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# Evaluation of Quartz Melt Rate Furnace with the Nitric-Glycolic Flowsheet

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August 2017

SRNL-STI-2017-00212, Revision 0



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**Printed in the United States of America**

**Prepared for  
U.S. Department of Energy**

**Keywords:** *alternate reductant, glycolic, DWPF, melter, offgas, REDOX, hydrogen, glass, cold cap*

**Retention:** *Permanent*

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## **ACKNOWLEDGEMENTS**

The authors would like to thank all those involved in the assembly and operation of the testing equipment: Jon Duvall, Phyllis Workman, and Devon McClane. Thanks to Gary Dobos for fabricating the quartz vessels. Thanks to Joel Jones for developing the LabVIEW automation and control software. Thanks to Whitney Riley, Beverly Wall, Courtney Burckhalter, Kim Wyszynski, and Mark Reboul for analyzing the numerous samples. Thanks to Jack Zamecnik and John Pareizs for reviewing offgas data and ensuring accurate corrections.

All of their high quality contributions to the construction and execution of the system and experiments are echoed in the robust operation of each individual component as part of the whole system.

## EXECUTIVE SUMMARY

The Savannah River National Laboratory (SRNL) was tasked to support validation of the Defense Waste Processing Facility (DWPF) melter offgas flammability model for the Nitric-Glycolic (NG) flowsheet. The work is supplemental to the Cold Cap Evaluation Furnace (CEF) testing conducted in 2014<sup>1</sup> and the Slurry-fed Melt Rate Furnace (SMRF) testing conducted in 2016<sup>2</sup> that supported Deliverable 4 of the DWPF & Saltstone Facility Engineering Technical Task Request (TTR).<sup>3</sup> The Quartz Melt Rate Furnace (QMRF) was evaluated as a bench-scale scoping tool to potentially be used in lieu of or simply prior to the use of the larger-scale SMRF or CEF. The QMRF platform has been used previously to evaluate melt rate behavior and offgas compositions of DWPF glasses prepared from the Nitric-Formic (NF) flowsheet but not for the NG flowsheet and not with continuous feeding.<sup>4</sup> The overall objective of the 2016-2017 testing was to evaluate the efficacy of the QMRF as a lab-scale platform for steady state, continuously fed melter testing with the NG flowsheet as an alternative to more expensive and complex testing with the SMRF or CEF platforms.

The QMRF was operated similarly to the SMRF experimentation performed in 2016 to permit possible comparisons between platforms. During each startup, the QMRF was charged with cullet prepared from a Melt Rate Furnace (MRF) beaker test involving the vitrification of a similarly prepared feed in an open stainless steel beaker. The melter feed was based on the Sludge Batch (SB) 6I Sludge Receipt and Adjustment Tank (SRAT) product used in both the 2014 CEF testing and 2016 SMRF testing. Acid remediations were performed on the feed to generate three batches: one unremediated, one nitric acid remediated, and one glycolic acid remediated. The unremediated feed had a predicted REDUction/OXidation (REDOX) value (ratio of  $\text{Fe}^{2+}$  to total Fe in the produced glass) of  $\sim 0.15$ . The glycolic acid remediation was to raise the target predicted REDOX value to  $\geq 0.3$ . The nitric acid remediation targeted a lower predicted REDOX value of  $\leq 0.1$ . Specific information concerning feed composition and remediations is detailed in the associated SMRF report.<sup>2</sup>

Testing consisted of examining two steady state condition sets per test, with vapor space (VS) temperatures ranging from  $\sim 250$  to  $750$  °C and corresponding slurry feedrates from  $\sim 1.25$  to  $5$  g/min. Relatively stable steady state VS temperature and offgas concentration conditions were achieved and maintained through adjustments to the slurry feedrate, VS heater current, and purge air flowrate.

Notable results from testing with the QMRF platform are as follows:

- Measured REDOX ( $\text{Fe}^{2+}/\Sigma\text{Fe}$ ) values of the glass sampled from the three feeds produced results consistent with the predicted values and the closed crucible – hot insertion ( $\text{CC}_{\text{Hot}}$ ) and MRF methods but were different from results produced during SMRF and CEF testing.
- Measured hydrogen concentrations versus the measured offgas temperatures were lower in the QMRF for all feeds when compared to SMRF testing. Hydrogen concentration trends versus temperature within the same feed ran counter to those observed in SMRF testing – as the gas temperature dropped in the QMRF, the hydrogen concentration either stayed statistically the same or also dropped whereas in the SMRF, the hydrogen concentrations rose as temperature dropped.
- Comparable operational conditions were achieved between the QMRF and the SMRF during the limited time frame of the QMRF tests. Similar vapor space temperatures were achievable with only minor modifications necessary for feeding issues.

Based on the QMRF testing observations and results, recommended items for future implementation of this platform are:

- Operation of the QMRF at DWPF nominal conditions for a single test (i.e., not manually varying the VS temperature) to achieve a single steady state at typical operating conditions for a longer period of time. Extended single steady state testing could ensure that true steady state conditions have been achieved as well as provide true melt rate analyses for the NG flowsheet feed.
- Development of a feed system that can operate at low feedrates of about 1 g/min so that accurately scaled steady state operation can be achieved at lower temperatures. Optimization of a slurry feed pump system that can operate at benchtop scales without segregation of the sludge and feed solids would allow for more accurate comparisons to larger scale platforms in terms of offgas production as a function of feed solids added to the melter.
- Modification of the QMRF vessel to allow for a deeper melt pool, glass pouring, or both, to extend testing durations. Currently, the runtime of the QMRF is limited by the volume within the vessel to receive feed and convert to cold cap and glass. With a deeper vessel or the ability to pour the glass during testing, the operating limit can be extended to allow additional time to observe multiple steady states. It would also then be possible to compare the pour stream glass to the solidified melt pool glass in terms of REDOX and more directly compare to glasses produced in the SMRF and CEF.
- The QMRF platform would be an ideal system for testing and implementation of an *in situ* melt pool REDOX probe to track the progression of REDOX in the melt as a function of VS temperature, melt pool location, and residence time in the melter that could then be applied to larger melter systems.



