9N 04/25/12 18:20 – 19:20 6.6% Moisture, 102% Isokinetic				
	Feed <sup>#</sup> (mg/min)	Output (mg/min)	% Emitted	DF	Feed <sup>#</sup> (mg/min)	Output (mg/min)	% Emitted	DF	
	Total <sup>\$</sup>	11682	71.8	0.61	163	16813	152	0.91	110
Particulate	Al	285	0.47	0.16	610	404	0.61	0.15	667
	As	15	0.34	2.33	43.0	21	0.91	4.38	22.8
	B	300	2.09	0.70	144	425	2.28	0.54	187
	Ca	22	0.14	0.66	152	31	0.34	1.08	92.2
	Cd	23	0.30	1.31	76.1	33	0.93	2.85	35.1
	Cl	13	1.81 <sup>¶</sup>	14.05	7.1	18	12.3 <sup>¶</sup>	67.1	1.5
	Cr	12	0.22	1.80	55.5	17	0.24	1.38	72.3
	Cu	4.3	<0.10	<2.34	>43	6.1	<0.10	<1.65	>61
	F	4.3	1.03 <sup>¶</sup>	23.7	4.2	6.1	2.03 <sup>¶</sup>	33.2	3.0
	Fe	890	2.49	0.28	358	1262	3.12	0.25	404
	I	10	<0.10 <sup>¶</sup>	<0.97	>103	15	<0.10 <sup>¶</sup>	<0.68	>146
	La	21	<0.10	<0.48	>209	30	<0.10	<0.34	>297
	Li	143	0.53	0.37	273	204	0.90	0.44	226
	Mg	71	0.23	0.33	307	101	0.32	0.32	315
	Mn	313	<0.10	<0.03	>3130	444	<0.10	<0.02	>4442
	Na	903	7.27	0.80	124	1282	11.3	0.88	113
	Ni	14	<0.10	<0.73	>137	19	<0.10	<0.52	>194
	P	4.2	<0.10	<2.39	>42	5.9	<0.10	<1.69	>59
	Pb	29	0.11	0.39	259	41	0.21	0.51	198
	Re	4.0	2.10	53.1	1.9	5.6	2.86	51.0	2.0
	S	6.2	2.13 <sup>¶</sup>	34.5	2.9	8.8	3.30 <sup>¶</sup>	37.6	2.7
	Sb	22	<0.10	<0.47	>215	31	0.14	0.46	218
	Se	11	3.37	31.3	3.2	15	5.18	33.9	3.0
	Si	2262	3.23	0.14	701	3211	4.07	0.13	789
	Sr	78	0.21	0.27	372	111	0.28	0.25	400
	Ti	8.5	<0.10	<1.17	>85	12.1	<0.10	<0.83	>121
	Zn	171	0.54	0.32	317	243	0.70	0.29	345
	Zr	19	<0.10	<0.51	>194	28	<0.10	<0.36	>276
Gas	B	300	2.95	0.98	102	425	4.11	0.97	104
	Cl	13	1.75	13.6	7.4	18	1.42	7.78	12.9
	F	4.3	2.62	60.7	1.6	6.1	2.21	36.0	2.8
	I	10	7.04	68.4	1.5	15	8.81	60.3	1.7
	S	6.2	2.52	40.7	2.5	8.8	2.23	25.4	3.9
	Se	11	0.39	3.61	27.7	15	<0.10	<0.65	>153

<sup>\$</sup> - From gravimetric analysis of filters and particulate nitric acid rinses<sup>#</sup> - Feed rate calculated from target composition and total glass production rate<sup>\*</sup> - Calculated from analysis of filter particulate by water dissolution and direct rinse analysis<sup>¶</sup> - Calculated from extrapolation of direct rinse analysis

NC - Not Calculated

Table 5.1. Results from DM10 Off-Gas Emission Samples (Continued).

Particulate	Test 1OX 04/26/12 18:44 – 19:44 10.5% Moisture, 106% Isokinetic				Test 2OX 04/30/12 17:00 – 18:00 10.3 % Moisture, 107% Isokinetic				
	Feed <sup>#</sup> (mg/min)	Output (mg/min)	% Emitted	DF	Feed <sup>#</sup> (mg/min)	Output (mg/min)	% Emitted	DF	
	Total <sup>\$</sup>	19440	109	0.56	179	18703	62.4	0.33	300
Al	518	0.29	0.06	1760	486	< 0.10	< 0.02	> 4862	
As	27	1.85	6.94	14.4	25	2.63	10.49	9.5	
B	545	0.97	0.18	564	512	0.41	0.08	1246	
Ca	40	0.12	0.30	337	37	< 0.10	< 0.27	> 372	
Cd	42	2.48	5.91	16.9	39	4.21	10.7	9.4	
Cl	23	5.20*	22.2	4.5	22	2.85*	13.0	7.7	
Cr	22	0.12	0.53	189	21	< 0.10	< 0.48	> 210	
Cu	7.8	< 0.10	< 1.29	> 78	7.3	< 0.10	< 1.37	> 73	
F	7.9	0.44*	5.66	17.7	7.4	0.26*	3.54	28.3	
Fe	1617	1.93	0.12	839	1519	0.64	0.04	2358	
I	19	< 0.10*	< 0.53	> 187	18	< 0.10*	< 0.57	> 176	
La	38	< 0.10	< 0.26	> 380	36	< 0.10	< 0.28	> 357	
Li	261	0.84	0.32	311	245	0.45	0.18	546	
Mg	129	0.17	0.13	755	122	< 0.10	< 0.08	> 1216	
Mn	569	< 0.10	< 0.02	> 5690	534	< 0.10	< 0.02	> 5343	
Na	1642	8.30	0.51	198	1542	5.48	0.36	281	
Ni	25	< 0.10	< 0.40	> 249	23	< 0.10	< 0.43	> 233	
P	7.6	< 0.10	< 1.32	> 76	7.1	< 0.10	< 1.40	> 71	
Pb	52	0.87	1.66	60.3	49	1.11	2.27	44.0	
Re	7.2	3.06	42.5	2.4	14.5	2.83	19.54	5.1	
S	11.3	1.58*	14.0	7.1	10.6	1.50*	14.17	7.1	
Sb	39	0.29	0.74	134	37	0.28	0.76	132	
Se	20	7.36	37.6	2.7	18	3.91	21.28	4.7	
Si	4113	1.56	0.04	2635	3862	0.55	0.01	6983	
Sr	143	0.17	0.12	850	134	< 0.10	< 0.07	> 1339	
Ti	15.5	< 0.10	< 0.65	> 155	14.5	< 0.10	< 0.69	> 145	
Zn	311	0.48	0.16	644	292	0.24	0.08	1217	
Zr	35	< 0.10	< 0.28	> 353	33	< 0.10	< 0.30	> 332	
Gas	B	545	6.45	1.18	84.5	512	5.71	1.12	89.6
	Cl	23	0.50	2.15	46.5	22	1.32	5.99	16.7
	F	7.9	2.96	37.6	2.7	7.4	2.48	33.6	3.0
	I	19	7.71	41.2	2.4	18	7.84	44.6	2.2
	S	11.3	8.07	71.7	1.4	10.6	10.4	98.5	1.0
	Se	20	< 0.10	< 0.51	> 196	18	< 0.10	< 0.54	> 184

<sup>\$</sup> - From gravimetric analysis of filters and particulate nitric acid rinses<sup>#</sup> - Feed rate calculated from target composition and total glass production rate<sup>\*</sup> - Calculated from analysis of filter particulate by water dissolution and direct rinse analysis

NC – Not Calculated

Table 5.1. Results from DM10 Off-Gas Emission Samples (Continued).

Particulate	Test 3OX 05/01/12 16:39 – 17:29 7.9% Moisture, 95.9% Isokinetic				Test 5OX 05/02/12 16:26 – 17:26 7.7% Moisture, 102% Isokinetic				
	Feed <sup>#</sup> (mg/min)	Output (mg/min)	% Emitted	DF	Feed <sup>#</sup> (mg/min)	Output (mg/min)	% Emitted	DF	
	Total <sup>\$</sup>	15531	106	0.68	146	17508	95.7	0.55	183
Particulate	Al	384	0.68	0.18	568	433	0.28	0.06	1566
	As	20	0.67	3.38	29.6	22	2.77	12.4	8.0
	B	404	3.43	0.85	118	456	2.81	0.62	162
	Ca	29	0.29	1.00	100	33	0.14	0.42	240
	Cd	31	2.38	7.65	13.1	35	4.80	13.7	7.3
	Cl	17	4.95 <sup>¶</sup>	28.50	3.5	20	1.63*	8.34	12.0
	Cr	17	< 0.10	< 0.60	> 166	19	< 0.10	< 0.53	> 187
	Cu	5.8	< 0.10	< 1.73	> 58	6.5	< 0.10	< 1.54	> 65
	F	5.8	0.78 <sup>¶</sup>	13.4	7.5	6.6	0.35*	5.31	18.8
	Fe	1200	3.27	0.27	367	1352	1.57	0.12	861
	I	14	< 0.10 <sup>¶</sup>	< 0.72	> 139	16	< 0.10*	< 0.64	> 157
	La	28	< 0.10	< 0.35	> 282	32	< 0.10	< 0.31	> 318
	Li	193	0.77	0.40	251	218	0.92	0.42	237
	Mg	96	0.42	0.44	227	108	0.23	0.22	464
	Mn	422	< 0.10	< 0.02	> 4220	476	< 0.10	< 0.02	> 4758
	Na	1218	8.00	0.66	152	1373	8.42	0.61	163
	Ni	18	< 0.10	< 0.54	> 184	21	< 0.10	< 0.48	> 208
	P	5.6	< 0.10	< 1.77	> 56	6.4	< 0.10	< 1.57	> 64
	Pb	39	0.98	2.53	39.5	44	1.55	3.54	28.2
	Re	5.3	2.40	45.0	2.2	6.0	3.08	51.1	2.0
	S	8.3	0.56 <sup>¶</sup>	6.75	14.8	9.4	2.74*	29.1	3.4
	Sb	29	< 0.10	< 0.34	> 290	33	0.48	1.47	67.9
	Se	15	7.44	51.2	2.0	16	7.62	46.5	2.1
	Si	3051	5.12	0.17	595	3439	1.51	0.04	2283
	Sr	106	0.26	0.25	401	119	0.13	0.11	933
	Ti	12	< 0.10	< 0.87	> 115	13	< 0.10	< 0.77	> 129
	Zn	231	0.90	0.39	255	260	0.43	0.16	608
	Zr	26	< 0.10	< 0.38	> 262	30	< 0.10	< 0.34	> 295
Gas	B	404	2.44	0.60	166	456	7.42	1.63	61.4
	Cl	17	0.44	2.52	39.6	20	4.64	23.7	4.2
	F	5.8	0.26	4.38	22.8	6.6	3.35	51.0	2.0
	I	14	18.5	133	0.8	16	12.3	78.8	1.3
	S	8.3	4.63	55.5	1.8	9.4	5.45	57.9	1.7
	Se	15	< 0.10	< 0.69	> 145	16	0.34	2.08	48.0

<sup>\$</sup> - From gravimetric analysis of filters and particulate nitric acid rinses<sup>#</sup> - Feed rate calculated from target composition and total glass production rate<sup>\*</sup> - Calculated from analysis of filter particulate by water dissolution and direct rinse analysis<sup>¶</sup> - Calculated from extrapolation of direct rinse analysis

NC - Not Calculated

**Table 5.2. Concentrations [ppmv] of Selected Species in Off-Gas Measured by FTIR Spectroscopy During the DM10 Tests.**

	1 N		2 N		3 N		9 N	
	Avg.	Range	Avg.	Range	Avg.	Range	Avg.	Range
N <sub>2</sub> O	<1.0	<1.0 – 2.2	<1.0	<1.0 – 1.1	<1.0	<1.0 – 1.1	8.3	2.3 – 17.5
NO	93.7	<1.0 – 794	115	43.2 – 330	144	2.3 – 405	150	18.3 – 392
NO <sub>2</sub>	22.0	<1.0 – 143	31.8	9.7 – 70.2	34.2	3.1 – 121	9.8	1.1 – 35.6
NH <sub>3</sub>	<1.0	NA	<1.0	NA	<1.0	NA	1.5	<1.0 – 3.6
H <sub>2</sub> O [%]	2.4	1.0 – 5.0	1.8	1.1 – 3.2	1.5	0.5 – 2.5	2.2	1.0 – 3.2
CO <sub>2</sub>	640	417 - 1961	596	475 - 823	585	430 - 931	872	529 - 1578
Nitrous Acid	<1.0	<1.0 – 5.6	<1.0	NA	<1.0	<1.0 – 2.2	<1.0	NA
Nitric Acid	<1.0	<1.0 – 2.2	<1.0	NA	<1.0	<1.0 – 1.2	<1.0	NA
HCN	<1.0	NA	<1.0	NA	<1.0	NA	<1.0	NA
CO	<1.0	<1.0 – 2.4	<1.0	<1.0 – 1.1	<1.0	<1.0 – 1.4	4.7	<1.0 – 11.0
HCl	<1.0	NA	<1.0	NA	<1.0	NA	<1.0	<1.0 – 1.0
HF	<1.0	<1.0 – 1.1	<1.0	NA	<1.0	NA	<1.0	NA

NA : Not applicable.

**Table 5.2. Concentrations [ppmv] of Selected Species in Off-Gas Measured by FTIR Spectroscopy During the DM10 Tests (Continued).**

	1 OX		2 OX		3 OX		5 OX	
	Avg.	Range	Avg.	Range	Avg.	Range	Avg.	Range
N <sub>2</sub> O	2.1	1.0 – 4.7	2.4	<1.0 – 9.5	5.2	<1.0 – 11.4	4.5	<1.0 – 14.2
NO	12.0	4.0 – 30.8	6.7	<1.0 – 37.4	29.0	<1.0 – 62.4	33.6	<1.0 – 78.6
NO <sub>2</sub>	<1.0	<1.0 – 1.3	<1.0	<1.0 – 4.1	<1.0	<1.0 – 1.5	2.7	<1.0 – 6.9
NH <sub>3</sub>	<1.0	<1.0 – 1.6	<1.0	NA	<1.0	NA	<1.0	NA
H <sub>2</sub> O [%]	2.9	1.5 – 5.3	2.6	1.1 – 4.4	2.3	1.6 – 3.1	2.3	1.5 – 3.3
CO <sub>2</sub>	993	638 - 1838	1036	465 - 3468	821	433 - 1217	912	461 - 1505
Nitrous Acid	<1.0	NA	<1.0	NA	<1.0	NA	<1.0	NA
Nitric Acid	<1.0	NA	<1.0	NA	<1.0	NA	<1.0	NA
HCN	<1.0	NA	<1.0	NA	<1.0	NA	<1.0	NA
CO	35.5	12.0 – 106	92.1	2.6 – 464	26.0	<1.0 – 67.6	75.2	<1.0 – 186
HCl	<1.0	NA	<1.0	NA	<1.0	NA	<1.0	NA
HF	<1.0	NA	<1.0	NA	<1.0	NA	<1.0	NA

NA : Not applicable.

**Table 6.1. Mass Balance for Volatile Species (%).**

	Feed (Wt % as Glass)			1 N			2 N			3 N			9 N		
	Measured Feed	Target	Used in Mass Balance	Glass	Emissions	Total									
As <sub>2</sub> O <sub>3</sub>	0.16	0.19	Measured	94	6.0	100	92	1.9	93	101	2.7	104	99	5.1	104
CdO	0.29	0.25	Measured	93	1.3	94	94	0.7	95	97	1.1	98	101	2.5	103
Cl	0.06	0.12	Target	42	14.7	56	37	13.8	51	39	21.8	61	38	74.9	112
Cr <sub>2</sub> O <sub>3</sub>	0.18	0.17	Measured	117	14.2	131	109	1.0	110	113	1.7	115	125	1.3	127
CuO	0.06	0.05	Measured	100	1.7	102	88	< 1.8	88	99	< 2.2	99	100	< 1.5	100
I	< 0.01	0.10	Target	< 1	87.1	87	< 1	78.4	78	< 1	68.4	68	< 1	60.3	60
PbO	0.29	0.30	Measured	107	1.1	108	99	< 0.4	99	95	0.4	96	97	0.5	98
Re <sub>2</sub> O <sub>7</sub>	0.02	0.05	Target	36	62.2	98	25	43.6	69	27	53.1	80	22	51	73
Sb <sub>2</sub> O <sub>3</sub>	0.30	0.25	Measured	83	0.7	84	76	< 0.5	76	91	< 0.5	91	101	< 0.5	101
SeO <sub>2</sub>	0.03	0.15	Target	13	50.2	64	11	22.2	33	10	34.9	45	9	33.9	43
SO <sub>3</sub>	0.22	0.15	Measured	95	50.7	146	87	39.5	127	77	51.3	128	70	43.0	113

**Table 6.1. Mass Balance for Volatile Species (%) (Continued).**

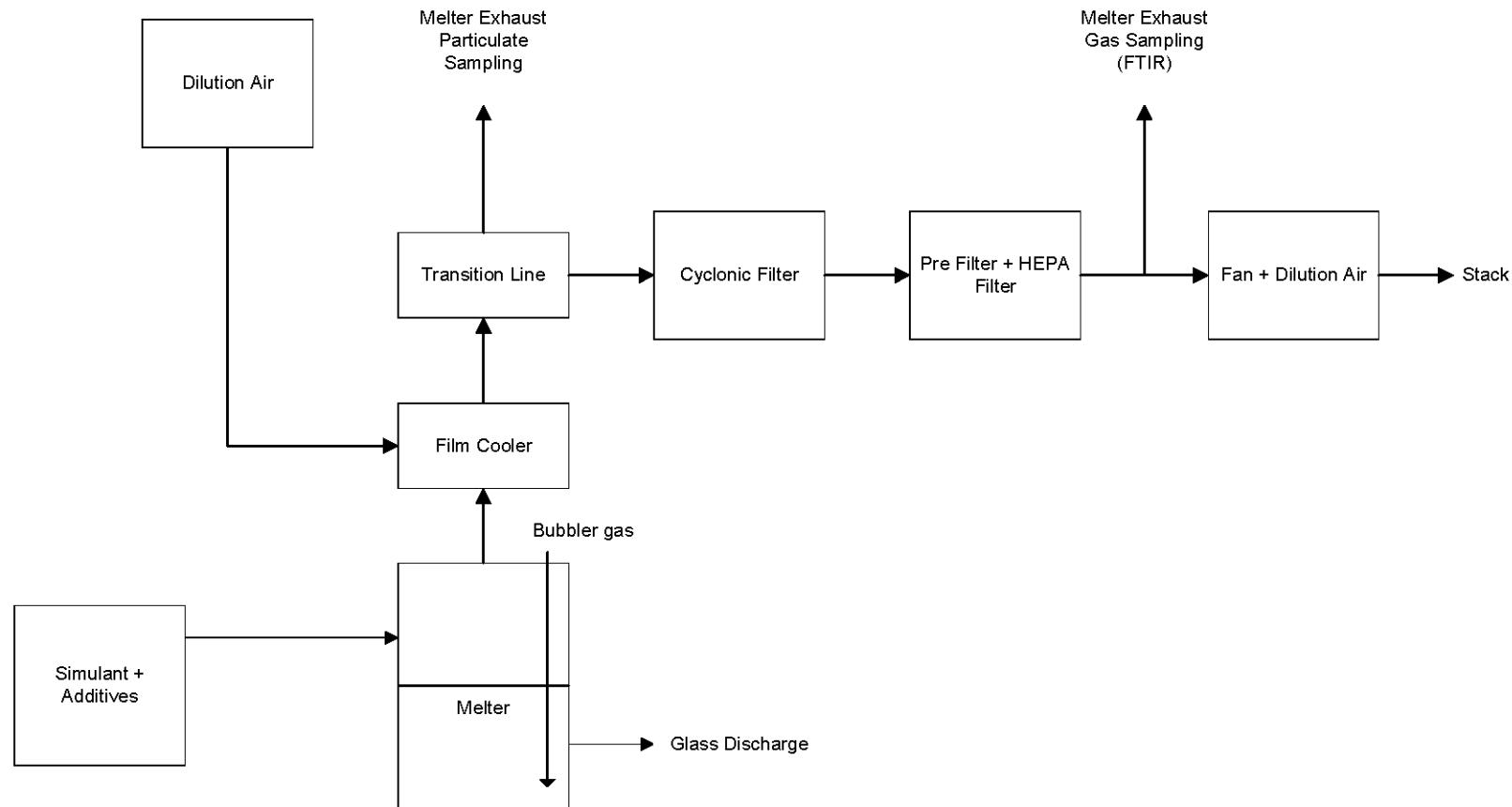
	Feed (Wt % as Glass)			1 OX			2 OX			3 OX			5 OX		
	Measured Feed	Target	Used in Mass Balance	Glass	Emissions	Total									
As <sub>2</sub> O <sub>3</sub>	0.16	0.19	Measured	95	8.2	104	94	12.3	107	98	4.0	102	88	14.6	103
CdO	0.29	0.25	Measured	94	5.1	99	89	9.2	98	94	6.6	100	86	11.8	97
Cl	0.06	0.12	Target	52	24.4	77	55	19.0	74	58	31.0	89	54	32.0	86
Cr <sub>2</sub> O <sub>3</sub>	0.18	0.17	Measured	104	0.5	104	98	< 0.5	98	98	< 0.6	98	92	< 0.5	92
CuO	0.06	0.05	Measured	97	< 1.3	97	88	< 1.3	88	92	< 1.7	92	90	< 1.5	90
I	< 0.01	0.10	Target	34	41.2	75	45	44.6	90	30	133	163	18	78.8	97
PbO	0.29	0.30	Measured	94	1.7	96	95	2.3	97	94	2.6	96	88	3.7	91
Re <sub>2</sub> O <sub>7</sub>	0.02	0.05	Target	34	42.5	76	22	19.54	41	42	45	87	31	51.1	82
Sb <sub>2</sub> O <sub>3</sub>	0.30	0.25	Measured	90	0.6	91	91	0.6	91	93	< 0.3	93	91	1.2	92
SeO <sub>2</sub>	0.03	0.15	Target	11	37.6	49	7	21.3	28	15	51.2	66	8	48.6	57
SO <sub>3</sub>	0.22	0.15	Measured	74	58.4	132	70	76.8	146	80	42.4	123	64	59.3	124