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## LIST OF ABBREVIATIONS

A	ampere
CC <sub>Hot</sub>	Closed (sealed) Crucible – Hot Insertion method
CEF	Cold Cap Evaluation Furnace
DAS	Data Acquisition System
DWPF	Defense Waste Processing Facility
ELN	Electronic Laboratory Notebook
FTIR	Fourier-Transform Infrared spectrometer
GC	Gas Chromatograph
ICP-AES	Inductively Coupled Plasma – Atomic Emission Spectroscopy
inwc	Inches of water column
LFL	Lower Flammability Limit
MRF	Melt Rate Furnace
mmol gas / g feed (solids)	Millimoles of gas species per grams of feed total solids
MS	Mass Spectrometer
NF	Nitric-Formic
NG	Nitric-Glycolic
PI	Principal Investigator
PPE	Personal Protective Equipment
QMRF	Quartz Melt Rate Furnace
R&D	Research & Development
REDOX	Reduction/Oxidation; specifically Fe <sup>2+</sup> /ΣFe ratio
sccm	Standard Cubic Centimeters per Minute (1 atm, 21.11 °C)
slm	Standard Liters per Minute (1 atm, 21.11 °C)
SB	Sludge Batch
SMRF	Slurry-fed Melt Rate Furnace
SRAT	Slurry Receipt and Adjustment Tank
SRNL	Savannah River National Laboratory
T <sub>actual</sub>	Actual offgas temperature
T <sub>measured</sub>	Measured vapor space temperature
TOC	Total Organic Carbon
TTQAP	Task Technical and Quality Assurance Plan
TTR	Technical Task Request
Wt %	Weight Percent

## 1.0 Introduction

The Savannah River National Laboratory (SRNL) was tasked to support validation of the Defense Waste Processing Facility (DWPF) melter offgas flammability model for the Nitric-Glycolic (NG) flowsheet. This work is supplemental to the Cold Cap Evaluation Furnace (CEF) testing conducted in 2014<sup>1</sup> and the Slurry-fed Melt Rate Furnace (SMRF) testing conducted in 2016<sup>2</sup> that supported Deliverable 4 of the DWPF & Saltstone Facility Engineering Technical Task Request (TTR) and Task Technical and Quality Assurance Plan (TTQAP).<sup>3, 5</sup> The Quartz Melt Rate Furnace (QMRF) was evaluated as a bench-scale scoping tool to potentially be used in lieu of or simply prior to the use of the larger-scale SMRF or CEF. The QMRF platform has been used previously to evaluate melt rate behavior and offgas compositions of DWPF glasses prepared from the Nitric-Formic (NF) flowsheet but not for the NG flowsheet and not with continuous feeding.<sup>4</sup> The overall objective of the 2016-2017 testing was to evaluate the efficacy of the QMRF as a lab-scale platform for steady state, continuously fed melter testing with the NG flowsheet as an alternative to more expensive and complex testing with the SMRF or CEF platforms.

Since its inception in 2004, the QMRF has been operated as a semi-batch melt rate furnace with limited offgas analyses. In the 2016-2017 testing, the QMRF was operated similarly to the continuously fed 2016 SMRF testing. Melter operations were performed to target steady state conditions at varying vapor space (VS) temperatures to simulate melter offgas flammability testing conditions utilized for modeling efforts. For each test, the QMRF was charged with cullet prepared from a Melt Rate Furnace (MRF) test involving the vitrification of a similarly prepared feed and was operated in such a way as to target steady state conditions at two different VS temperatures. The melter feed was based on the Sludge Batch (SB) 6I Sludge Receipt and Adjustment Tank (SRAT) product used in both the 2014 CEF testing and 2016 SMRF testing. Acid remediations were performed on the feed to generate three batches: one unremediated, one nitric acid remediated, and one glycolic acid remediated. The unremediated feed had a predicted REDuction/OXidation (REDOX) value (ratio of  $\text{Fe}^{2+}$  to total Fe in the produced glass) of  $\sim 0.15$ . The glycolic acid remediation was to raise the target predicted REDOX value to  $\geq 0.3$ . The nitric acid remediation targeted a lower predicted REDOX value of  $\leq 0.1$ . Specific information concerning feed composition and remediation is detailed in the associated SMRF report.<sup>2</sup>

Due to the novel parameters under which the QMRF was operated, a run plan and detailed R&D directions were written to describe the specific goals and tasks during testing.<sup>6, 7</sup> This report is intended to describe the operating conditions under which the QMRF was tested in order to evaluate its efficacy with the NG flowsheet. Selected analytical data will be presented, as they are relevant to the scope of the discussion.

### 1.1 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in Manual E7 Procedure 2.60. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.

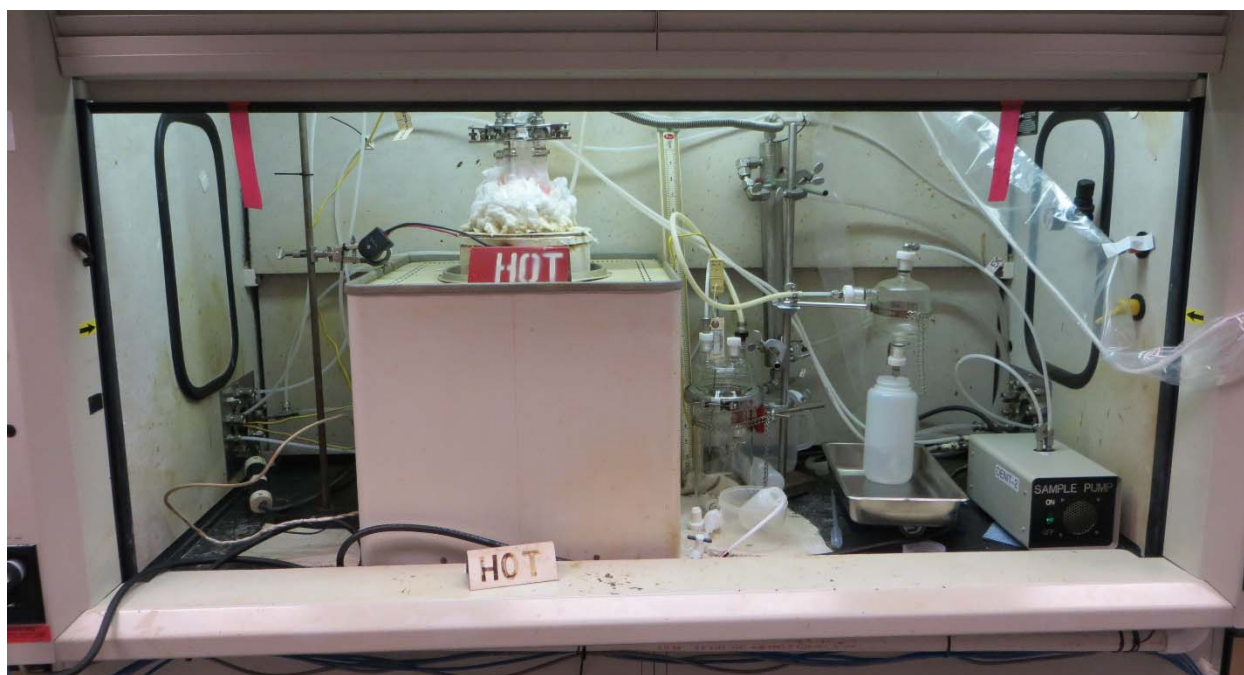
Details of various portions of the experiments are contained in the following Electronic Laboratory Notebooks (ELN):

- D. H. Miller, "Alt Reductant Melter Offgas Flammability", ELN experiment T8786-00095-11.
- M. S. Williams, "Alt Reductant Melter Offgas Flammability – Volume 2", ELN experiment I7770-00157-17.

## 2.0 System Description

### 2.1 Quartz Melt Rate Furnace System

The QMRF is comprised of a top loading furnace, a slurry feeding system, a control system, a quartz vessel, and an offgas conditioning and sampling train. The furnace and offgas train are small enough to fit within a standard laboratory hood (Figure 2-1) while the feeding and control systems occupy adjacent lab benchtop space. The SMRF and CEF platforms, though significantly larger, share many design aspects with the QMRF, most notably slurry feeding. The smaller platforms of the closed (sealed) crucible for hot insertion ( $CC_{Hot}$ ) and the Melt Rate Furnace (MRF) share less physical characteristics but produce similar results in terms of the glass products. The  $CC_{Hot}$  crucibles are sealed from the outside atmosphere with a lid and nepheline gel that cures *in situ* as the crucible is wholly inserted into a hotbox furnace. The MRF open top stainless steel beakers isolate the vitrifying glass by the formation of a “cold cap” due to the directional heating of being placed in a similar top loading furnace. The  $CC_{Hot}$  methodology is the currently accepted REDOX standard method due to tiebacks with the DWPF melter pour spout and the NF flowsheet.



**Figure 2-1. Quartz Melt Rate Furnace (QMRF).**

#### 2.1.1 Melter and Quartz Vessel

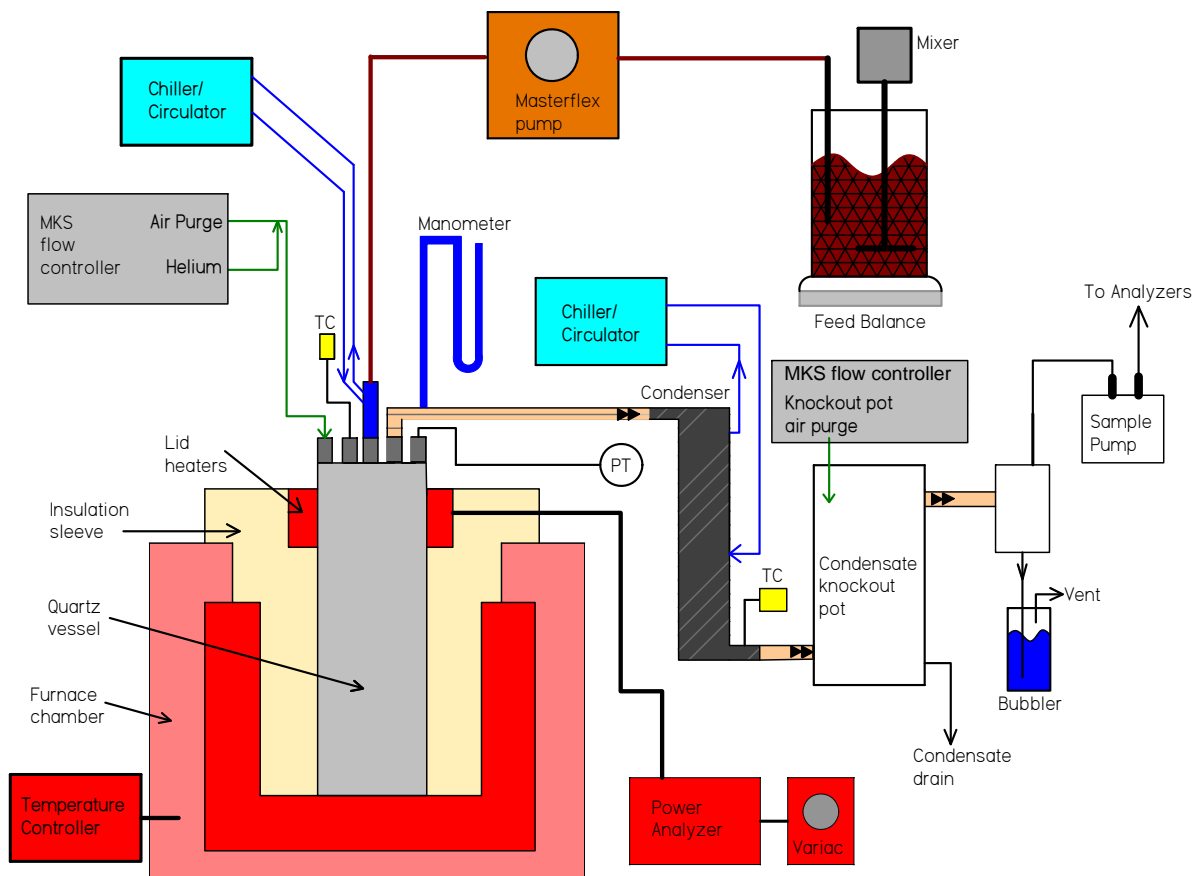
The QMRF is designed to mimic the heat transfer characteristics of a large-scale joule-heated melter, which is accomplished by providing heat in one dimension through the bottom of a single-use 4" diameter quartz glass vessel (Figure 2-2) using a radiant heater coil below the vessel.