**Table 6.2. Nitrogen Oxides and Carbon Monoxide Mass Balance.**

Test	Feed		Emissions			
	Carbon Feed rate (mol/hr)	NO <sub>x</sub> Feed rate (mol/hr)	Gaseous Species	Rate (mol/hr)	Feed Carbon Emitted as CO (%)	Feed NO <sub>x</sub> in Exhaust (%)
1 N	0.000	0.741	CO	< 0.01	NA	116
			NO	0.694		
			NO <sub>2</sub>	0.163		
			Total NO <sub>x</sub>	0.857		
2 N	0.000	0.889	CO	< 0.01	NA	122
			NO	0.851		
			NO <sub>2</sub>	0.235		
			Total NO <sub>x</sub>	1.087		
3 N	0.000	1.203	CO	< 0.01	NA	110
			NO	1.066		
			NO <sub>2</sub>	0.253		
			Total NO <sub>x</sub>	1.319		
9 N	1.354	1.696	CO	0.035	2.57	70
			NO	1.110		
			NO <sub>2</sub>	0.073		
			Total NO <sub>x</sub>	1.183		
1 OX	2.340	0.002	CO	0.263	11.2	4812
			NO	0.089		
			NO <sub>2</sub>	< 0.01		
			Total NO <sub>x</sub>	0.096		
2 OX	4.393	0.002	CO	0.682	15.5	2850
			NO	0.050		
			NO <sub>2</sub>	< 0.01		
			Total NO <sub>x</sub>	0.057		
3 OX	3.456	0.403	CO	0.192	5.57	55
			NO	0.215		
			NO <sub>2</sub>	< 0.01		
			Total NO <sub>x</sub>	0.222		
5 OX	5.874	0.457	CO	0.557	9.48	56
			NO	0.249		
			NO <sub>2</sub>	0.007		
			Total NO <sub>x</sub>	0.256		



**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.**

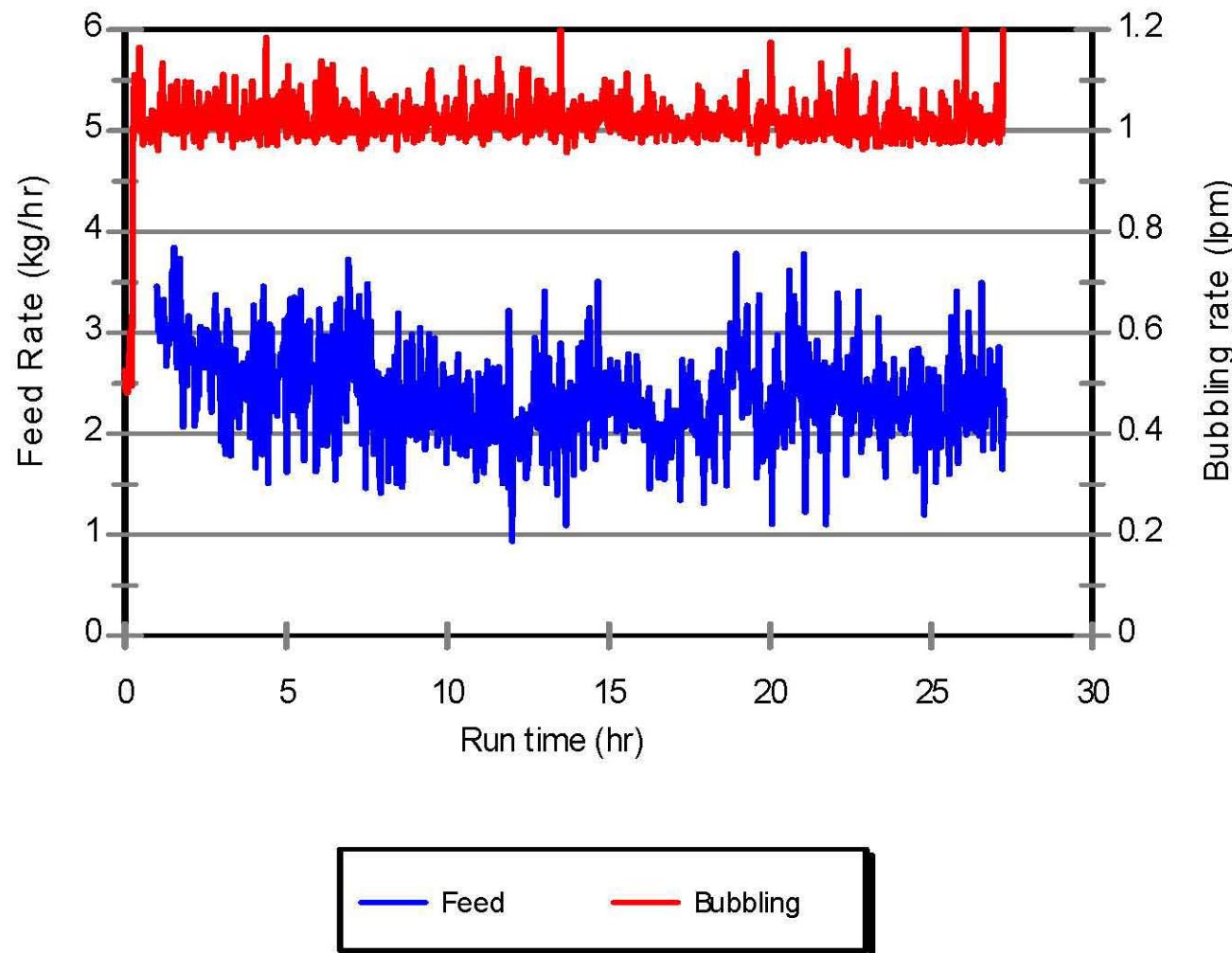
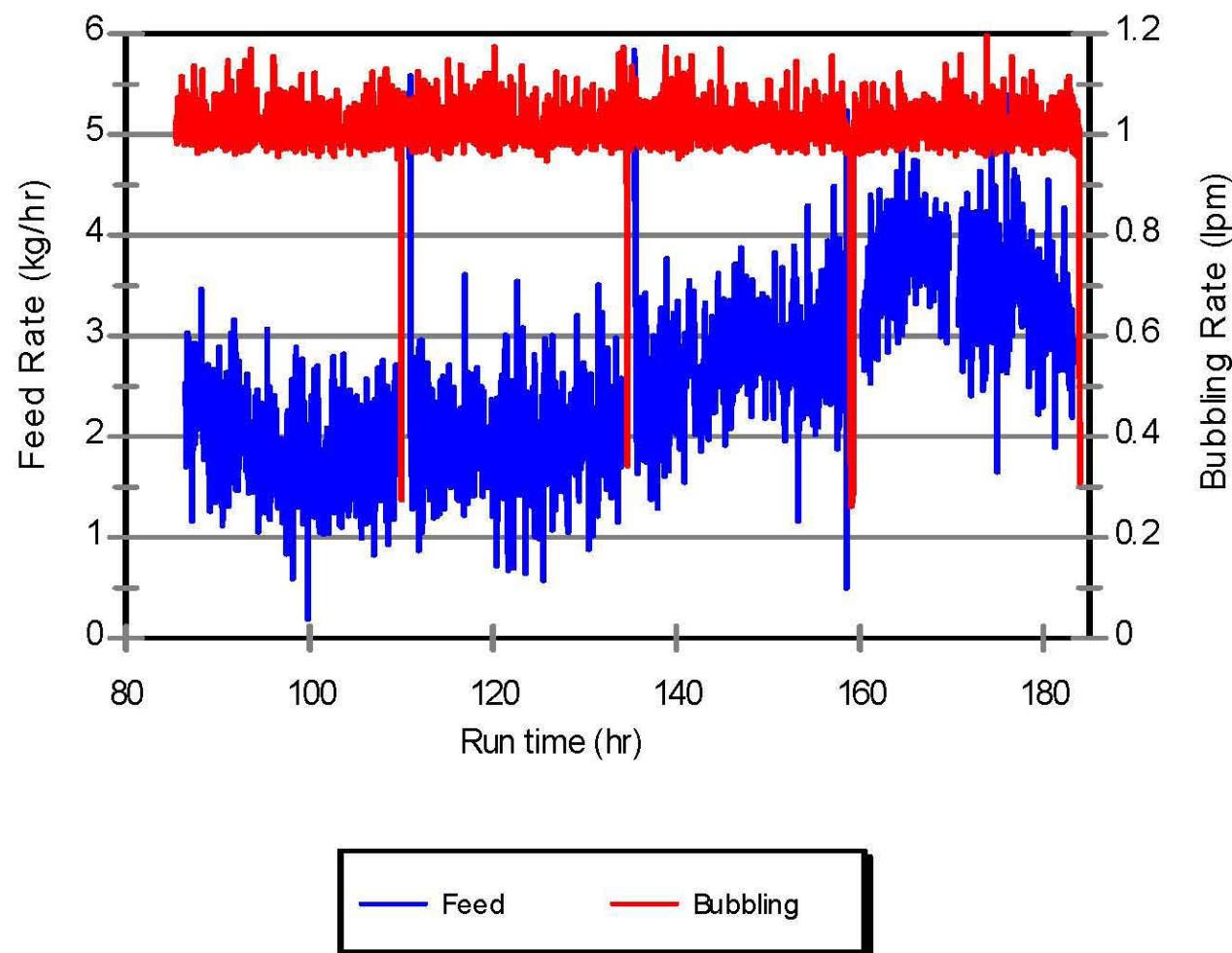
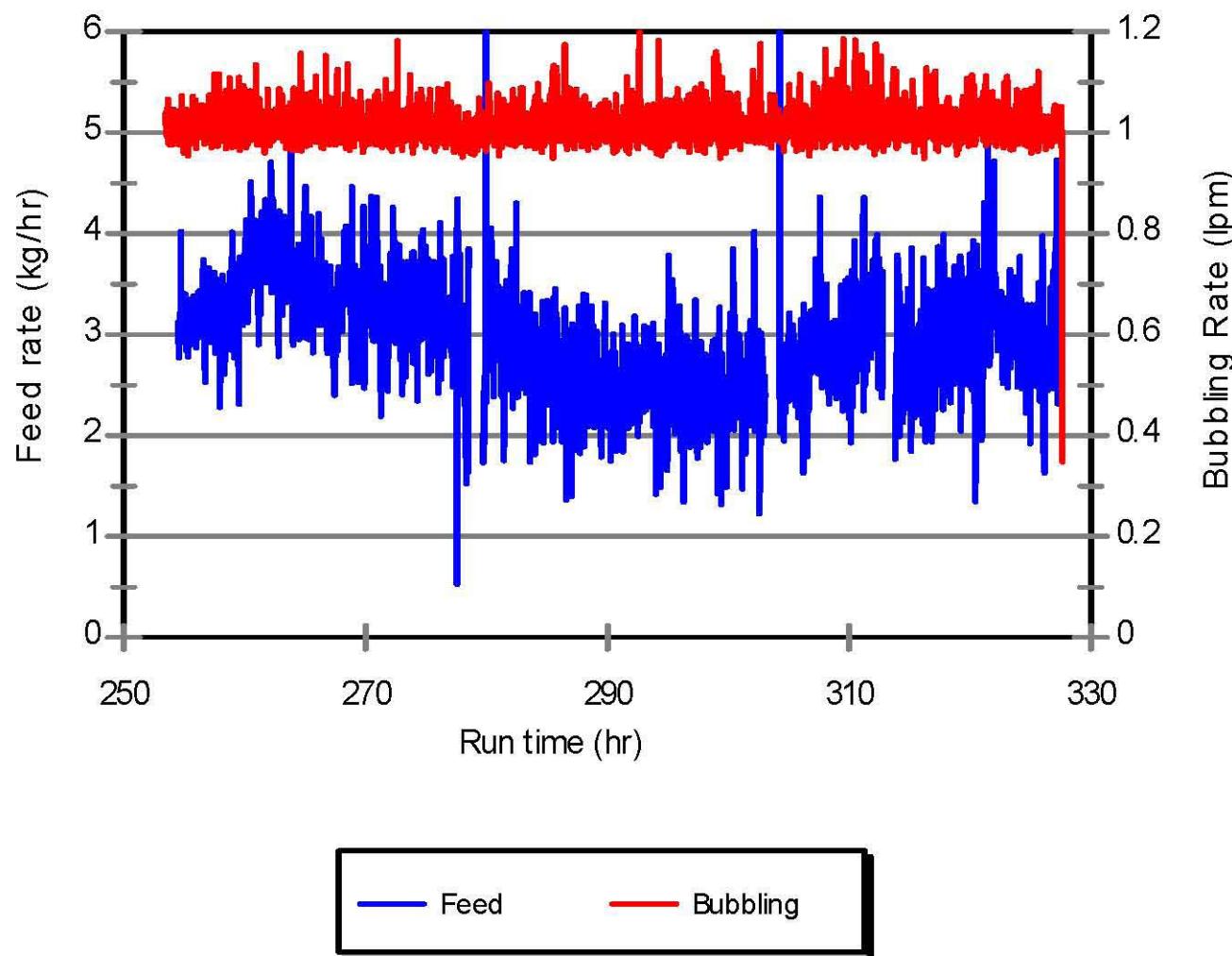


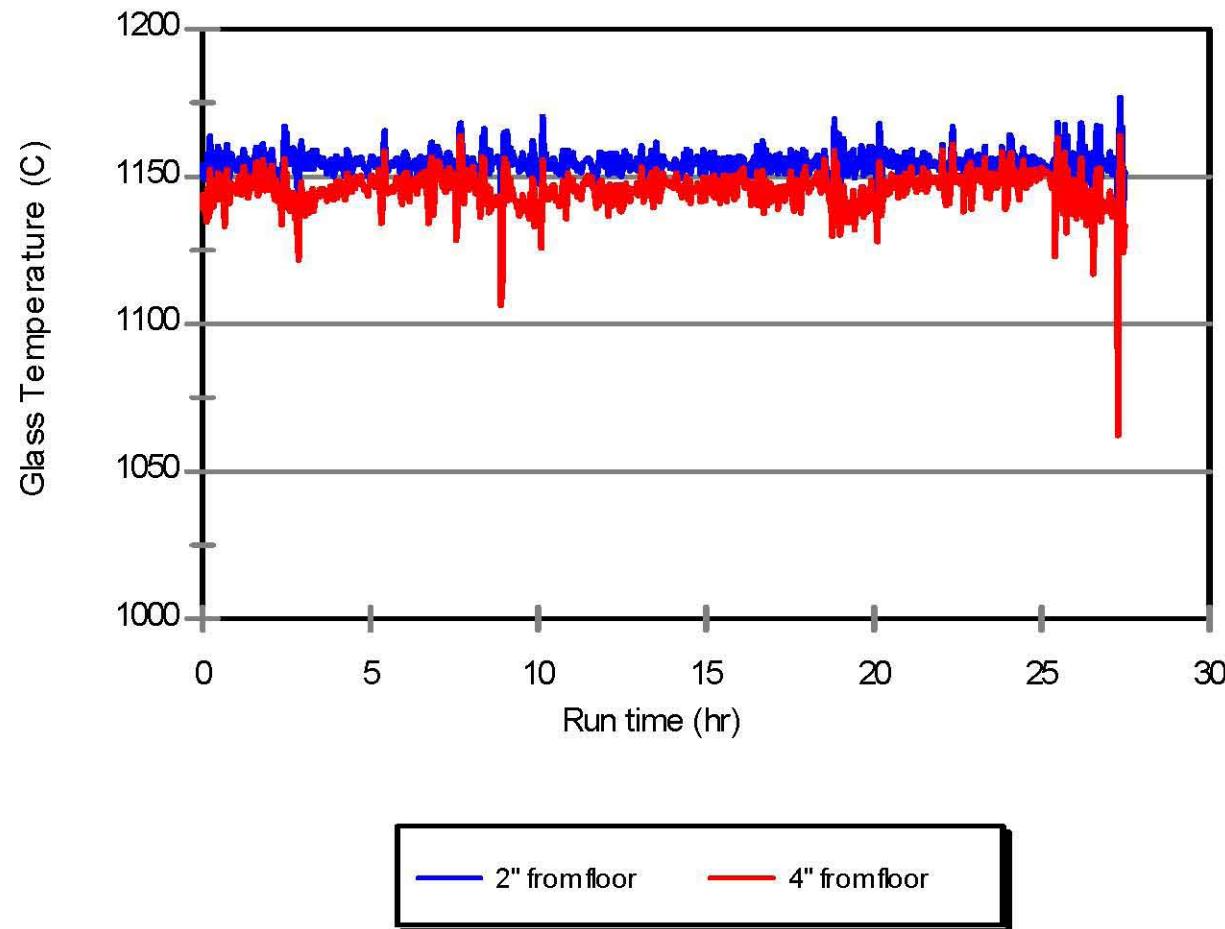
Figure 3.1.a. Feed and bubbling rates for DM10 Test 1 N.



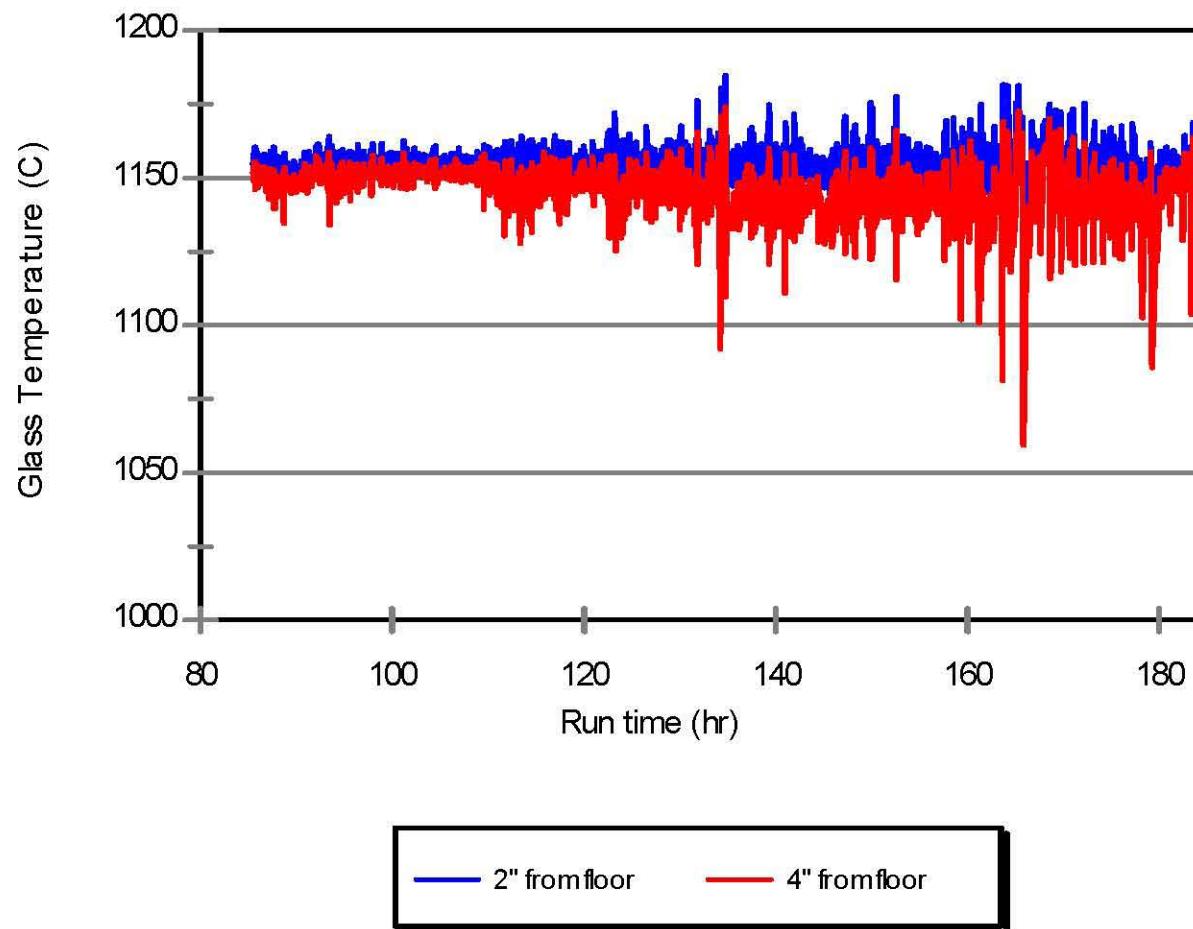
**Figure 3.1.b. Feed and bubbling rates for DM10 Tests 2 N, 3 N, 9 N, and 1 OX.**



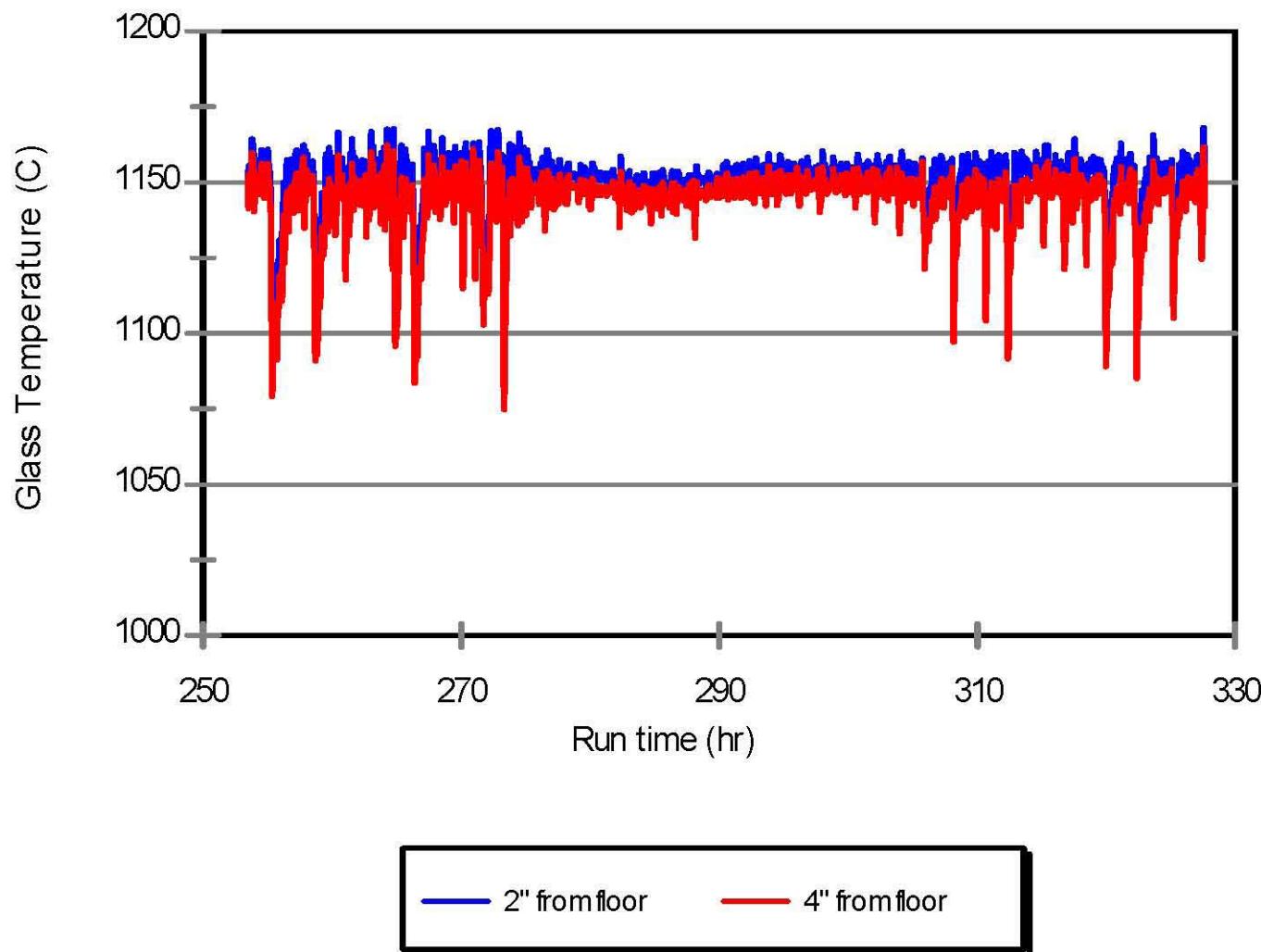
**Figure 3.1.c. Feed and bubbling rates for DM10 Tests 2 OX, 3 OX, and 5 OX.**