A sketch of the entire system is shown in Figure 2-3 with detailed information pertaining to system components listed in Appendix Table A-1. The sides of the vessel are insulated in the melt pool area to minimize both radial heat transfer to or from the melt pool and heat exchange with the plenum (i.e. the vapor space above the glass melt pool). This directional heating melter configuration relies on convective and conductive heat transfer between the glass pool and cold cap. The cold cap is the region where the feed undergoes physical and chemical changes as it is progressively converted from feed slurry to dried material to molten glass. The vessel heaters were controlled by a thermocouple mounted inside the

bottom of the furnace and maintained a temperature of  $\sim 1150$  °C throughout testing. Additional heating (separate from that supplied to the melt pool) is applied to the plenum above the melt pool through a radiant heating collar that surrounded the upper part of the vessel. The plenum heater was set according to a thermocouple inserted into the vapor space of the vessel.



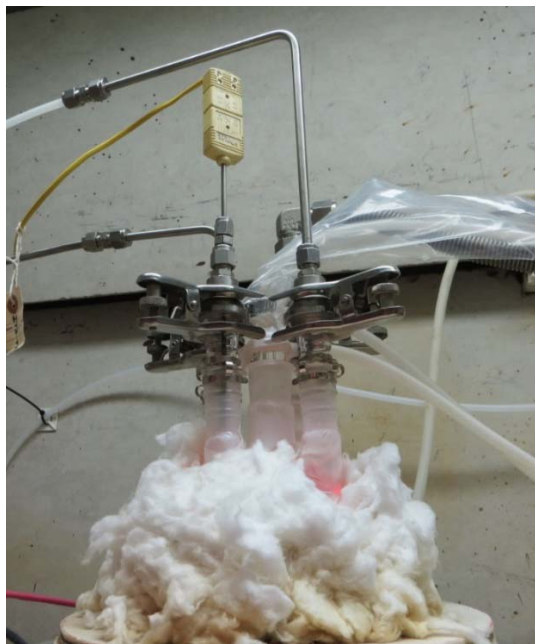
**Figure 2-2. QMRF Vessel.**



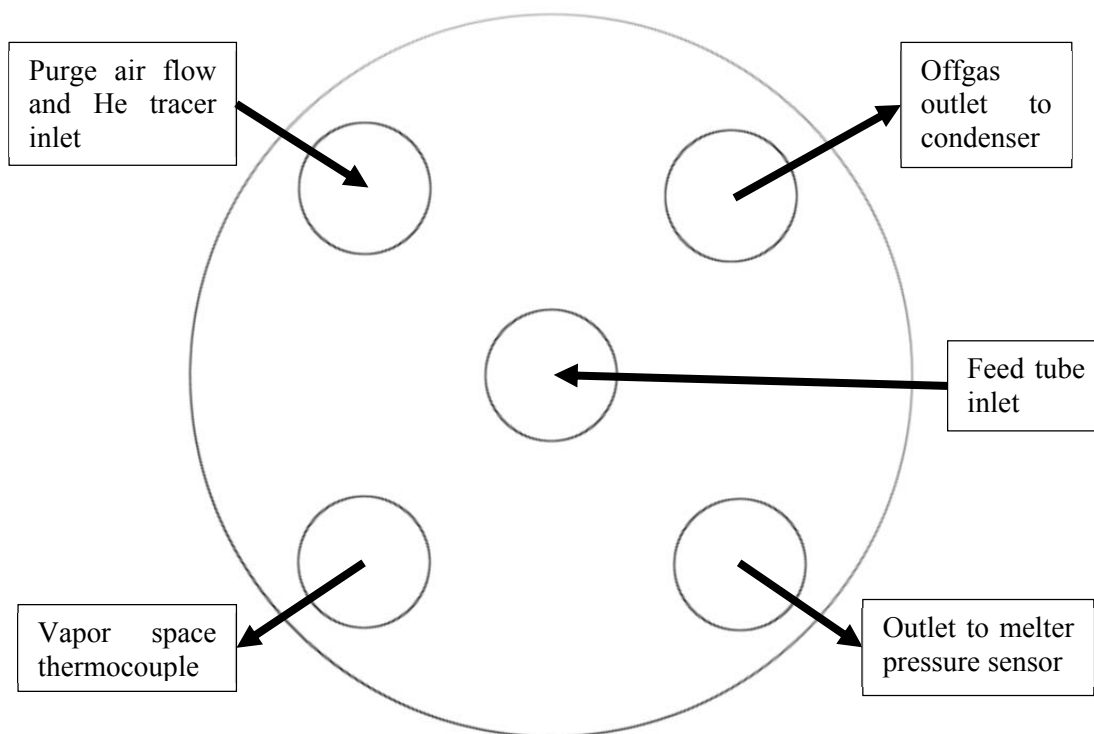
**Figure 2-3. QMRF Diagram.**

*2.1.1.1 Quartz Vessel Top*

The top of each quartz vessel had five ports (Figure 2-4). The ports are diagramed and labelled in Figure 2-5.



**Figure 2-4. QMRF Vessel Top.**



**Figure 2-5. QMRF Top Ports Diagram**

The four perimeter ground glass ports received the quartz ball half of ball-and-socket connections; metal socket halves of the ball-and-socket fittings were held to the vessel with metal clamps. Swagelok fittings were utilized to seal the system around the gas lines and thermocouple probe. The central ground glass port received the ground glass feed tube. Rubber tubing was used for the slurry feed and chilled water, and was clamped over the inlet fittings. Angel hair glass wool was used to insulate the vessel top, reducing excess heat loss and the impact of thermal shine on the connections above the melter.

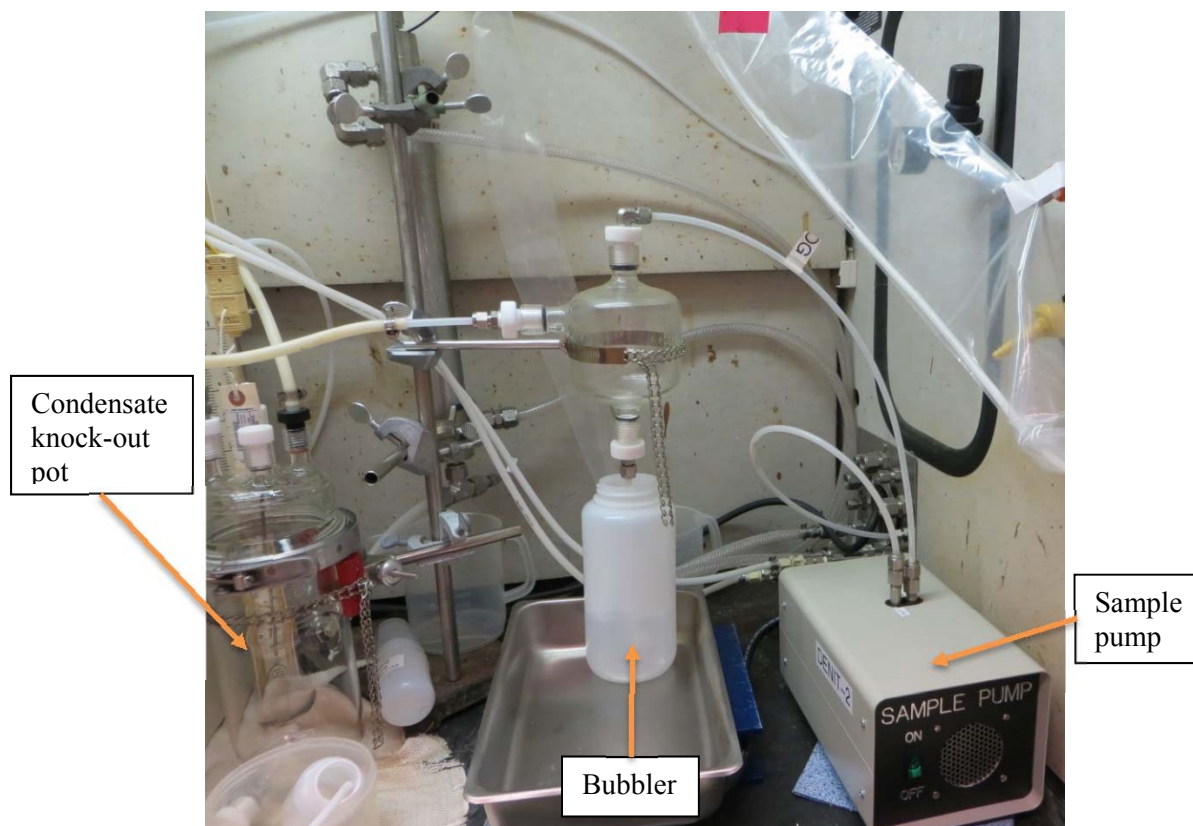
#### *2.1.1.2 Purge Air*

The melter purge served as dilution air and as a cooling source for low temperature testing. The purge was introduced into the vapor space through a top port located across from the offgas port. The same line was utilized for the addition of the He tracer gas employed in offgas dilution calculations. The purge air is supplied by the building system, which automatically dries the air to approximately 1000 ppm H<sub>2</sub>O.

#### *2.1.1.3 Offgas Sampling*

The offgas port connected the melter to the offgas conditioning and sampling train. Flexible metal tubing connected the offgas port of the QMRF vessel to a vertical condenser in line before the knockout pot. The knockout pot was purged with a low airflow (50 sccm) and connected to a bubbler utilized to isolate the system from the ambient atmosphere and be a visual check of the offgas sampling. The offgas sampling line was attached to the offgas system before the bubbler. A sample pump was used to draw off a small volume of gas and supply it, under pressure, to the mass flow controller cluster of the mass spectrometer (MS) and Fourier-transform infrared spectrometer (FTIR). The bubbler ensured that the sampling pump was not sampling more gas than the offgas from the melter, which would have resulted in unintended dilution of the sample. Figure 2-6 shows the condensate knockout pot, the bubbler, and the offgas sampling pump for the analyzers.