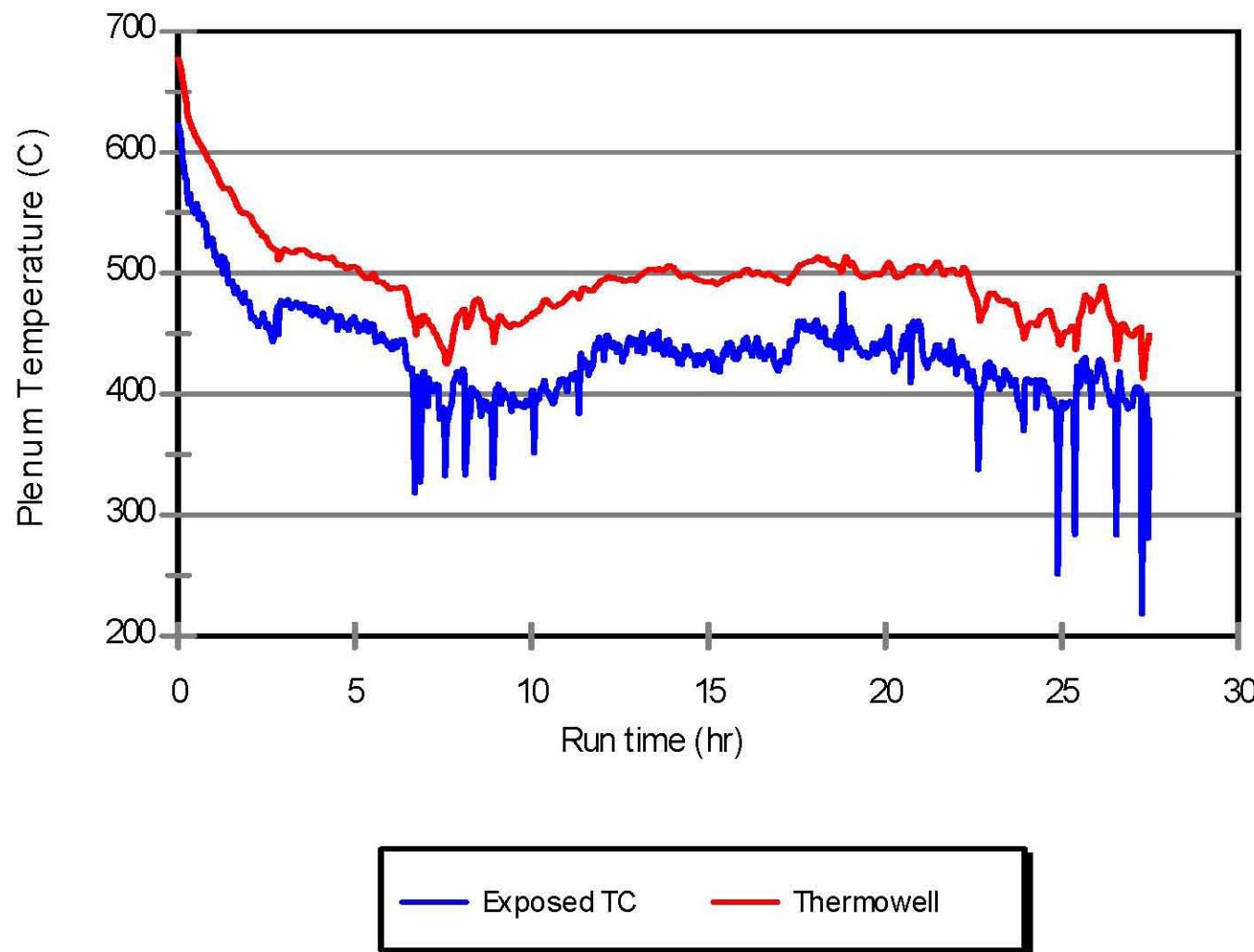
**Figure 3.2.a. Glass temperatures for DM10 Test 1 N.**



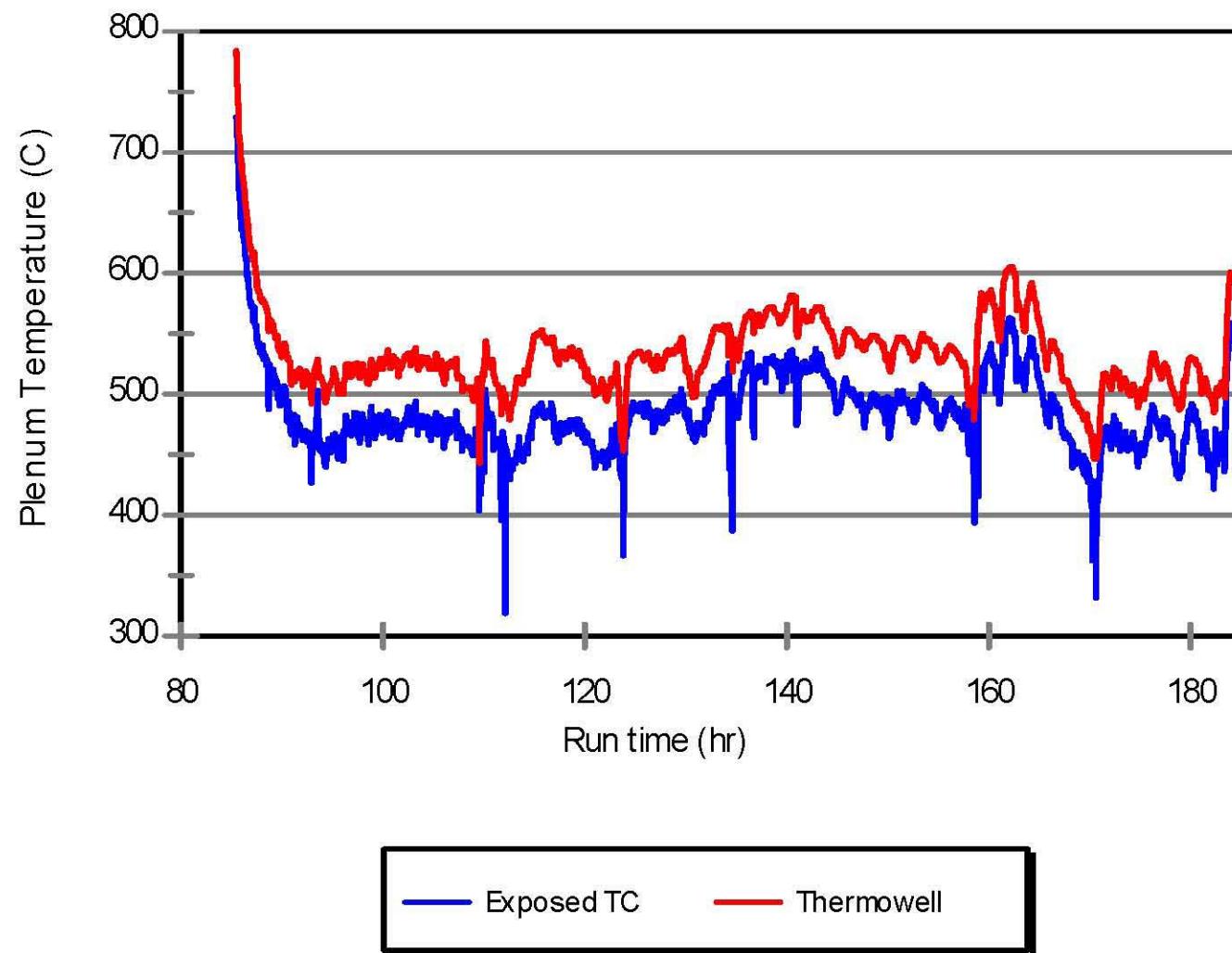
**Figure 3.2.b Glass temperatures for DM10 Tests 2 N, 3 N, 9N, and 1 OX.**



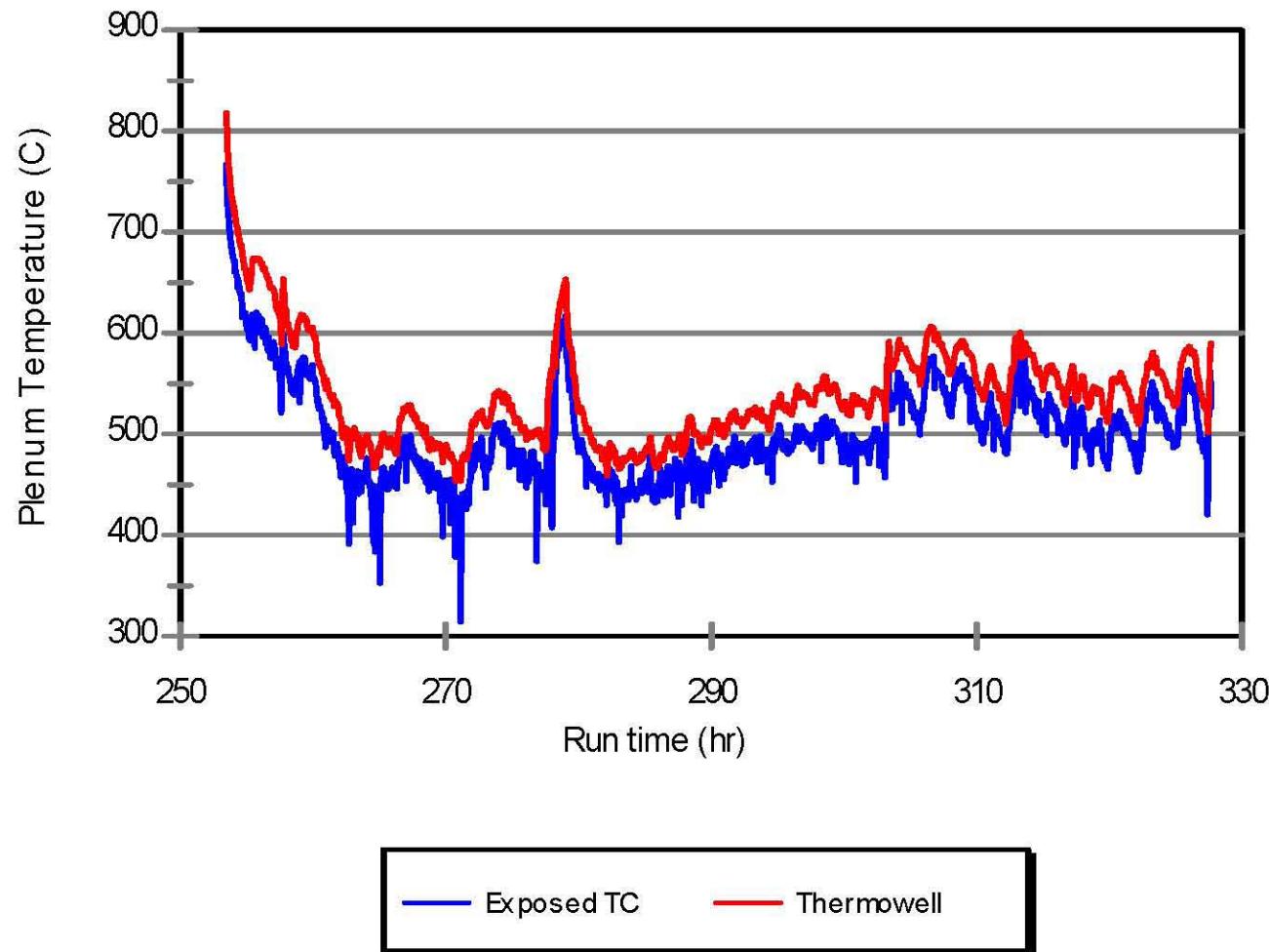
**Figure 3.2.c Glass temperatures for DM10 Tests 2 OX, 3 OX, and 5 OX.**



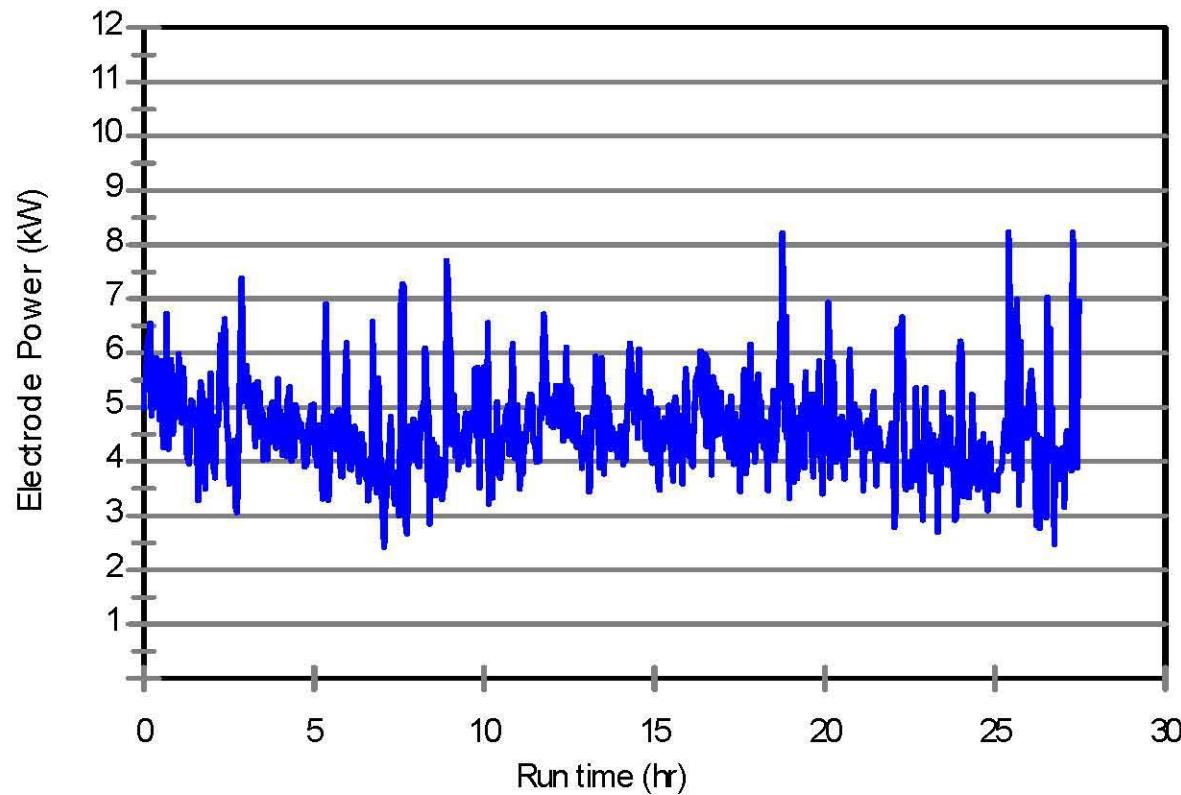
**Figure 3.3.a. Plenum temperatures for DM10 Test 1 N.**



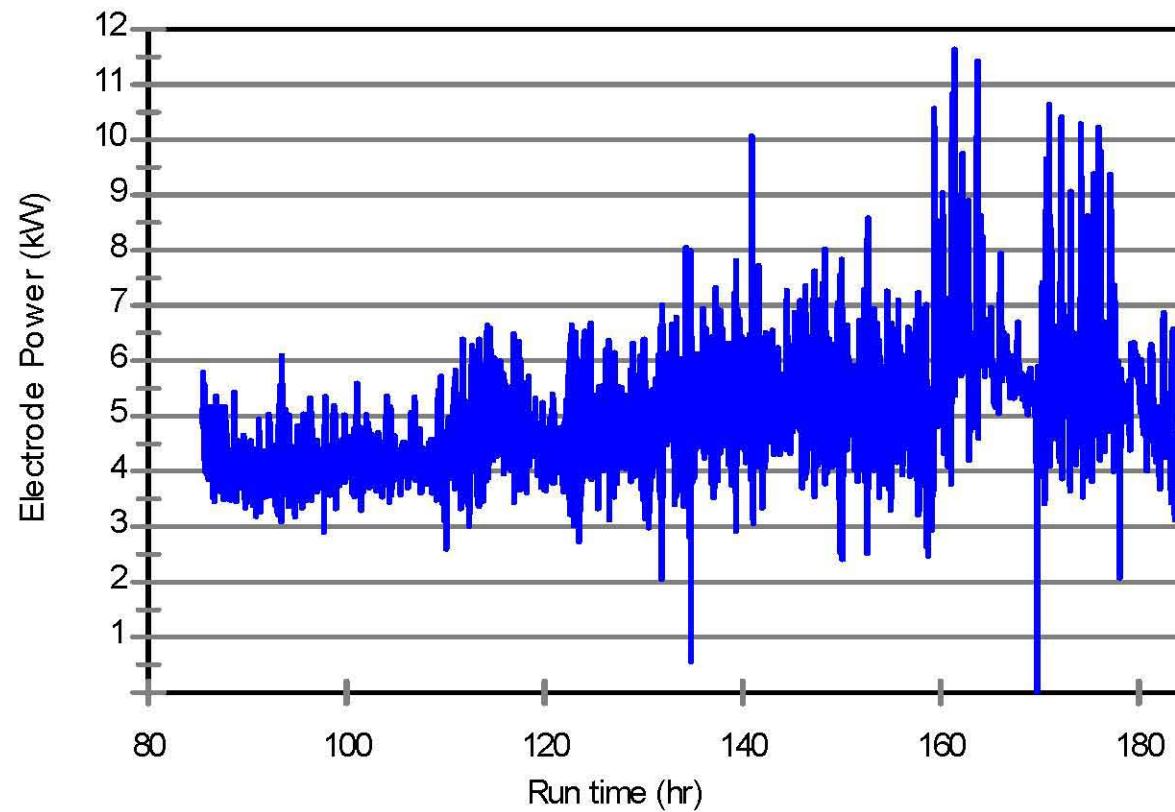
**Figure 3.3.b. Plenum temperatures for DM10 Tests 2 N, 3 N, 9 N, and 1 OX.**



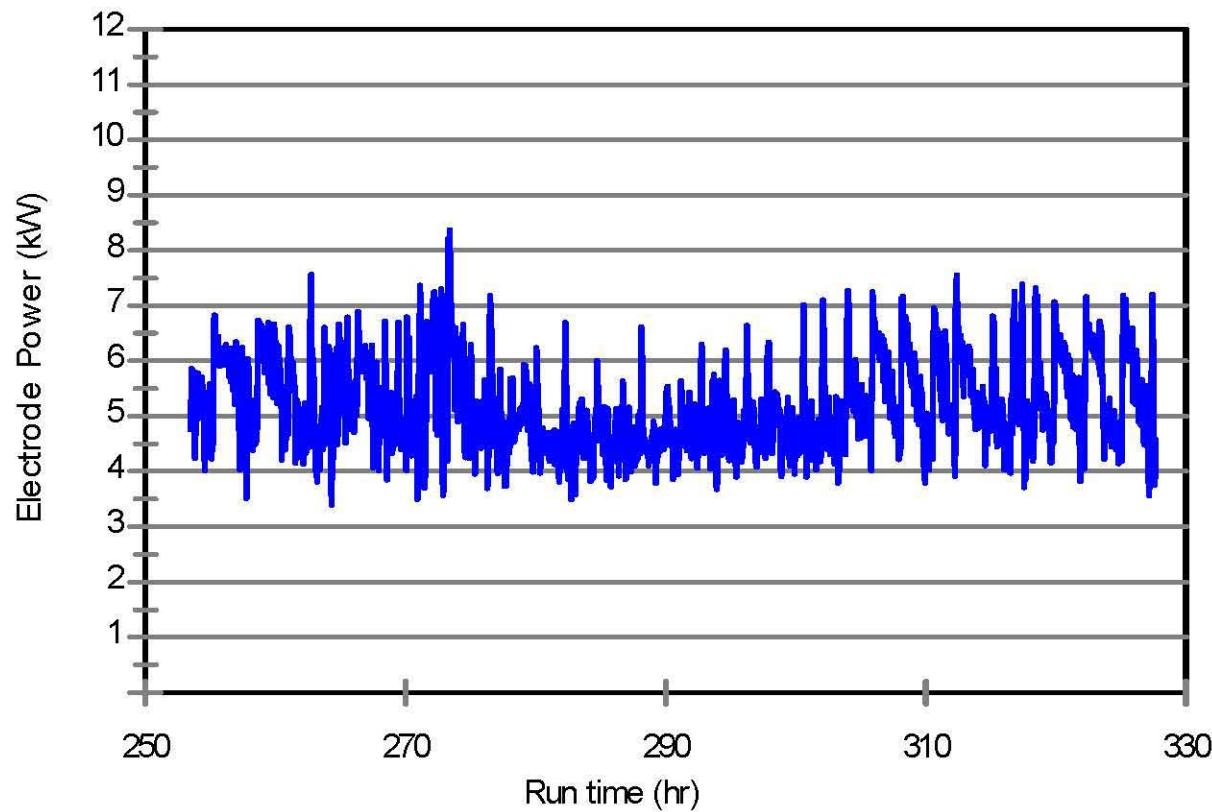
**Figure 3.3.c. Plenum temperatures for DM10 Tests 2 OX, 3 OX, and 5 OX.**