**Figure 2-6. QMRF Offgas Sampling and Ventilation System.**

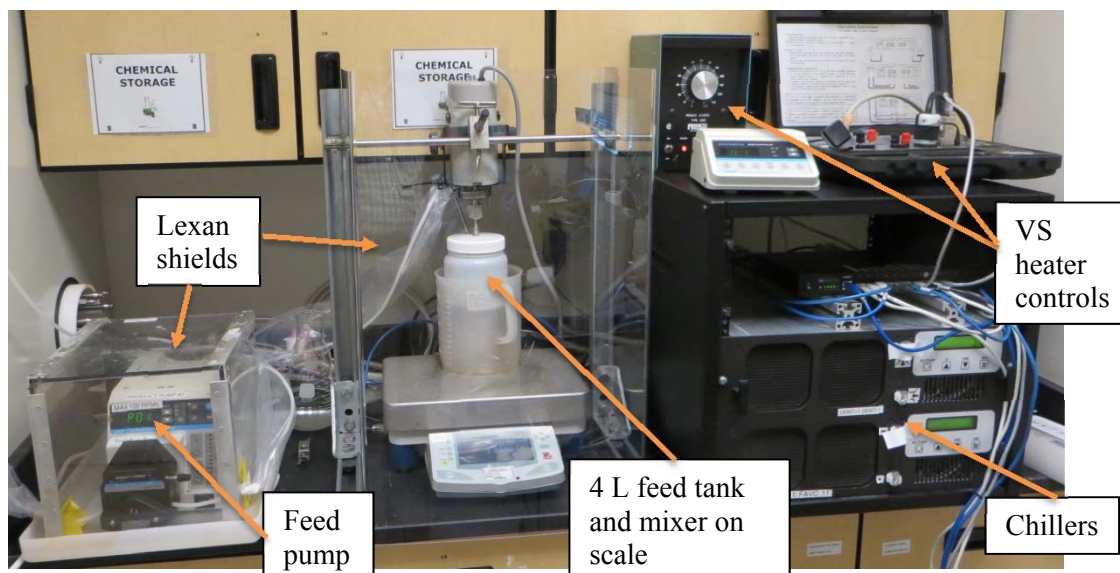
#### *2.1.1.4 Helium tracer gas*

Helium (He) was introduced into the melter through the air purge line (5–10 sccm). The helium served as a tracer gas to allow estimates of offgas dilution. The helium flow was turned on and allowed to stabilize on the MS for approximately 5–10 minutes. In some tests, the He remained on during the entire test, while in others the He was turned off during steady state conditions. The advantage of turning the He off during steady state testing relates to analytical overlap on the instrumentation. When utilizing a gas chromatograph, helium and hydrogen elute very near one another, making quantification of low concentrations of hydrogen difficult.

#### *2.1.2 Melter Feed System*

The QMRF feed system consisted of an agitated feed tank sitting on a platform scale, a peristaltic feed pump, the melter feed tube assembly and a chiller to provide cooling water to the feed tube assembly.

Figure 2-7 shows the feed system along with the VS heater controls situated on the lab bench. Secondary containment around the feed system consisted of a Lexan box and plastic pan around the pump, plastic sheathing (i.e. “lay-flat” tubing) around the feed line, and a plastic pitcher and shield around the feed tank and mixer.



**Figure 2-7. QMRF Melter Feed System, Water Chillers and VS Heater Controller.**

The four liter poly feed tank was agitated by a  $\frac{1}{4}$  horsepower laboratory mixer using a 3" flat blade impeller. There were no baffles inside the container. The agitator speed was controlled by the Data Acquisition System (DAS) and mixing speed was set visually. The speed of the mixer was such that there was minimal air entrainment in the slurry and when probed, minimal solids were felt settling out on the bottom of the vessel. The QMRF feed tube assembly (Figure 2-4) fit securely in the center vessel top port via a ground glass fitting and a metal clamp and was cooled via chilled water flowing through an integrated water jacket. A chiller provided chilled water flow to the water jacket to prevent thermal drying of feed in the feed tube.

The Master-Flex adjustable-speed peristaltic feed pump was controlled by the DAS. Master-Flex Tygon® tubing, size L-15, was used for the pump tubing. The Tygon® tubing ran from the feed inlet wand to the melter feed tube and was wrapped with "lay-flat" tubing for secondary containment in case of a leak or rupture. The feed inlet wand was made from  $\frac{1}{4}$ " stainless steel tubing with the end crimped shut and multiple slots machined into the side above the crimp. The slots allowed feed into the wand and were positioned away from the direction of rotation (downstream side) of feed in the container. Between tests, the Tygon® tubing was repositioned in the pump head (moved towards the low-pressure side) to relocate the wear spot created by the pump rollers and, on occasion, the entire length of Tygon® tubing was replaced.

### 2.1.3 Data Acquisition System

The DAS consisted of a PC using LabVIEW software provided by Research & Development (R&D) Engineering in SRNL. The DAS recorded the output of QMRF instrumentation as well as operational data from the heater controls of the melter and VS heaters. A list of QMRF instrumentation is shown in Table A-1. The DAS screen displayed data from the instrumentation and heaters (Figure A-1). The DAS provided on-screen control of the feed tank agitator speed, feed pump rpms, and system gas flows through an MKS mass flow controller. Visible and audible alarms on the DAS were associated with critical temperature and pressure readings in the melter as well as with the feed container scale.

## 2.2 System Checkout

Prior to the start of testing at temperature, each subsystem was checked to verify that it functioned properly. All instruments were successfully operated from the control computer. Alarms and interlocks

were verified using simulated signals. Water runs were completed to verify the accuracy of the feed delivery system and weight scales. The data collection, graphing and storage systems were verified during the initial shakedown testing. Operation of the DAS, computer-instrumentation interface, interlocks, and alarms were also verified.

### **3.0 Experimental Procedure**

#### **3.1 Feed Preparation**

The melter feed utilized in this testing was from the concurrent SMRF testing.<sup>2</sup> The composition of the source SRAT product of the feeds utilized is detailed in Table 3-1. Frit was added to form a slurry targeting a waste loading of approximately 36 weight percent (wt %) and a total solids of approximately 49 wt %. The glycolic and nitric acid remediations were calculated based on targeted glass REDOX values. Either 70 wt % glycolic acid or 50 wt % nitric acid was added directly to the sludge with no additional processing. Dilution water was added to the acid remediations to reduce the total solids wt % down to approximately 42 wt % to improve feeding and preserve comparisons to the SMRF and CEF. For the unremediated test, the slurry was used as produced. Since these feeds were the same as those utilized in the SMRF, they were also subjected to pouring through a mesh screen to remove any large particles.

**Table 3-1. Source SRAT Product Composition.**

Element	Elemental wt. %		Oxide	Oxide wt %
Al	14.01		Al <sub>2</sub> O <sub>3</sub>	26.47
Ba	0.14		BaO	0.16
Ca	1.26		CaO	1.77
Cr	0.18		Cr <sub>2</sub> O <sub>3</sub>	0.27
Cu	0.12		CuO	0.16
Fe	22.32		Fe <sub>2</sub> O <sub>3</sub>	31.92
K	0.42		K <sub>2</sub> O	0.51
Mg	1.05		MgO	1.75
Mn	6.79		MnO	8.75
Na	13.49		Na <sub>2</sub> O	18.20
Ni	2.99		NiO	3.80
P	<0.100		P <sub>2</sub> O <sub>5</sub>	<0.23
Pd	<0.100		PdO	<0.12
Rh	<0.100		RhO <sub>2</sub>	<0.13
Ru	<0.100		RuO <sub>4</sub>	<0.16
S	0.30		SO <sub>4</sub>	0.89
Si	1.57		SiO <sub>2</sub>	3.36
Sn	<0.100		SnO	<0.11
Ti	<0.100		TiO <sub>2</sub>	<0.17
Zn	0.11		ZnO	0.14
Zr	0.21		ZrO <sub>2</sub>	0.28
<b>Caustic quenched anions</b>	<b>Concentration (mg/kg)</b>		<b>Total Solids (wt. %)</b>	<b>33.5%</b>
F	<500		<b>Insoluble Solids (wt. %)</b>	<b>18.7%</b>
Cl	<500		<b>Soluble Solids (wt. %)</b>	<b>14.7%</b>
NO <sub>2</sub>	<500		<b>Calcined Solids (wt. %)</b>	<b>18.7%</b>
NO <sub>3</sub>	67650		<b>pH</b>	<b>5.72</b>
SO <sub>4</sub>	1770			
C <sub>2</sub> O <sub>4</sub>	1780			
PO <sub>4</sub>	<500			
HCO <sub>2</sub>	3190			
C <sub>2</sub> H <sub>3</sub> O <sub>3</sub>	51050			

The feeds were loaded into 4L poly feed bottles and placed on the feed scale. The agitator blade and feed suction wand were added to the poly bottle through the lid and the scale was tared with all equipment in place and operational.

### 3.2 Initial Startup

MRF tests were run with each of the QMRF feeds to prepare ~500 g of fully vitrified glass for REDOX verifications and batch cullet. For the QMRF tests, the vessels were charged with ~200g of cullet from the associated MRF test. Once loaded, the melter was ramped to 1150 °C and the VS heaters were set to ~3 amps. After arriving at operational temperature, the melter was allowed to soak and stabilize for ~20 minutes. Once the melter temperature was stable, feeding was initiated to fill the feed line (pump speed of ~40 rpm) and build the initial cold cap (pump speed of ~20 rpm). After sufficient feed had been fed to establish a stable cold cap (approximately 5 minutes of feeding), the feedrate and VS heater power were reduced to target steady state conditions.

### 3.3 Testing Conditions

Target test conditions for each of the feeds were set to produce similar VS temperatures as in the SMRF testing.<sup>2</sup> The conditions listed in Table 3-2 were starting points based on previous QMRF operations<sup>4</sup>; however, the portions of testing determined to represent steady states were based on where the system self-stabilized with the combination of targeted variable inputs for the VS heater, feedrate, and purge air flow. Each variable is utilized in conjunction to adjust the VS temperature; for example, to lower the VS temperature, the VS heater power can be lowered and the purge airflow can be increased. A wide range of VS temperatures was targeted to examine the utility of the QMRF to support melter offgas flammability modeling validations.

**Table 3-2. Summary of target steady state testing conditions**

Target VS temperatures (°C) to compare to SMRF	Target VS heater current outputs (amps)	Target feedrates (g/min)
650, 550, 450, 350, <300	From 0 to 3	From 1.25 to 5

The duration of each test was limited due to the lack of a pouring mechanism in the QMRF vessel design; only a finite amount of material may be added to the vessel before it fills up. Due to the time restriction of the current QMRF, only two steady states were targeted during testing for each feed. The testing conditions progressed from high to low VS temperatures.<sup>2</sup>

#### 3.3.1 Steady State Test Conditions

Just as with the CEF and SMRF testing, steady state conditions were defined to be a time interval during which the following conditions were maintained:

- Vapor space temperature ( $\pm 25$  °C )
- Feedrate ( $\pm 1$  g/min)
- Offgas concentration readings ( $\pm 15\%$ )
- Melter vessel air purge ( $\pm 15\%$ )

The variables were adjusted to approach a stable condition and once a combination of variables produced a steady state, an attempt was made to maintain that state for a time period between one and two hours.

#### 3.3.2 Sampling and Analyses

Upon completion of each test, after cooling, glass samples were taken directly from the vessel and were analyzed for elemental composition and REDOX ( $\text{Fe}^{2+}/\Sigma\text{Fe}$  ratio) values. To retrieve glass samples, the vessel was broken away from the vitrified portion of the feed and samples were pulled from the interior portion of glass. The  $\text{Fe}^{2+}/\Sigma\text{Fe}$  ratio was determined utilizing UV/Vis spectroscopy and elemental composition was determined by Inductively Coupled Plasma–Atomic Emission Spectroscopy (ICP–AES).<sup>8,9</sup>

The offgas composition was continuously analyzed via MS and FTIR. The periods for each analysis were ~16 seconds for FTIR and ~7 seconds for MS. Various species were analyzed with each instrument with some redundancies to ensure accurate measurements (Table 3-3).