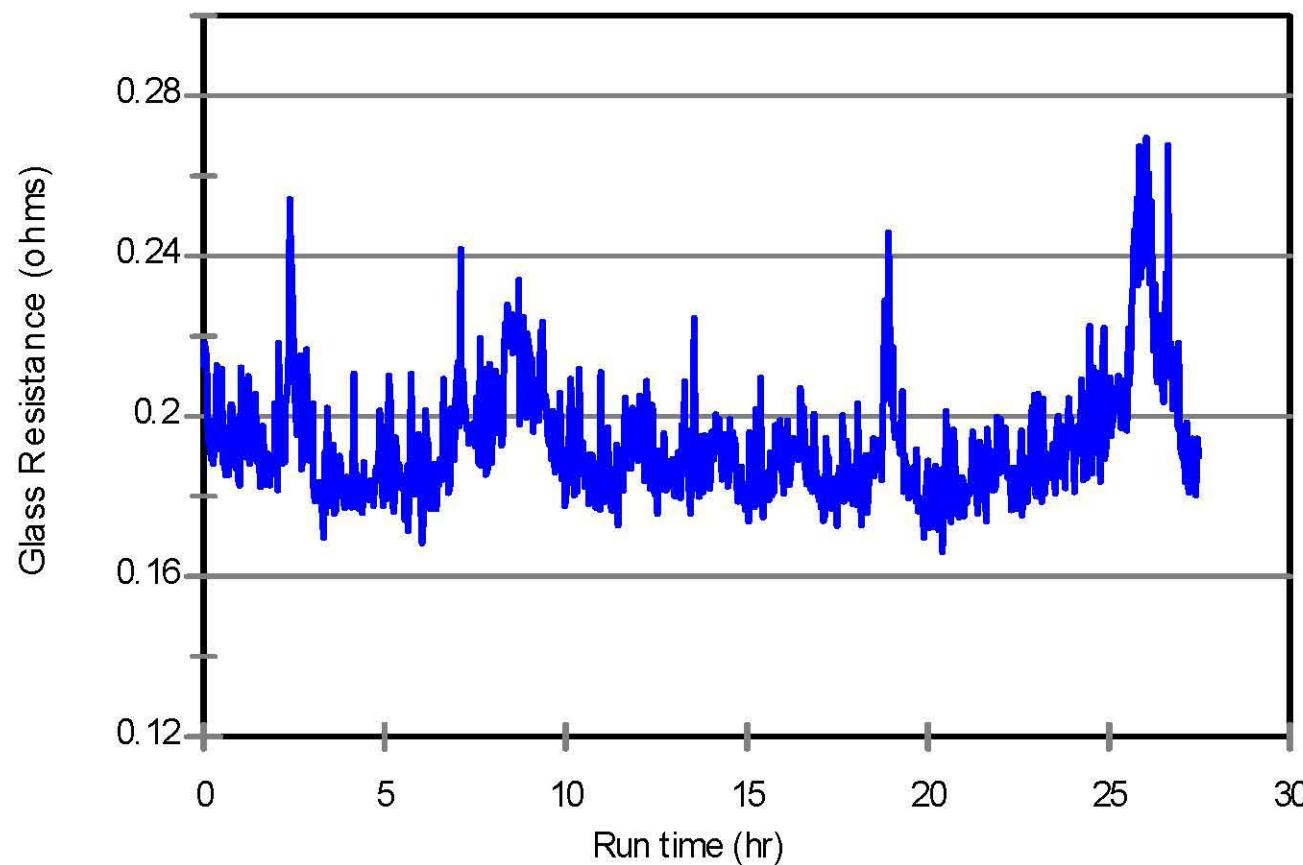
**Figure 3.4.a. Electrode power for DM10 Test 1 N.**



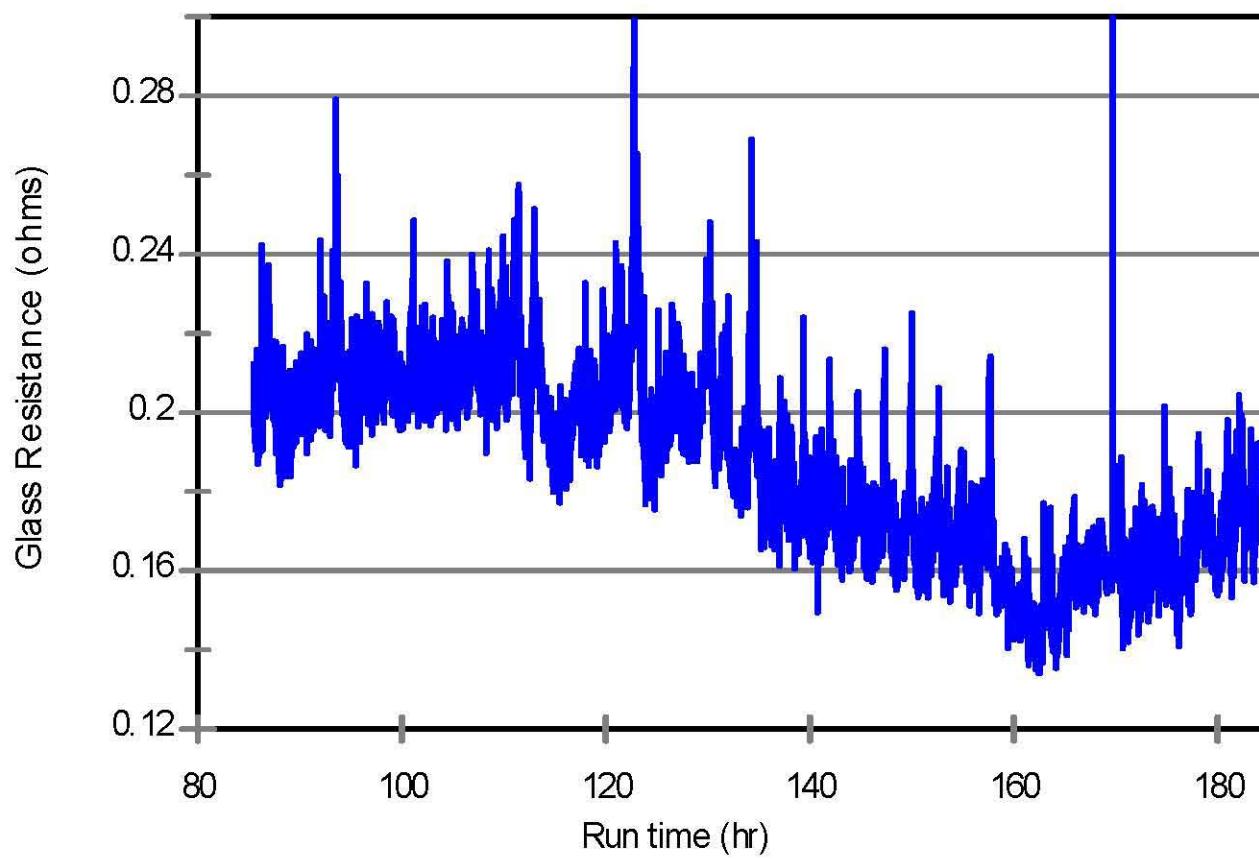
**Figure 3.4.b. Electrode power for DM10 Test 2 N, 3 N, 9 N, and 1 OX.**



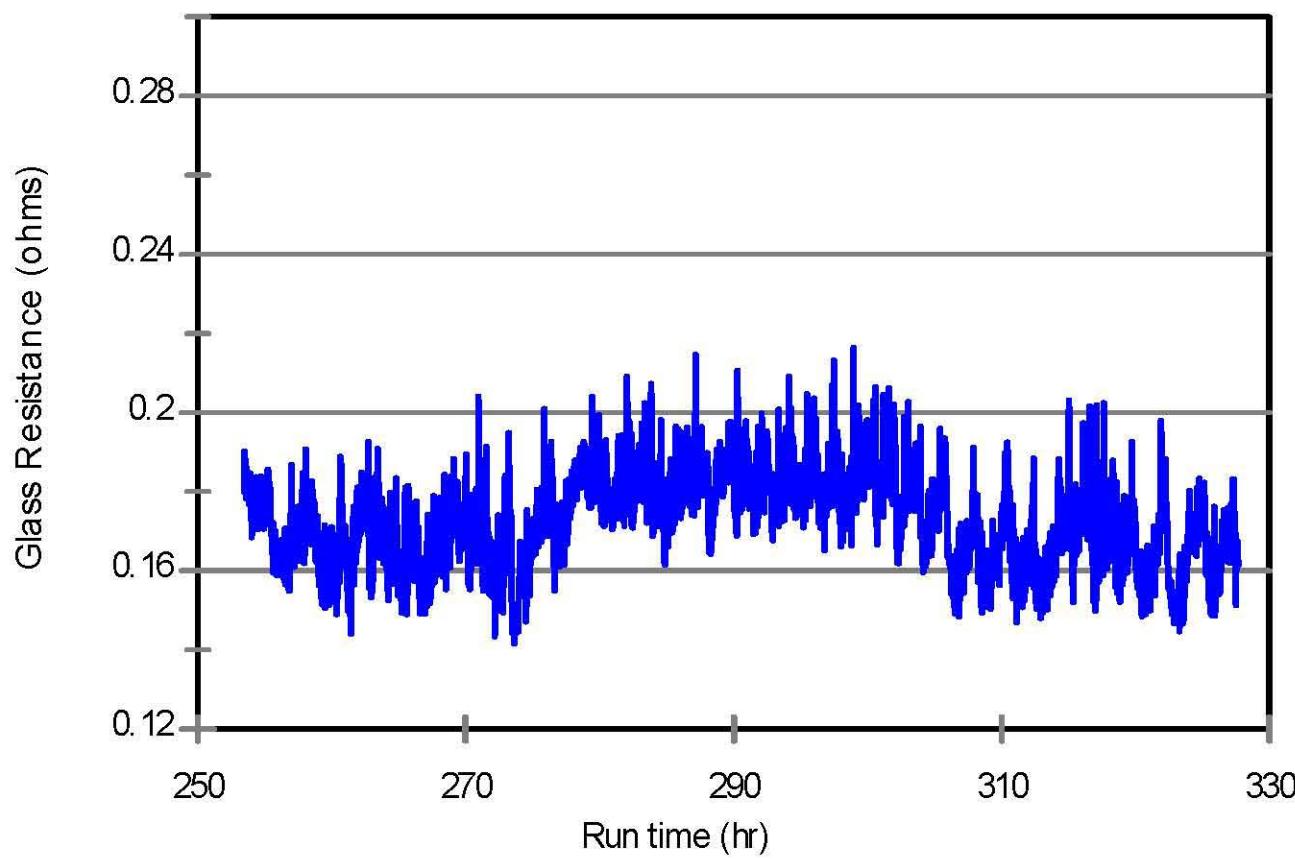
**Figure 3.4.c. Electrode power for DM10 Test 2 OX, 3 OX, and 5 OX.**



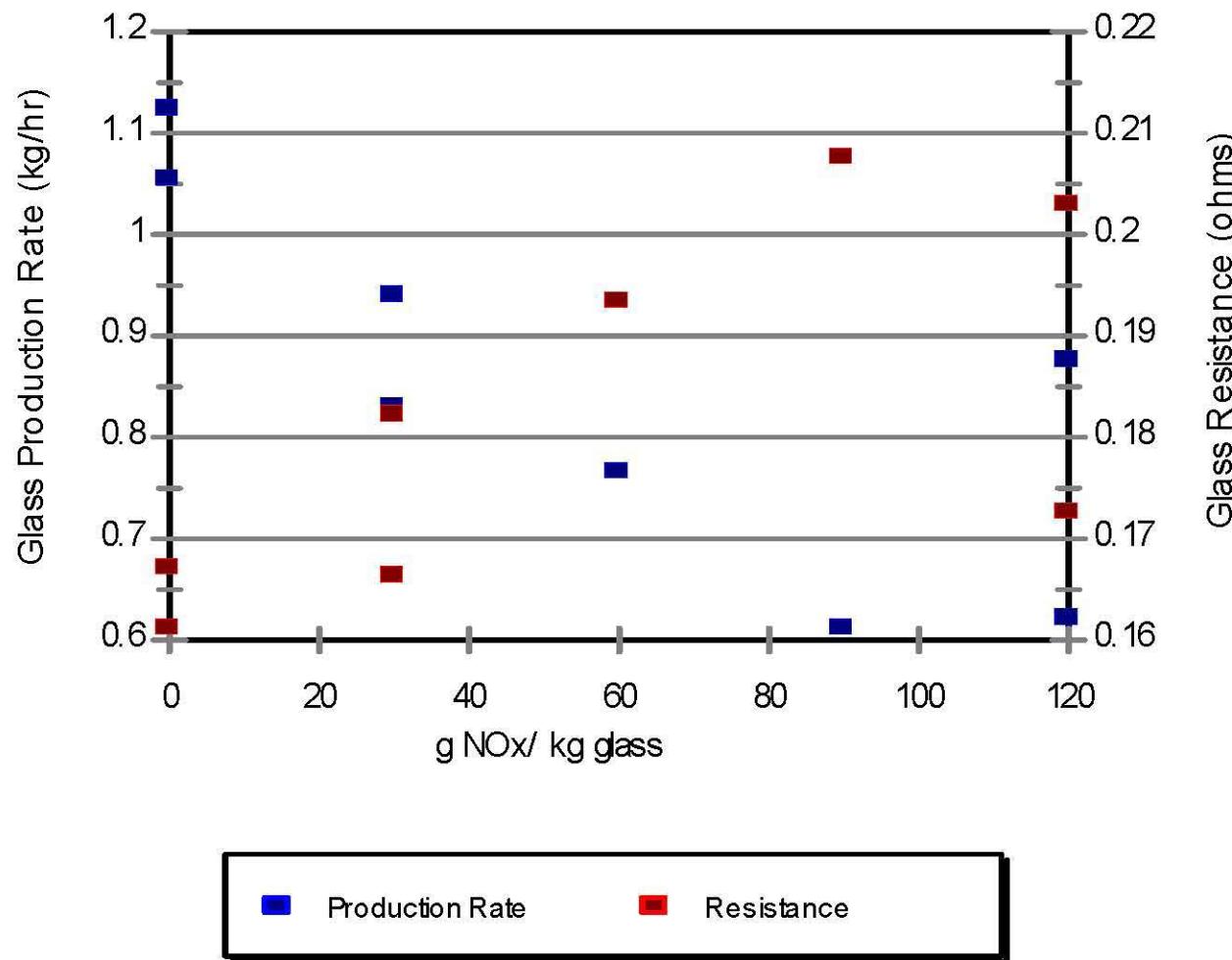
**Figure 3.5.a. Glass pool resistance during DM10 Test 1 N.**



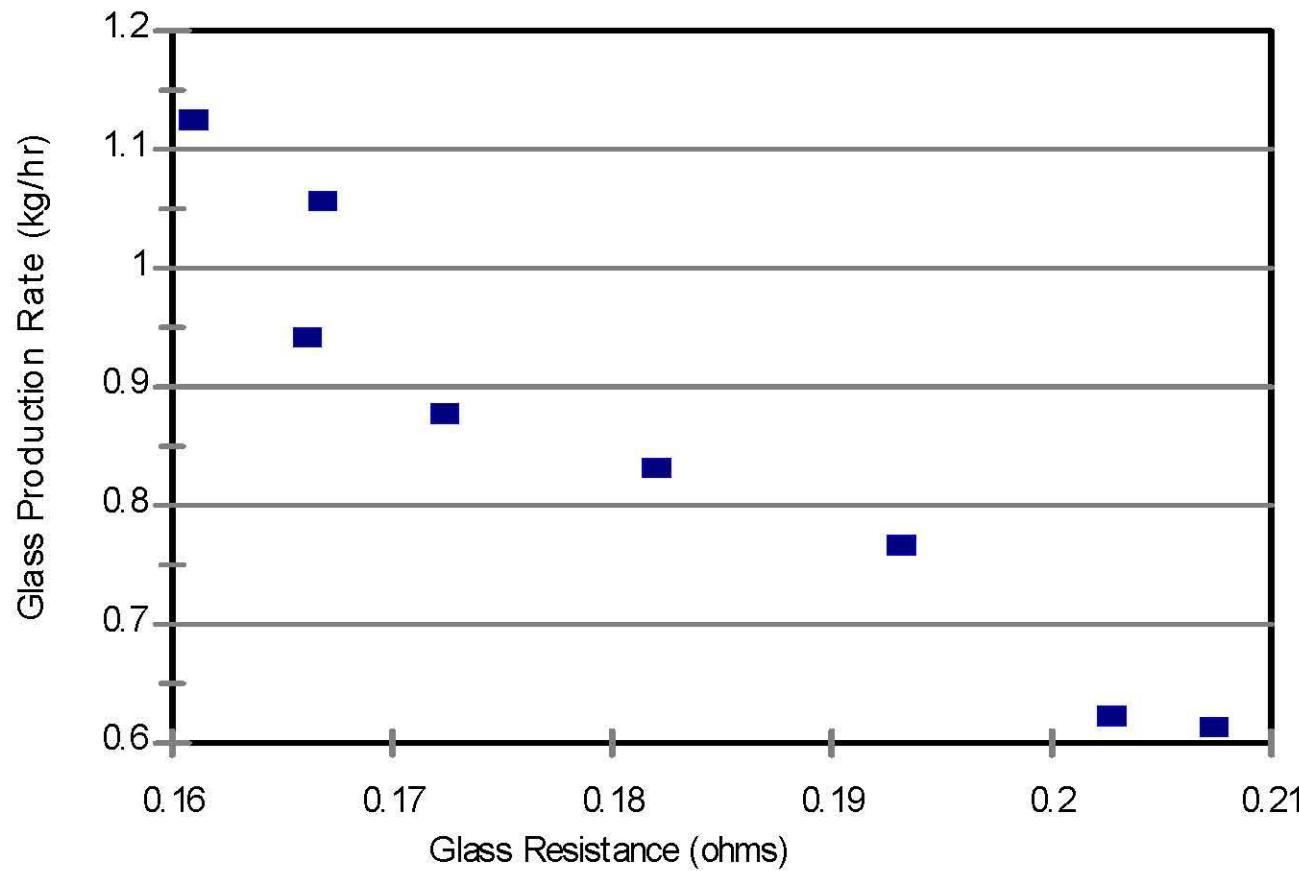
**Figure 3.5.b. Glass pool resistance during DM10 Tests 2 N, 3 N, 9 N, and 1 OX.**



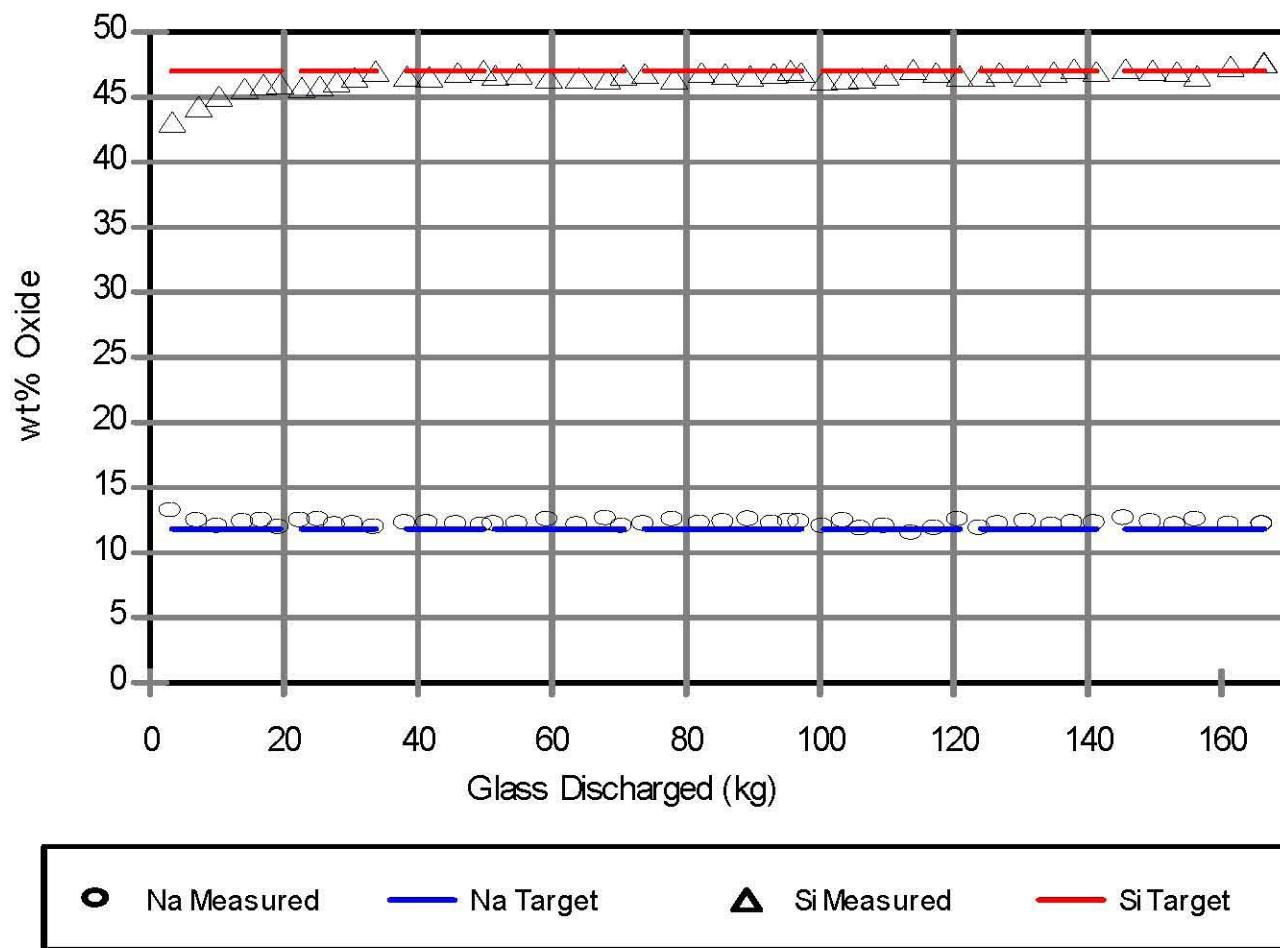
**Figure 3.5.c. Glass pool resistance during DM10 Tests 2 OX, 3 OX, and 5 OX.**



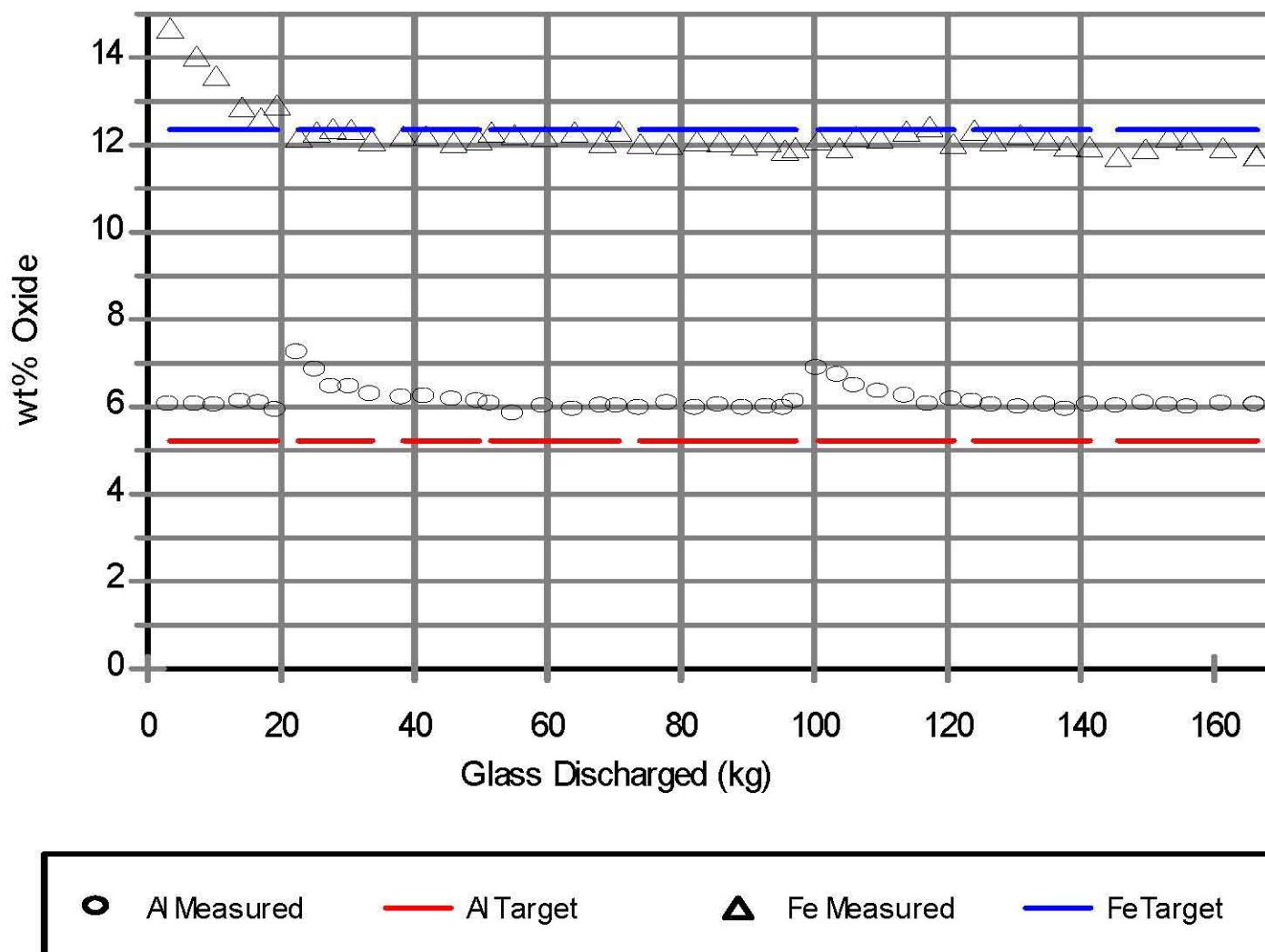
**Figure 3.6. Test Average glass production rate and resistance versus feed nitrate content.**



**Figure 3.7. Test Average glass production rate versus glass pool resistance.**



**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.**

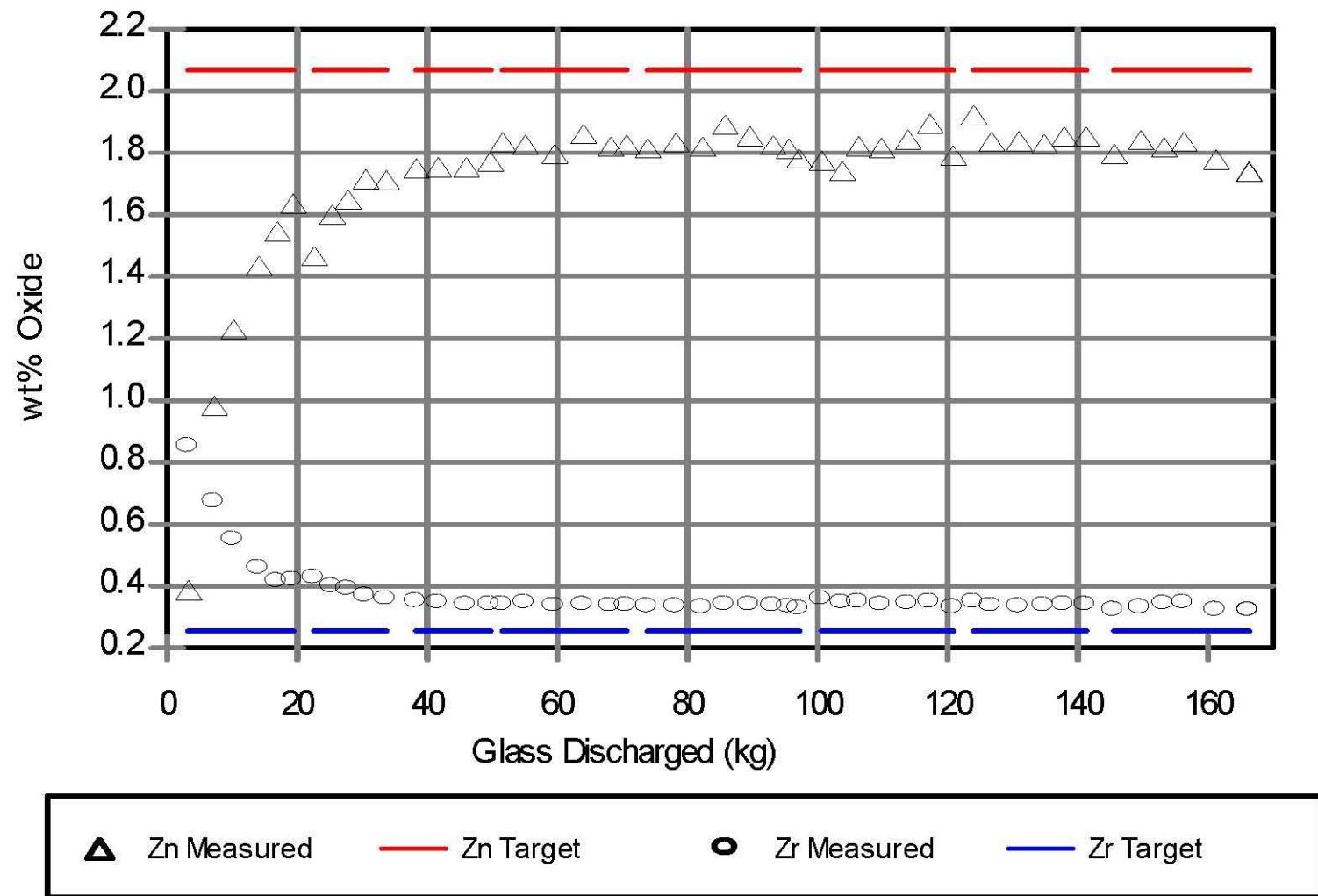
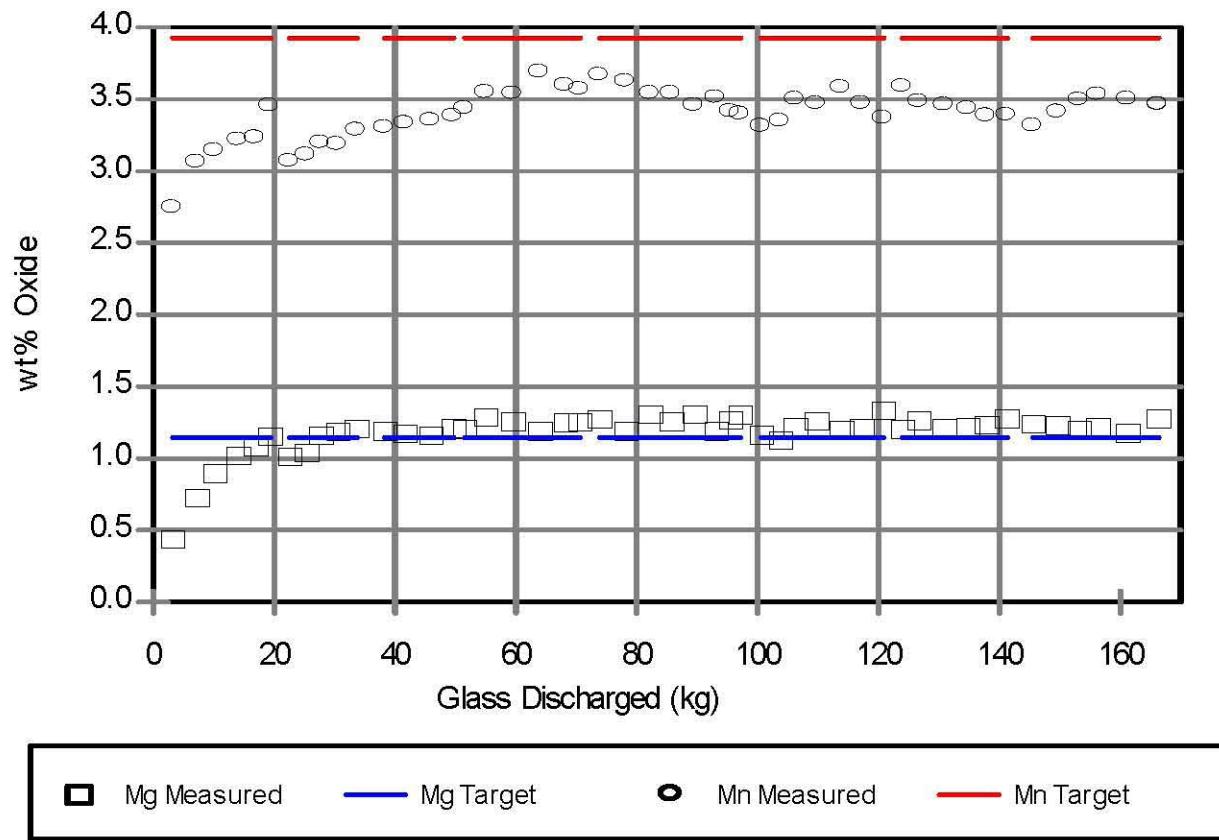


Figure 4.3. DM10 product and target glass zinc and zirconium oxide concentrations determined by XRF.



**Figure 4.4. DM10 product and target glass magnesium and manganese oxide concentrations determined by XRF.**

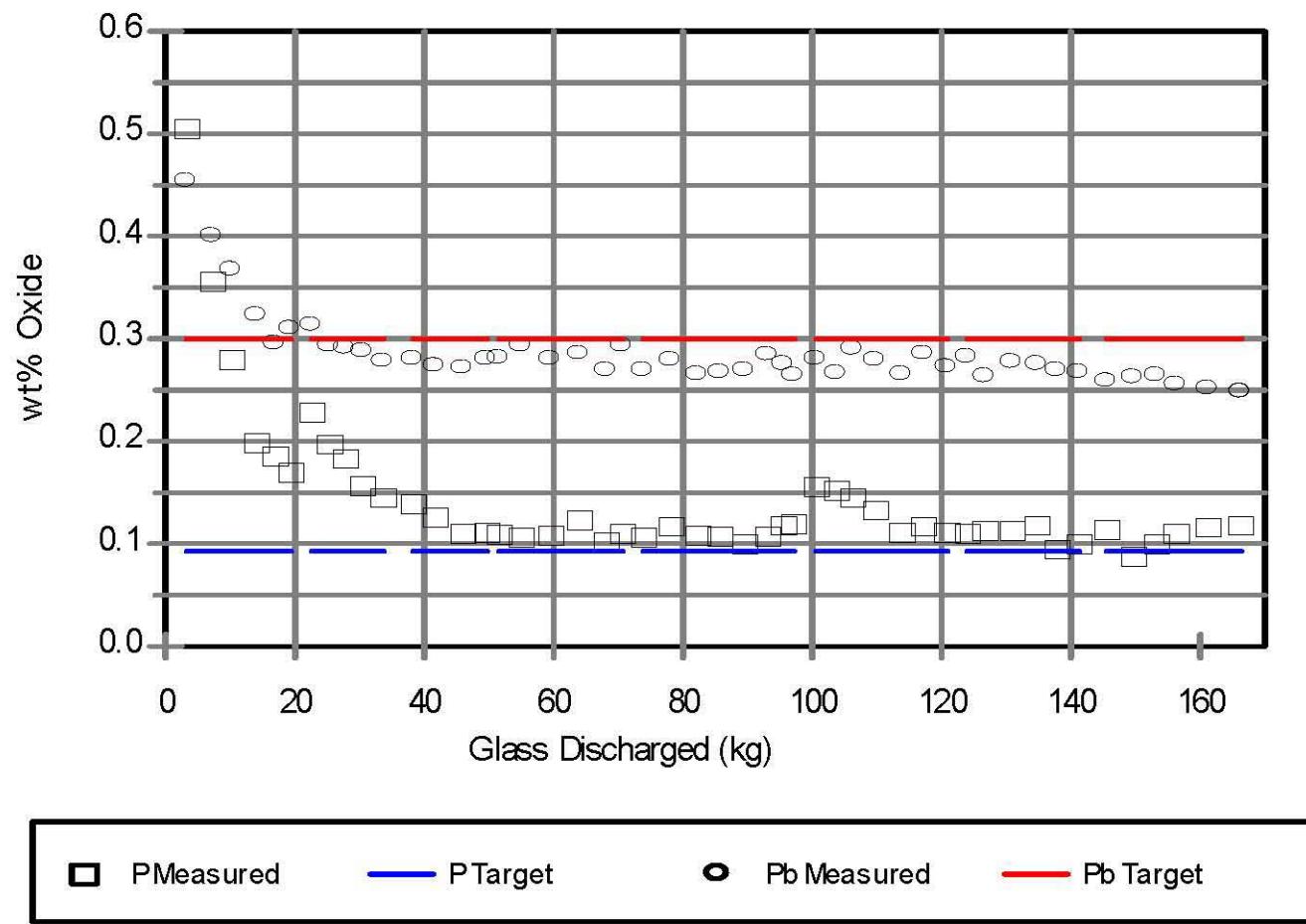


Figure 4.5. DM10 product and target glass lead and phosphorous oxide concentrations determined by XRF.

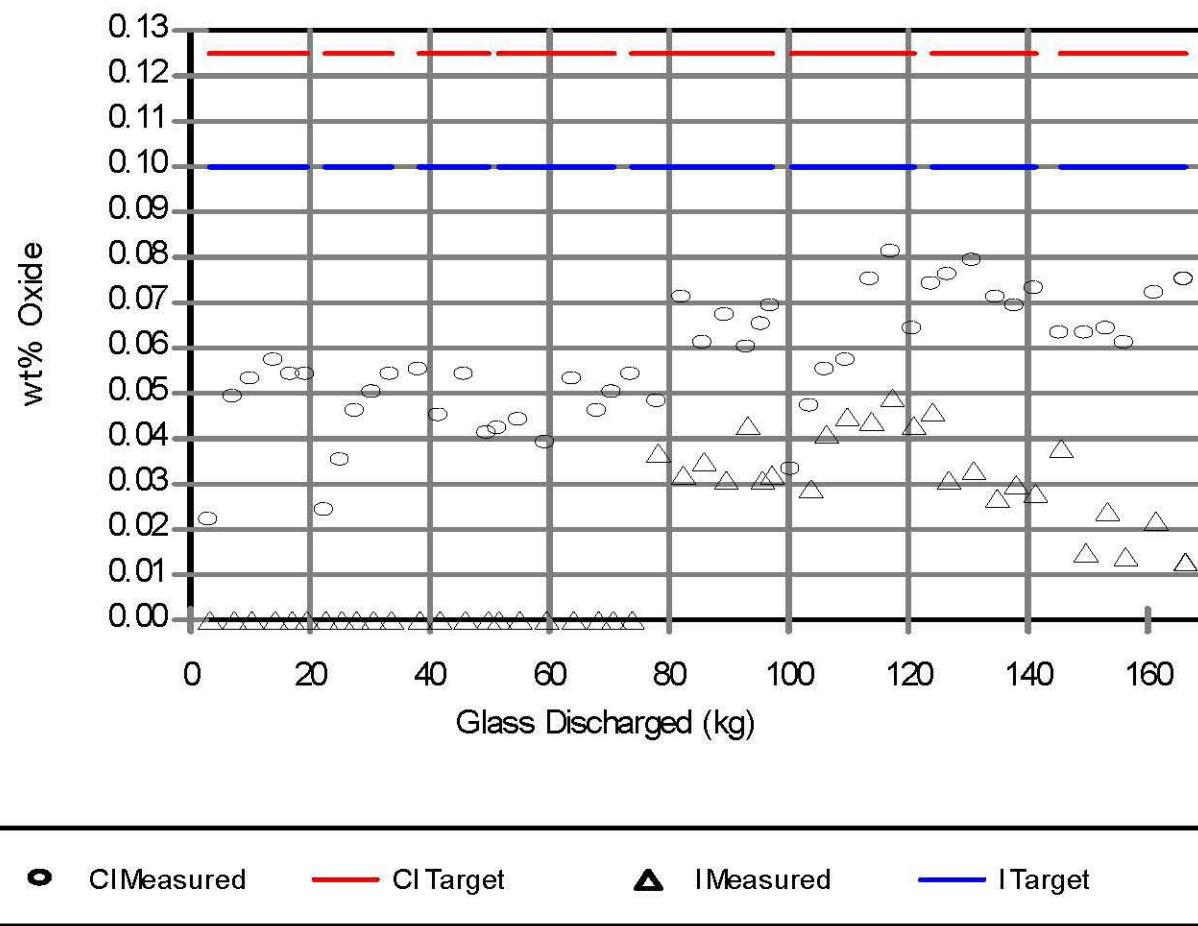
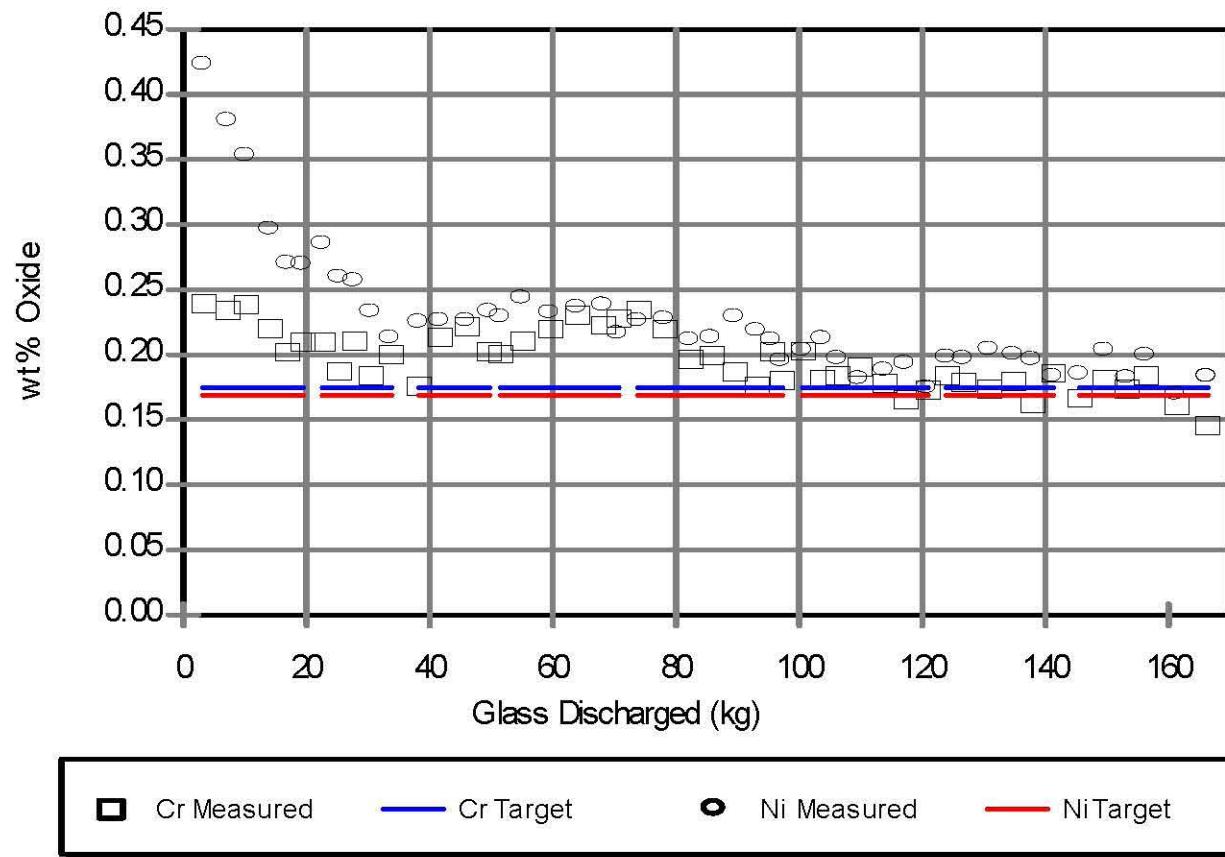


Figure 4.6. DM10 product and target glass chlorine and iodine concentrations determined by XRF.



**Figure 4.7. DM10 product and target glass chromium and nickel oxide concentrations determined by XRF.**

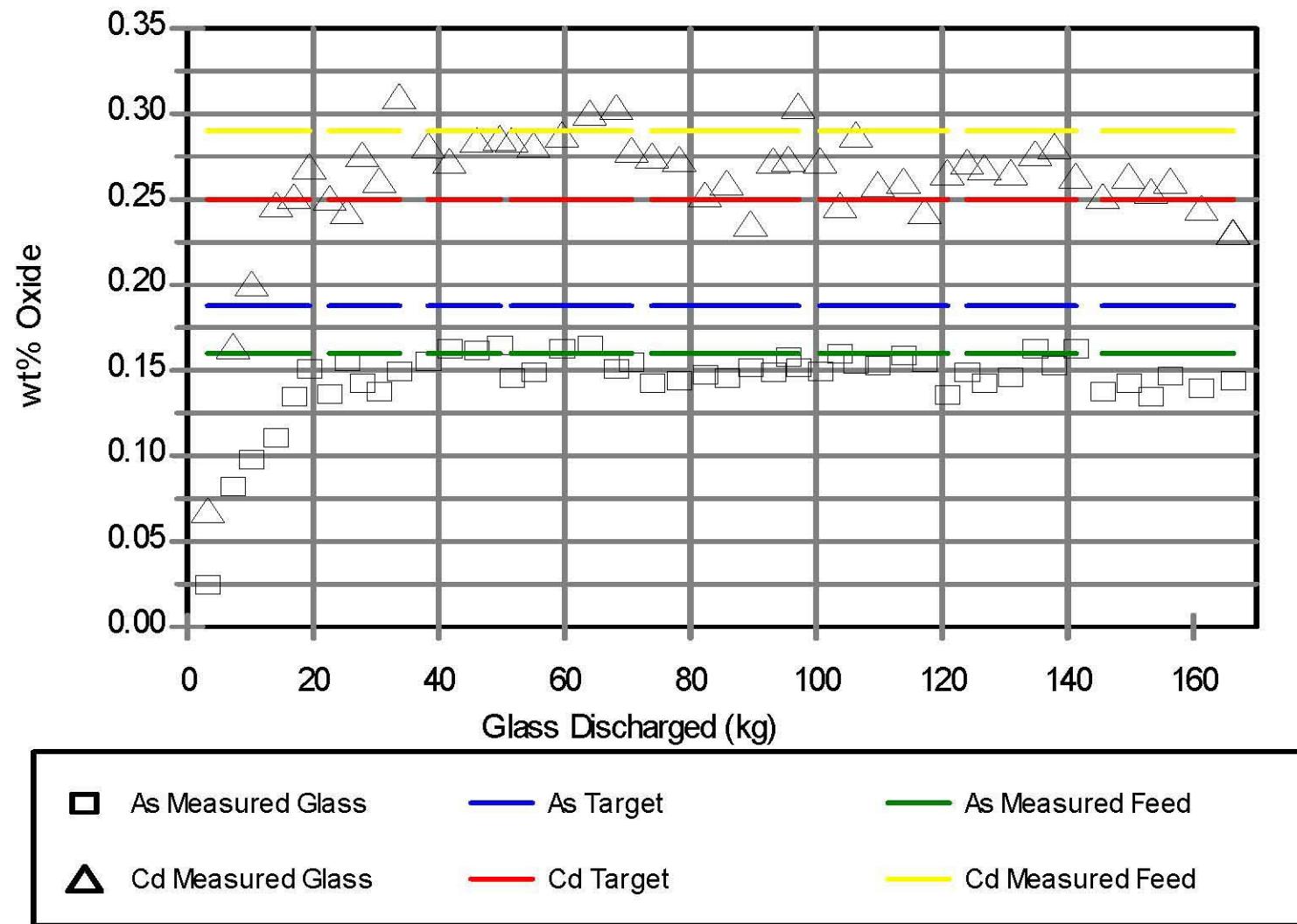
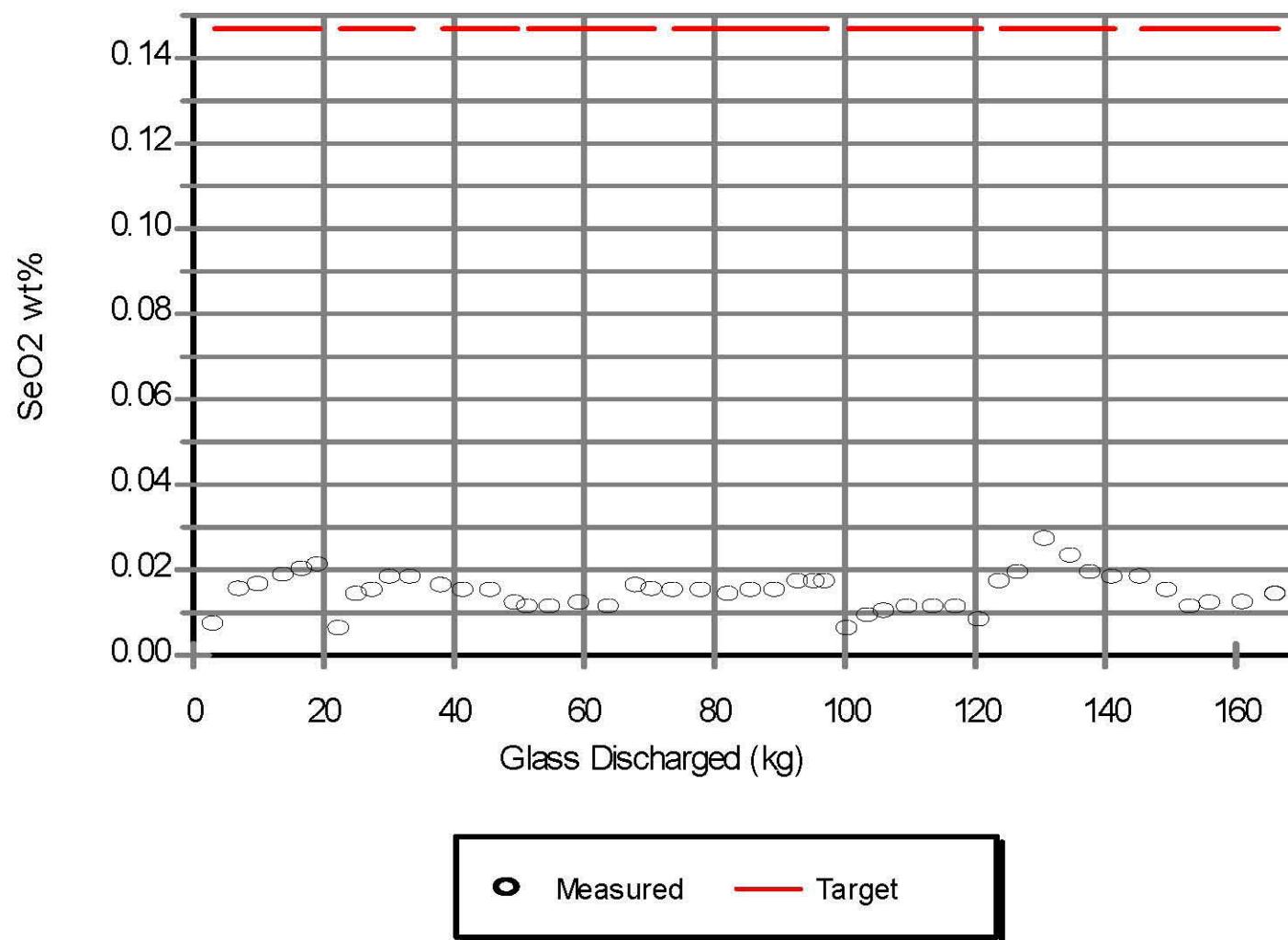


Figure 4.8. DM10 product and target glass arsenic and cadmium oxide concentrations determined by XRF.



**Figure 4.9. DM10 product and target glass selenium oxide concentrations determined by XRF.**

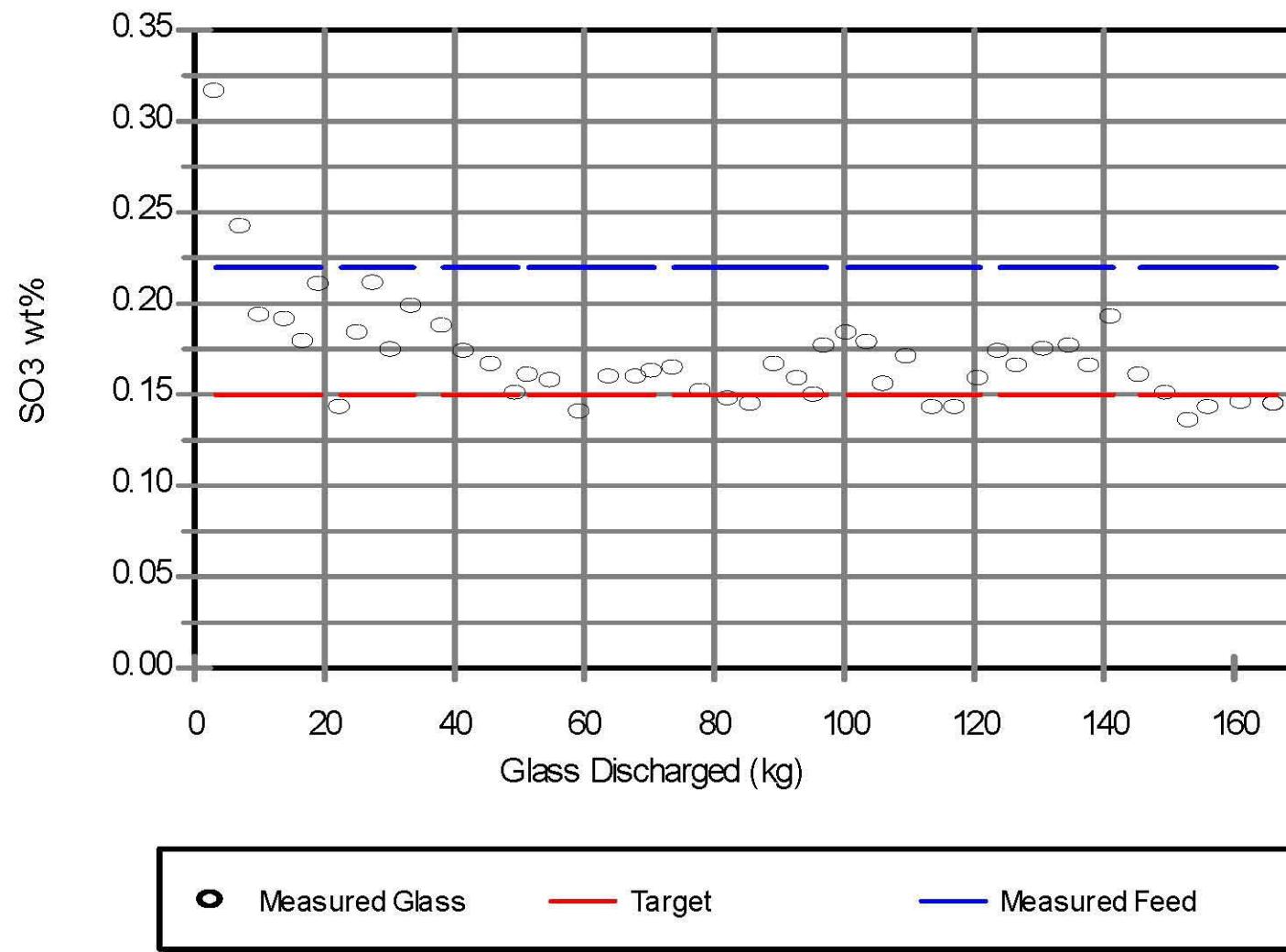
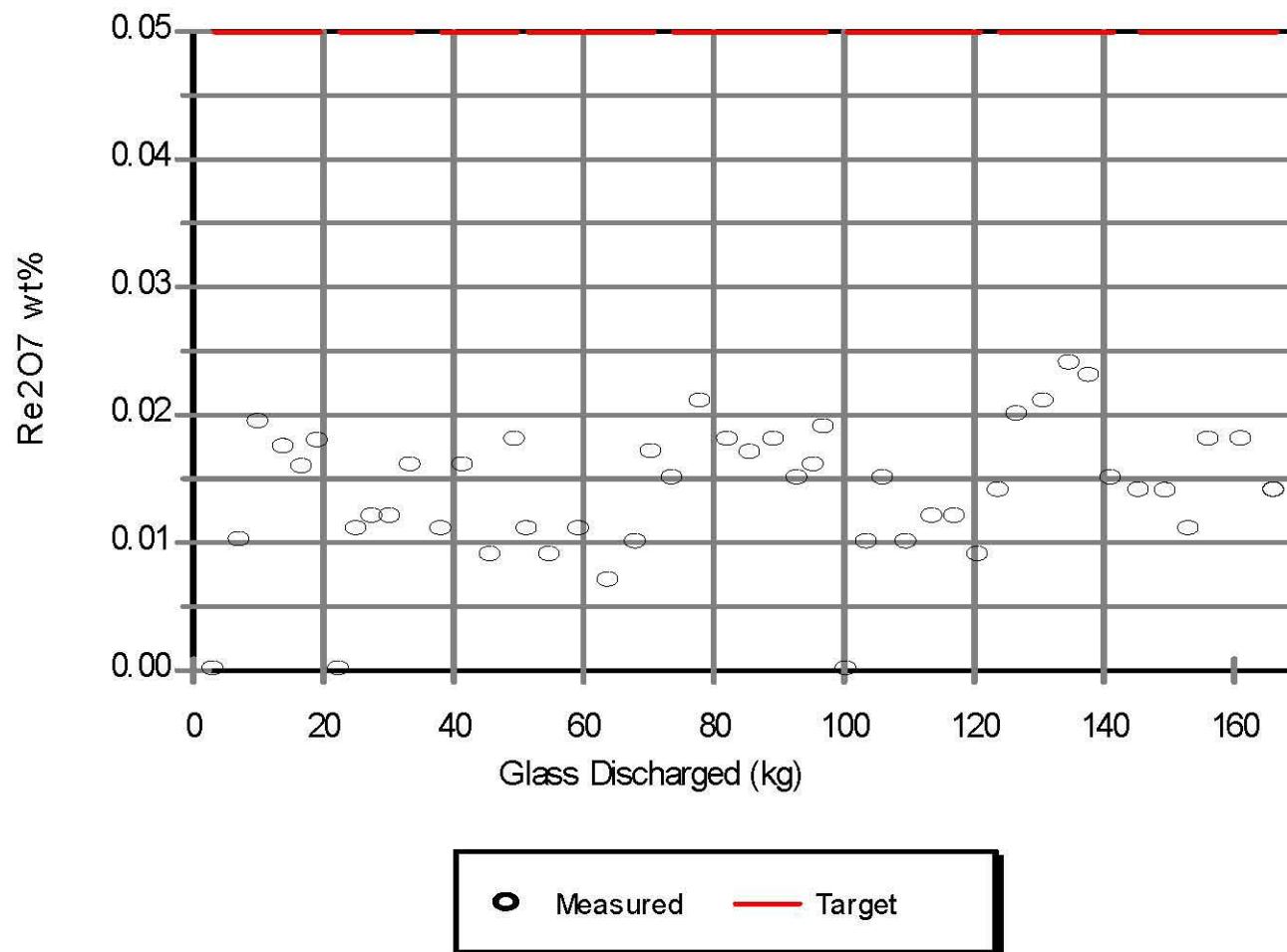
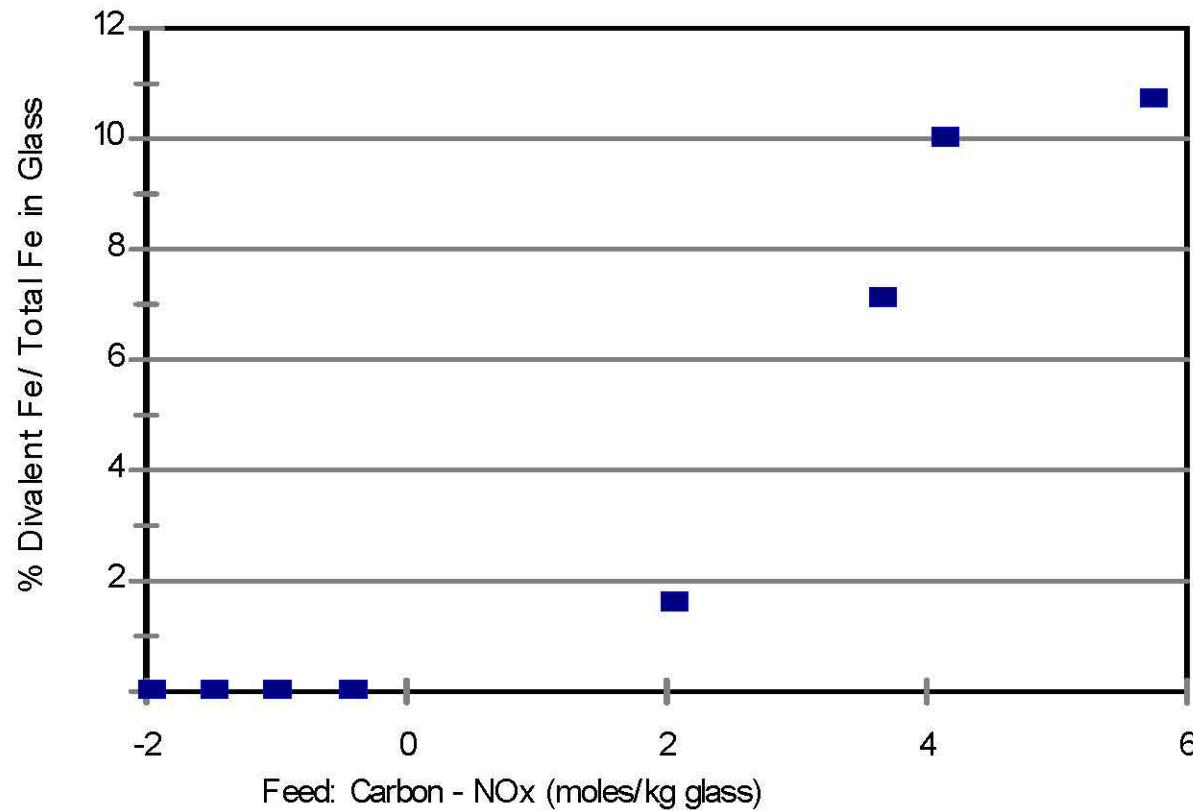


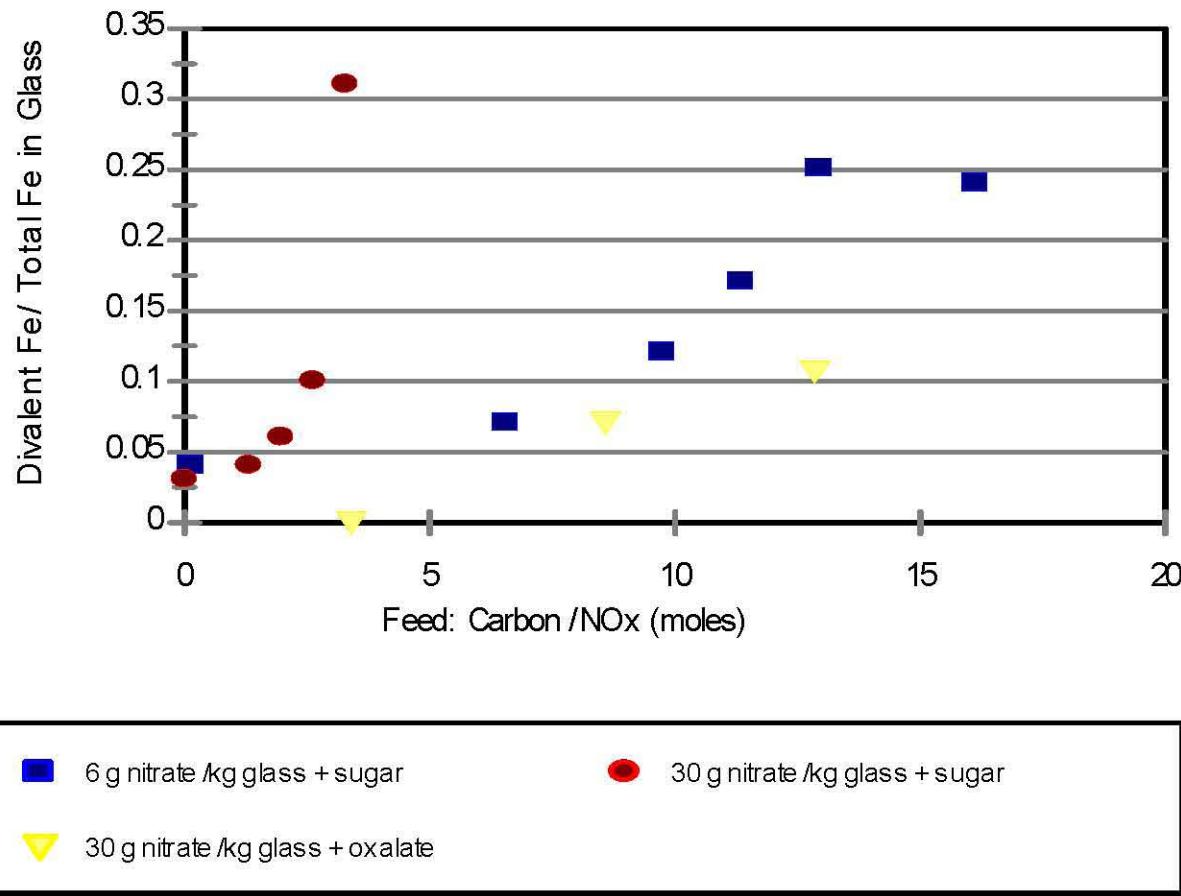
Figure 4.10. DM10 product glass, target glass, and feed sulfur oxide concentrations determined by XRF.



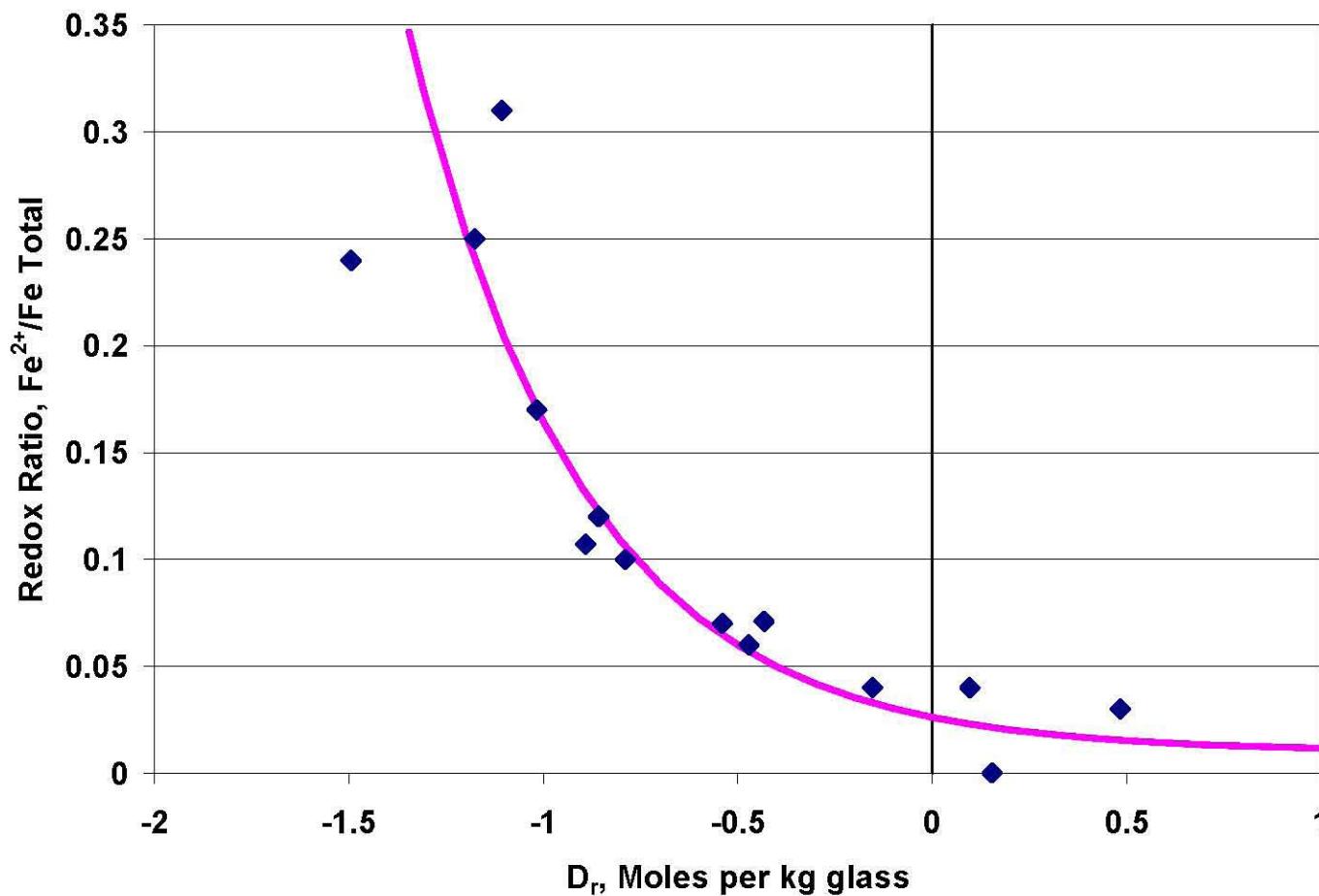
**Figure 4.11. DM10 product and target glass rhenium oxide concentrations determined by XRF.**



**Figure 4.12. Measured iron oxidation state in glass pool samples versus the difference in carbon and NOx contents in the feed.**



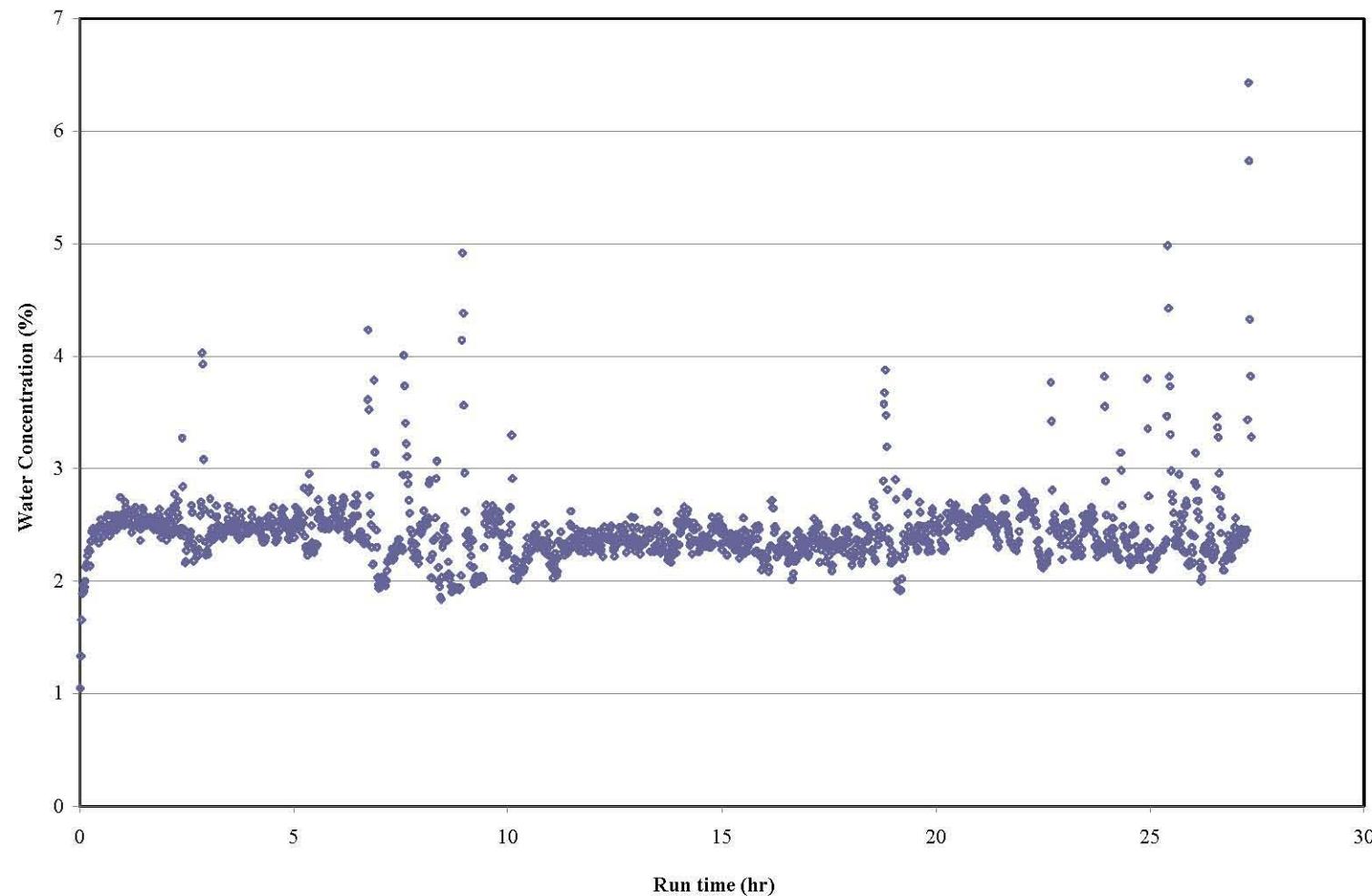
**Figure 4.13. Measured iron oxidation state in glass versus feed carbon/ nitrate mole ratio from this work and from previous tests [28, 30].**



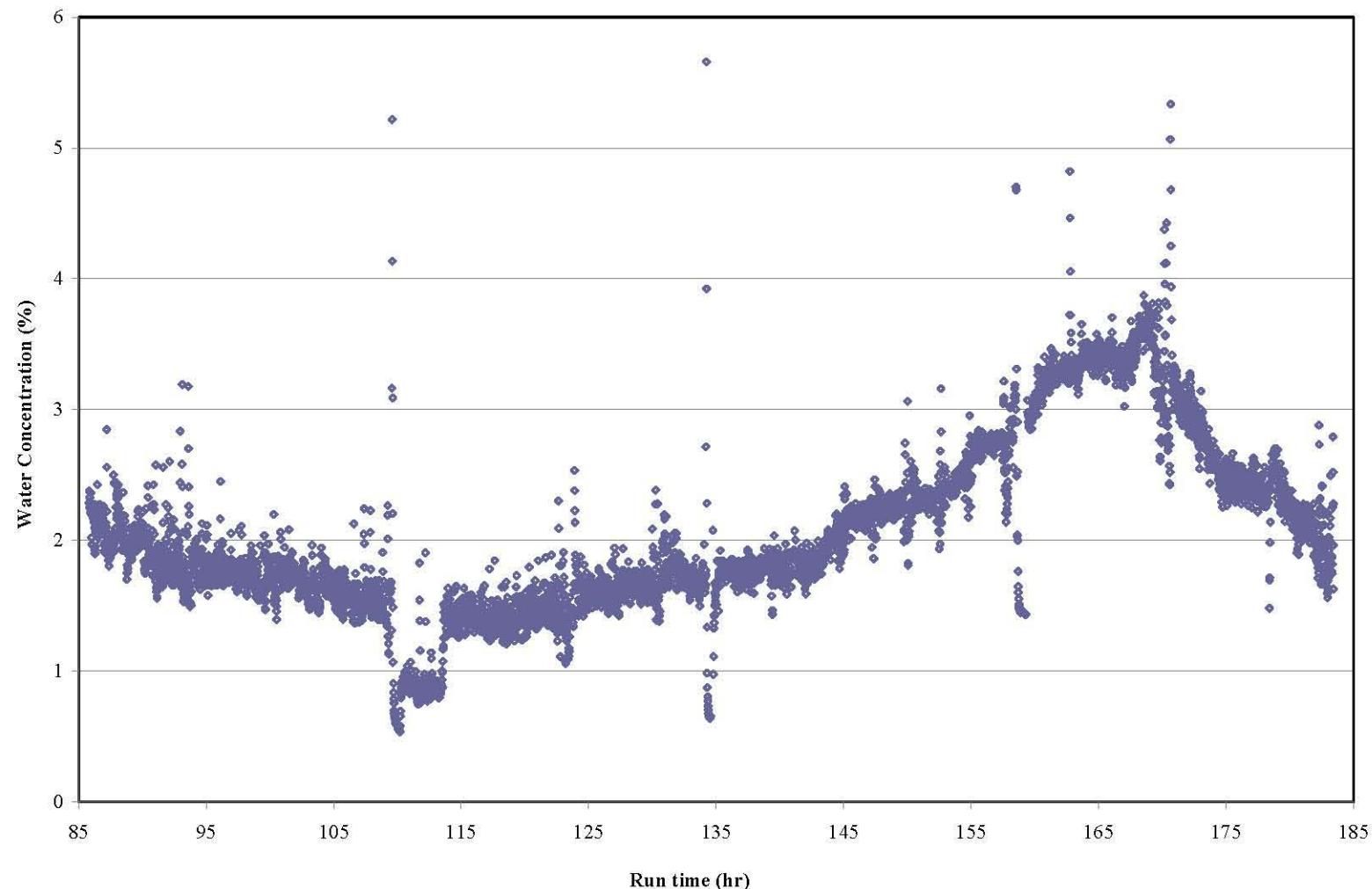
**Figure 4.14. Measured iron oxidation state in glass from this work and from previous tests [28, 30] (data from Figure 4.13) versus the variable  $D_r$  defined in Section 4.2.1.**



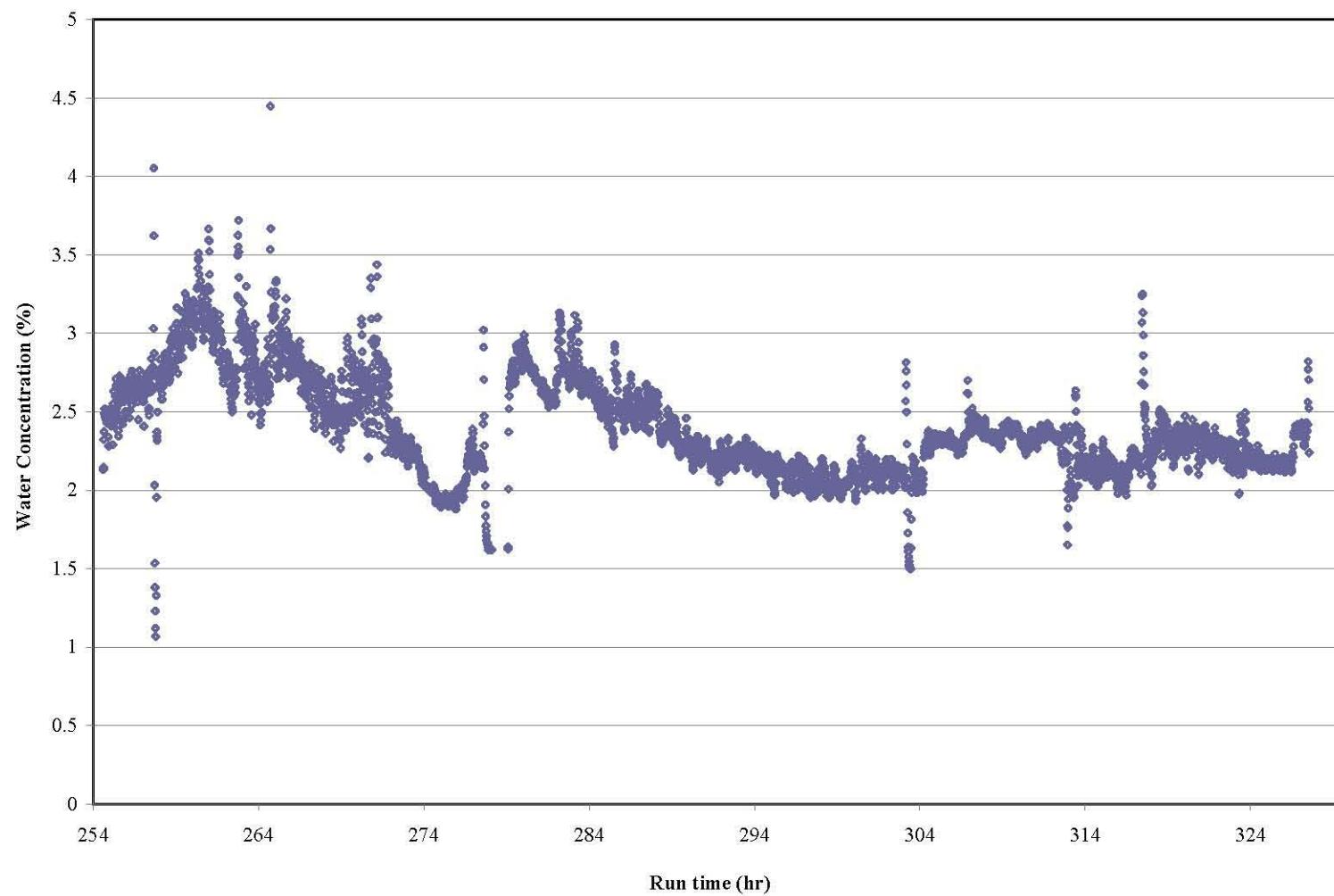
**Figure 4.15. Secondary phase with metallic appearance observed on top of suction sample taken after Test 5 OX.**



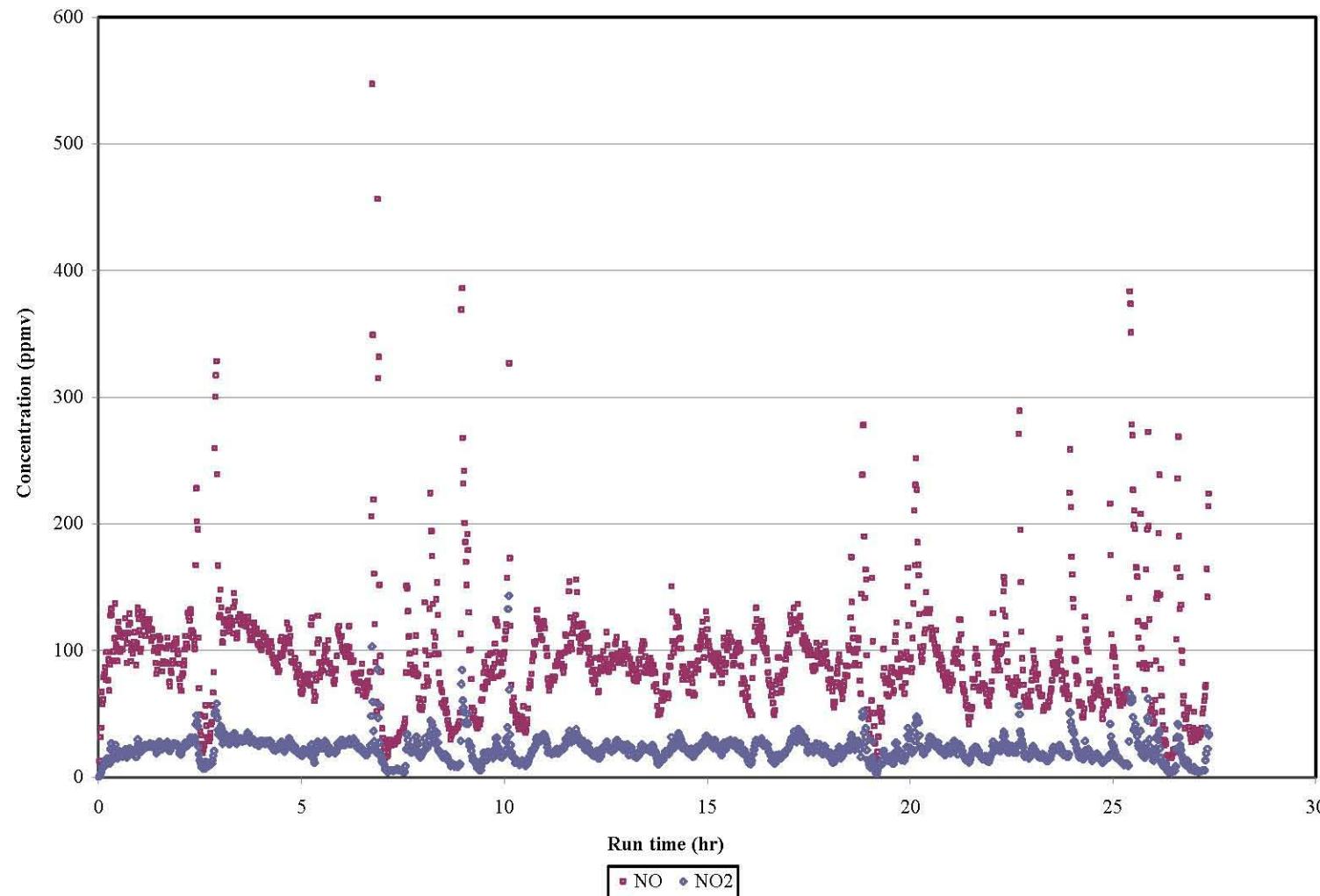
**Figure 5.1.a. Water measured in off-gas system exhaust, Test 1 N.**



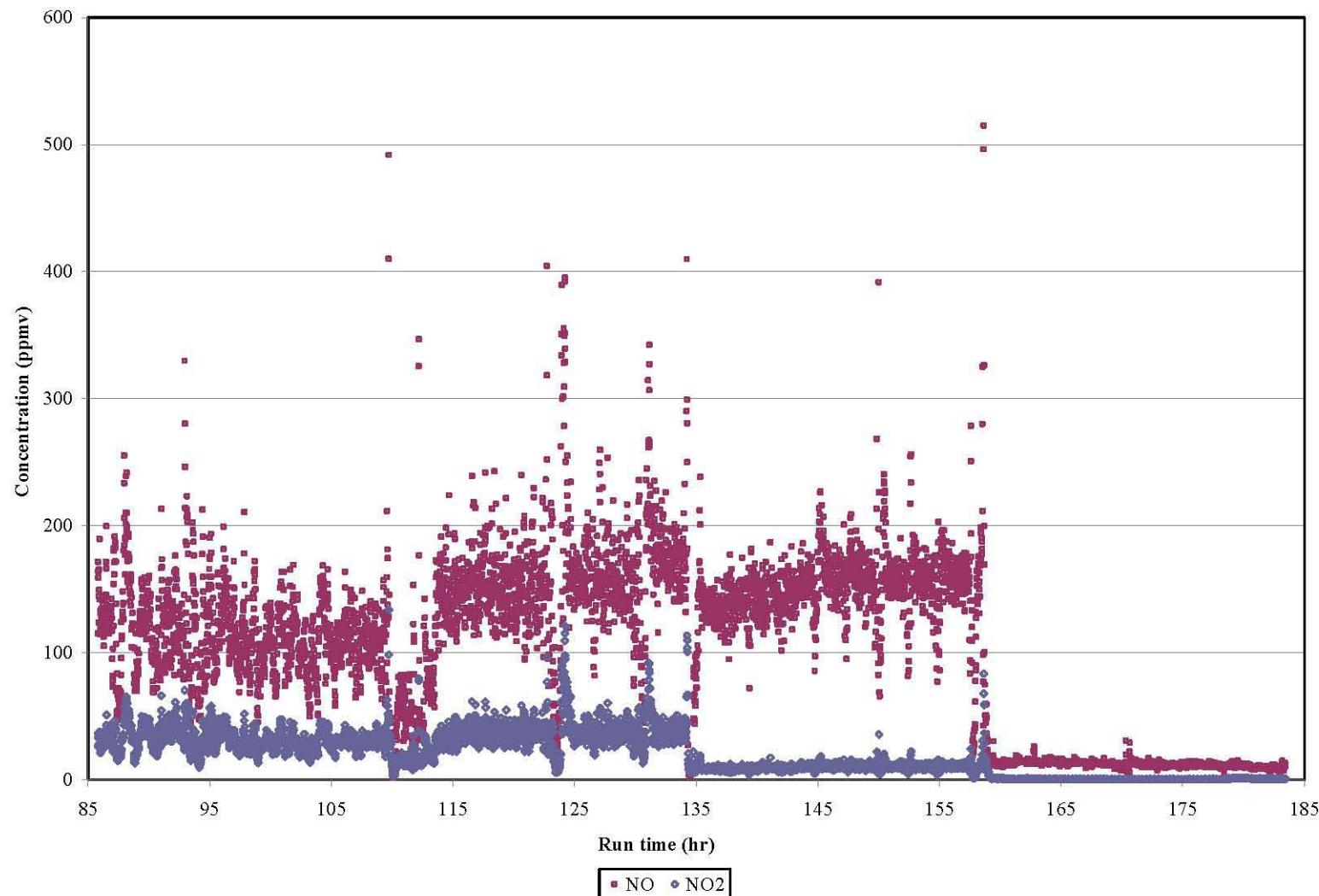
**Figure 5.1.b. Water measured in off-gas system exhaust, Tests 2 N, 3 N, 9 N, and 1 OX.**



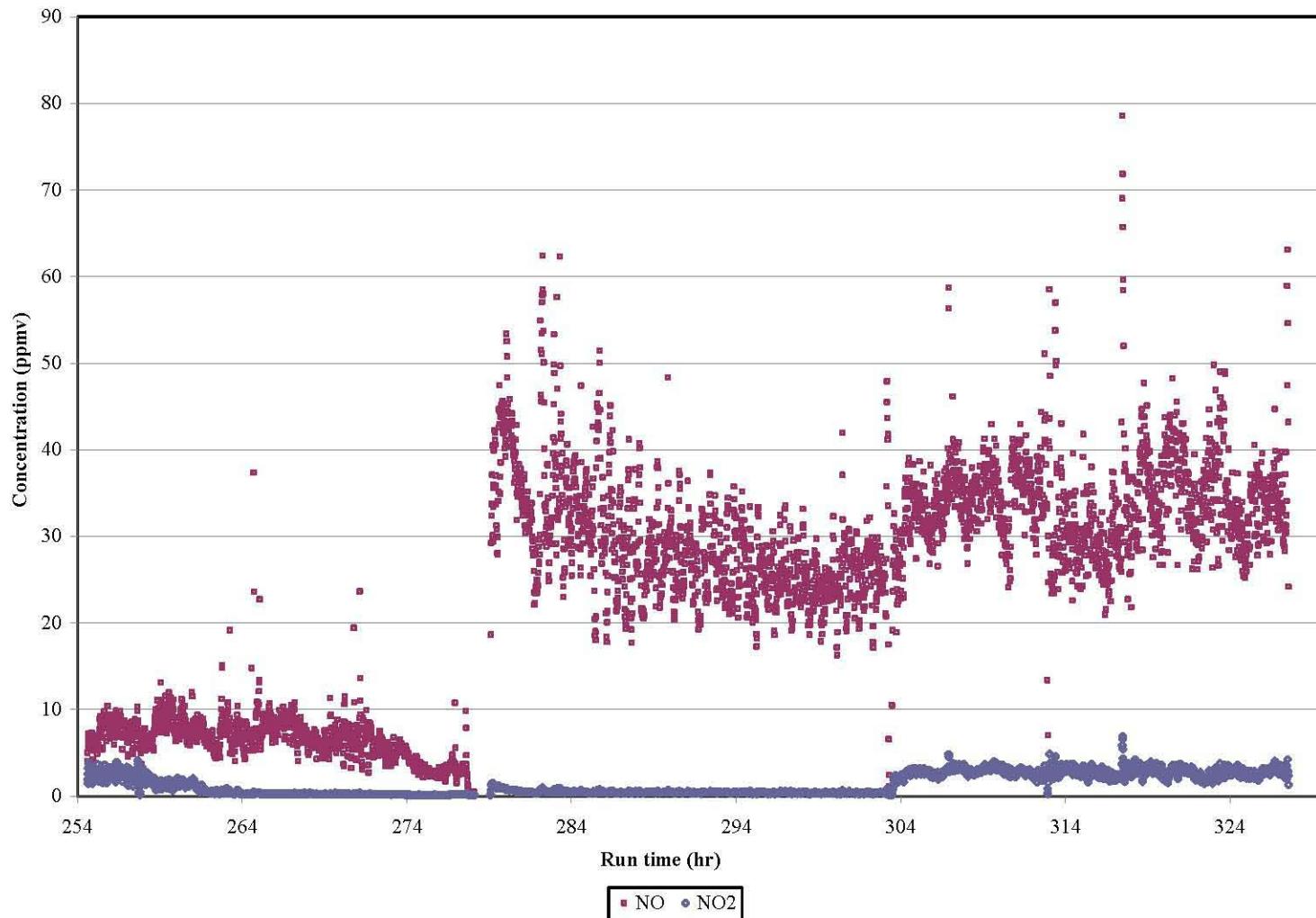
**Figure 5.1.c. Water measured in off-gas system exhaust, Tests 2 OX, 3 OX, and 5 OX.**



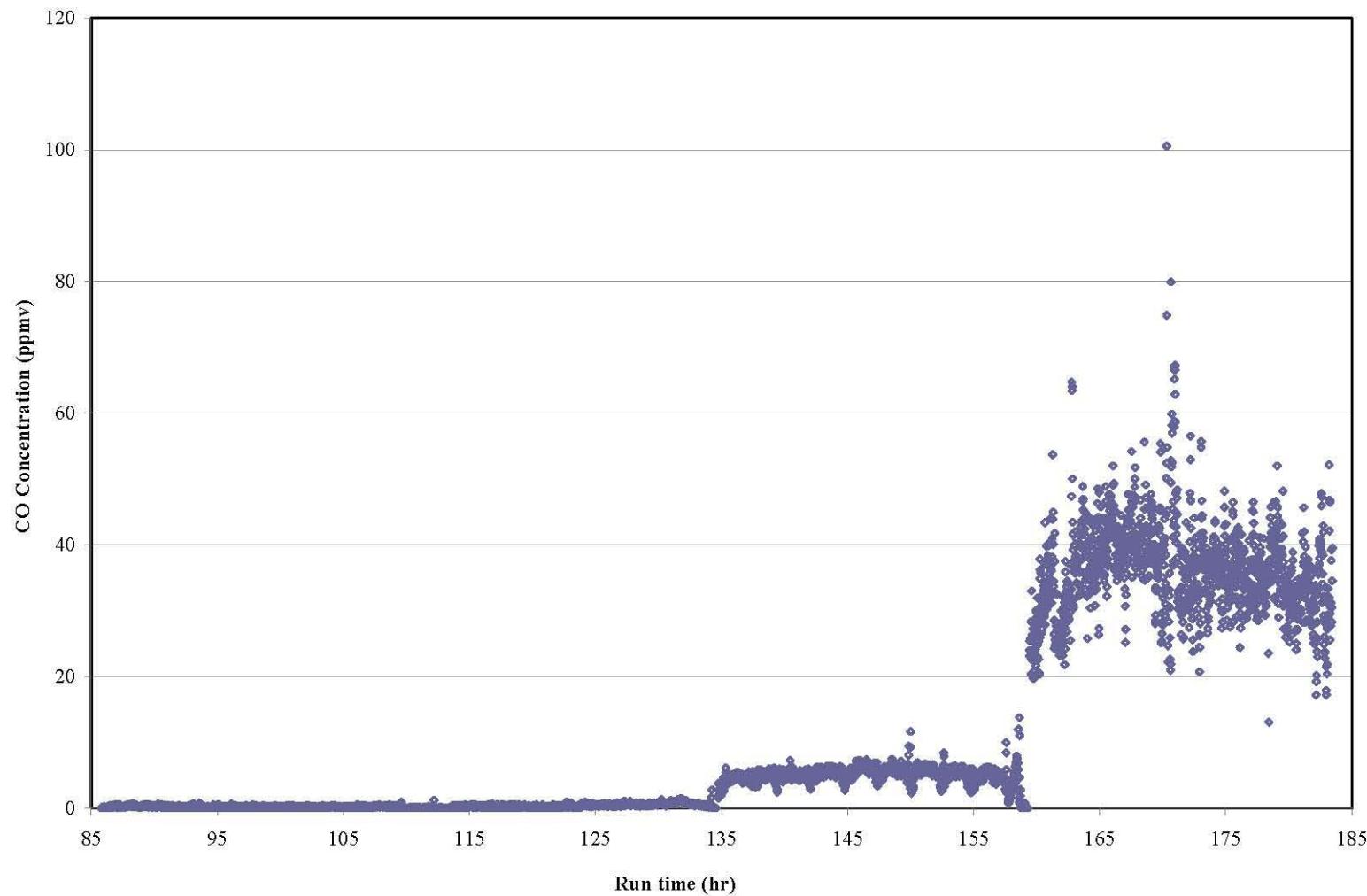
**Figure 5.2.a. Nitrogen oxides measured in off-gas system exhaust, Test 1 N.**



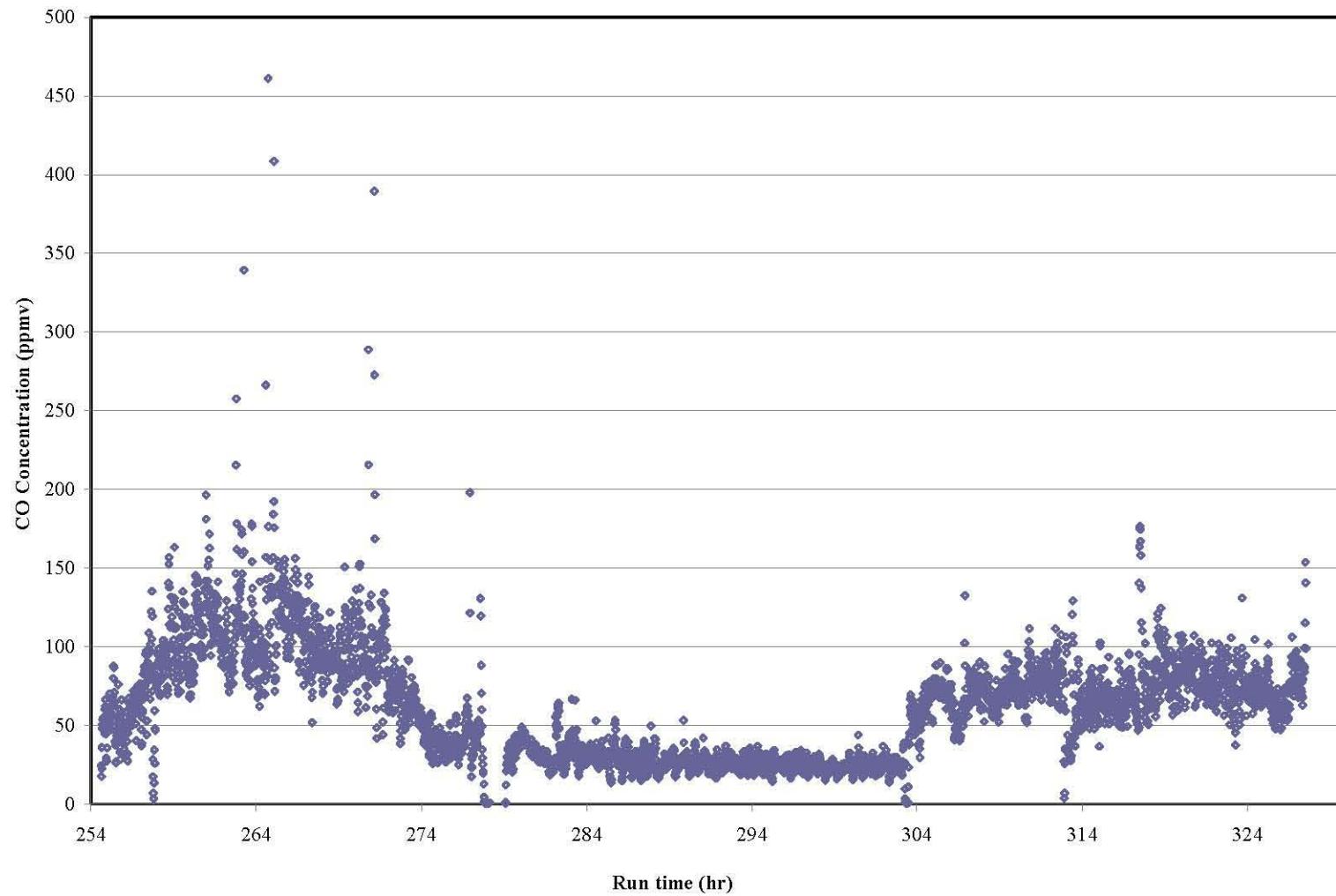
**Figure 5.2.b. Nitrogen oxides measured in off-gas system exhaust, Tests 2 N, 3 N, 9 N, and 1 OX.**



**Figure 5.2.c. Nitrogen oxides measured in off-gas system exhaust, Tests 2 OX, 3 OX, and 5 OX.**



**Figure 5.3.a. Carbon monoxide measured in off-gas system exhaust, Tests 2 N, 3 N, 9 N, and 1 OX.**



**Figure 5.3.b. Carbon monoxide measured in off-gas system exhaust, Tests 2 OX, 3 OX, and 5 OX.**

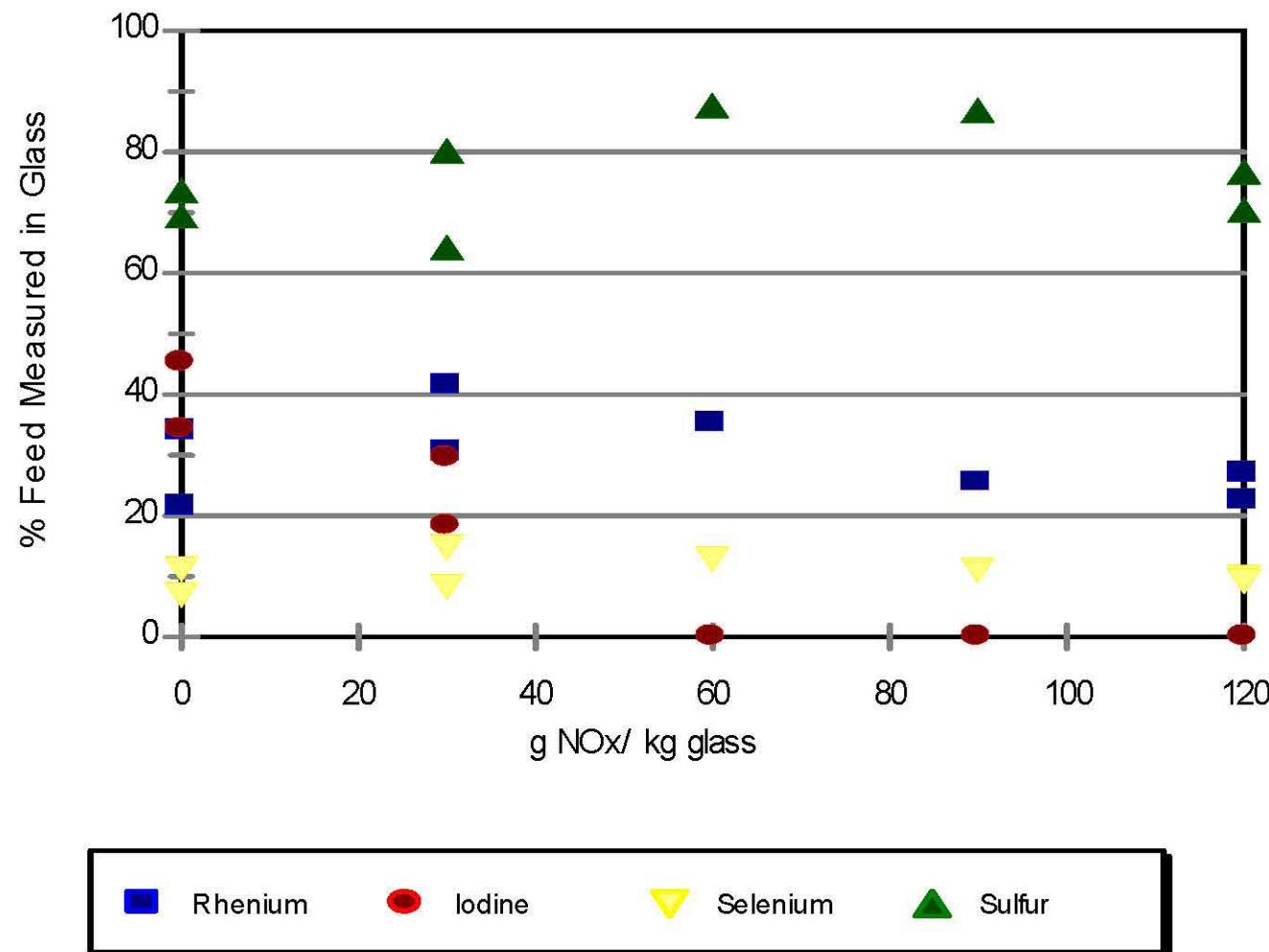
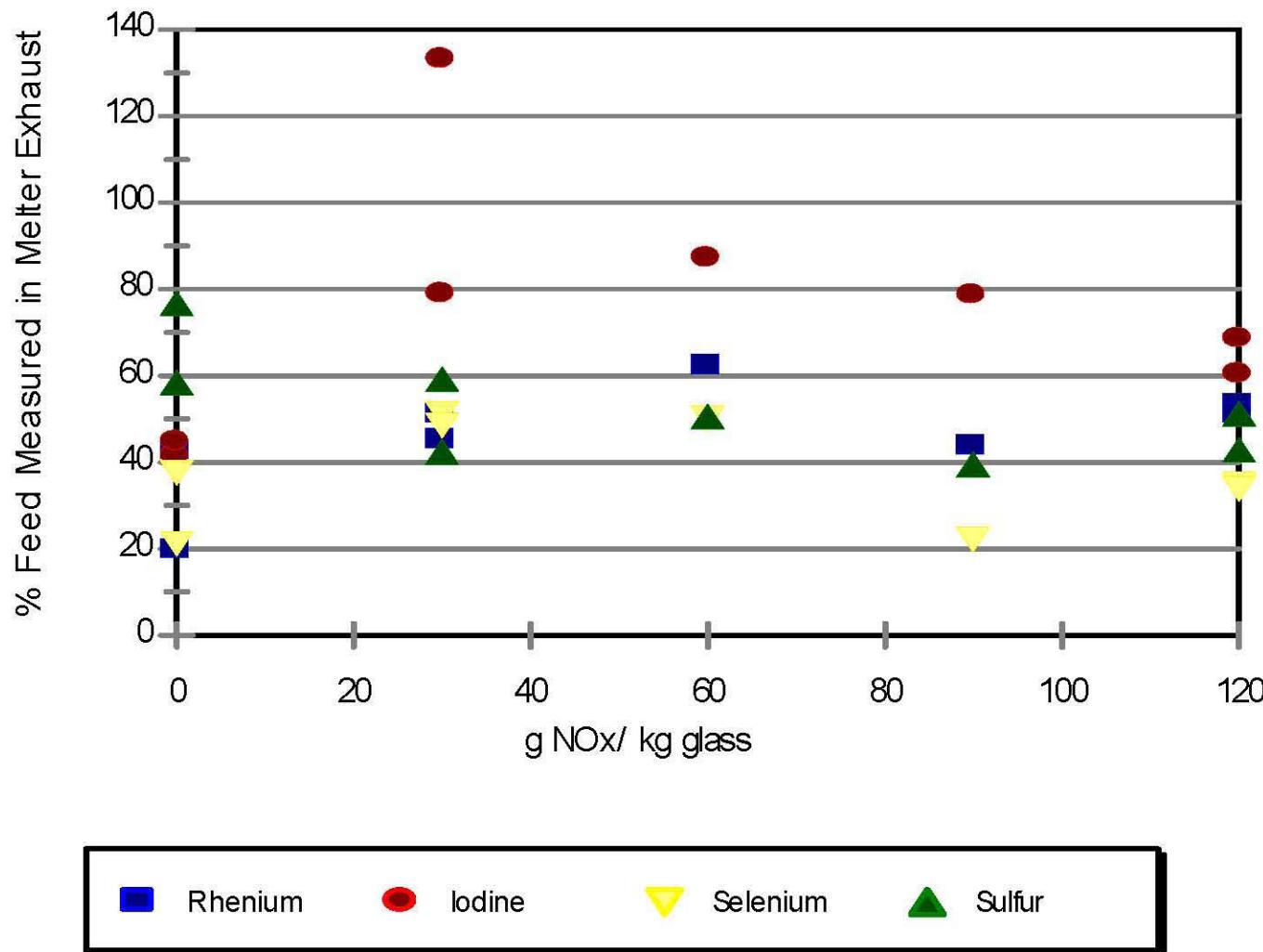


Figure 6.1. Volatile retention in glass as a function of feed nitrate concentration.



**Figure 6.2. Volatiles in melter exhaust as a function of feed nitrate concentration.**

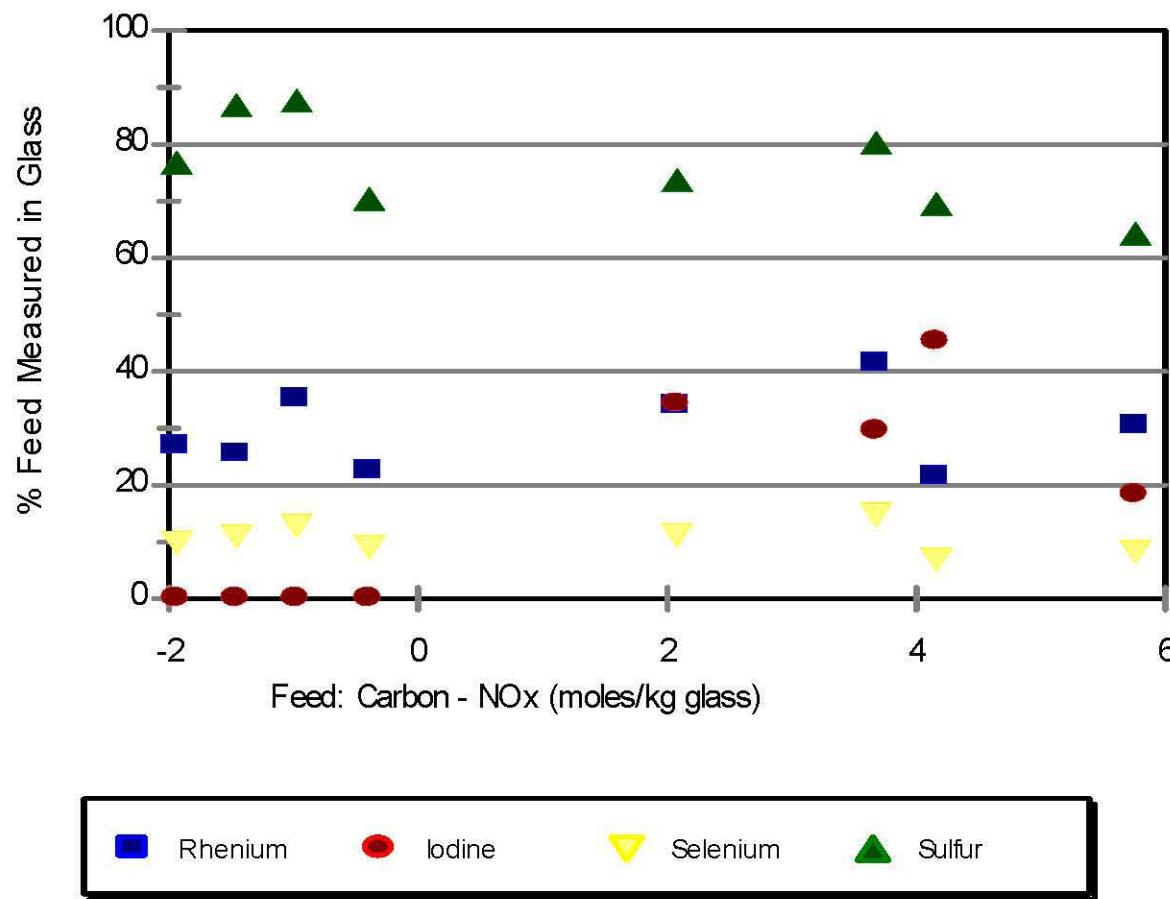
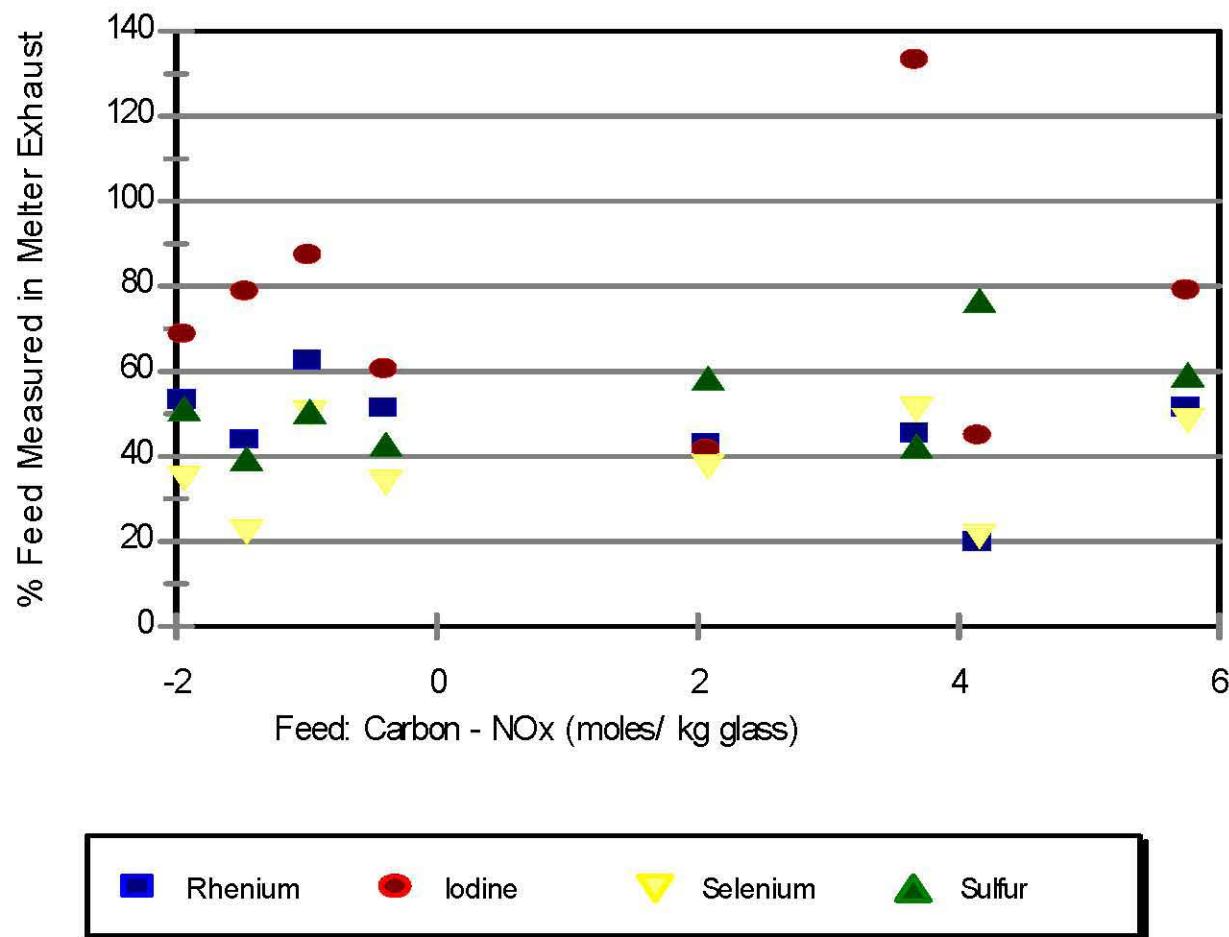


Figure 6.3. Volatile retention in glass versus the difference in carbon and NOx contents in the feed.



**Figure 6.4. Volatiles in melter exhaust versus the difference in carbon and NOx contents in the feed.**