**Table 3-3. Offgas speciation by instrumentation**

Offgas species	MS	FTIR
H <sub>2</sub>	X	
He	X	
CO		X
CO <sub>2</sub>	X	X
O <sub>2</sub>	X	
N <sub>2</sub>	X	
N <sub>2</sub> O		X
NO	X	X
NO <sub>2</sub>	X	X
Ar	X	

The MS was calibrated using a series of cylinders of standard calibration gas mixtures. Each gas mixture was National Institute of Standards and Technology (NIST) traceable and the certificates of analysis are reported in the associated ELN.<sup>a</sup>

The FTIR uses internal calibration spectra to generate concentration data; the uncertainty of each analysis is based on the accuracy of the calibration setup and the interferences of overlapping analytes.

## 4.0 Results and Discussion

### 4.1 Melter Operations and Steady State Conditions

Melter operations were significantly simplified compared to those of the SMRF and CEF. Each QMRF test could be performed during typical business hours because of the finite limit on the amount that can be fed to a vessel without a pouring mechanism. This limited timeframe along with the minimal footprint of the equipment also reduced the personnel required to operate the platform. A standard two-person team (either one principal investigator (PI) and one technician or two PIs) could perform all of the functions of operating the feed system, the melter system, the purge system, and the offgas instrumentation as compared to the shifts of individuals necessary to operate the SMRF or CEF platforms during 24-hour operations over multiple days. These simplified operations potentially lower costs as well, as long as the data produced from the QMRF is as useful as that generated from the SMRF and CEF.

Melter performance targeted conditions detailed in Table 3-2. Ideally, the target conditions for feedrate and power would be scaled to the glass surface area of the melter. The QMRF surface area is approximately four times smaller than the SMRF, so the feedrates and air purges should be about four times smaller in the QMRF.

Typical feedrates in the SMRF ranged from approximately 5 to 25 grams per minute. Scaled to the QMRF, the feedrate would be as low as 1.25 grams per minute. With the current peristaltic pump configuration, this low of a rate was not achievable without significantly increasing the probability of feed segregation (i.e., separation of the sludge and frit in the feed transfer tubing) or plugging. Therefore, the feedrate was set to the slowest rate practically achievable with the QMRF setup (~5 grams per min). Scaling of the feedrate presents an opportunity for continued improvement to develop a feed system that can be operated at low flowrates.

<sup>a</sup> Documentation for MS calibration gases is in the ELN T7909-00035-02.

Likewise, the total airflow into the SMRF including air leakage could not be scaled because it was too high. The purge airflow, not counting leakage, could be lowered to scaled values, so this value was scaled appropriately. The typical SMRF purge airflow, discounting leakage, was on the order of 20 slm. Scaled to the QMRF, this flow would be approximately 5 slm. The purge air flowrates were set to between 3 and 6 slm to maintain less than 10 inwc pressure in the melter.

The actual steady state operating conditions were based on where the system self-stabilized with the variable combinations that could be achieved. Figure 4-1 thru Figure 4-3 show the VS temperature, VS heater current output, melter temperature, feed solids addition rate and purge airflow profiles for each test; the regions declared as steady states are highlighted in orange. Some variable combinations resulted in more stable conditions than others, but overall, the operational variables provided sufficient control over the testing conditions. The test of controllability was the ability to stabilize target VS temperatures within the target range by adjusting the operating conditions and then being able to maintain those stable temperature and offgas measurements without making any operational adjustments.

The one condition that showed the least consistency was the rate of feed solids addition. The calculation for feed solids added to the melter was based on feedback from the feed tank scale as the tank was stirred with a mixer blade and feed was pumped out to the melter. In the first test with unremediated feed, the tank weight fluctuated through periods of rapid decline and slow decline even while the pump revolutions per minute (rpm) remained constant. During the subsequent tests (second test with glycolic-remediated feed and final test with nitric-remediated feed), the fluctuations decreased significantly. The source of the fluctuations in the first test is unknown, but, when averaged, produces comparable overall feedrates. The oscillations in the feedrate did not result in oscillations of the offgas production so utilizing an average feedrate value was acceptable. The major impact of the oscillations would have been on the plots of offgas production rates as a function of feedrate. An averaged feedrate was employed to eliminate the artificial offgas fluctuations in the plots.

**Table 4-1. QMRF and SMRF Averaged Steady State Conditions and Offgas Production Rates**

Feed remediation	Melter platform	Average measured VS temp	Average measured feed solids rate from feed tank weight	Average measured H <sub>2</sub> production	Average measured CO production
Units		°C	g/min	mmol gas / g feed solids	mmol gas / g feed solids
Unremediated	QMRF	517	2.4	0.032	0.032
	SMRF	500	2.8*	0.041	0.021
	QMRF	468	3.0	0.037	0.016
Nitric	QMRF	509	2.4	0.035	0.016
	SMRF	496	3.3*	0.031	0.013
	SMRF	382	1.1*	0.058	0.021
	QMRF	339	2.5	0.027	0.007
Glycolic	QMRF	459	2.1	0.067	0.022
	SMRF	421	1.3*	0.068	0.023
	SMRF	324	1.1*	0.081	0.025
	QMRF	282	2.2	0.043	0.014

\* Scaled to QMRF (SMRF values divided by four)

The trends of H<sub>2</sub> and CO production versus temperature would be expected to be the same in the QMRF as in the SMRF (and CEF) with the H<sub>2</sub> rate increasing with decreasing temperature. For the unremediated feed, the range of temperatures (468–517 °C; ±25 °C) was not sufficiently wide to necessarily expect significant differences in the H<sub>2</sub> rates. The two QMRF rates are both lower than the SMRF rate at the intermediate temperature, but not significantly enough to conclude that they are actually lower. At steady state, the scaled feedrates would be expected to also be nearly the same. The range of values from 2.4 to 3.0 g/min is reasonably similar.

For the nitric remediated data, the QMRF and SMRF H<sub>2</sub> values at about 500 °C match very well. The SMRF value at 383 °C is higher as expected, but the QMRF value at 339 °C is lower rather than higher. The feedrate for the SMRF data at 382 °C is significantly lower than at the higher temperatures, but the QMRF feedrate is about the same as at 509 °C. As noted previously, there was a lower limit to the QMRF feedrate that when scaled to the SMRF, was higher than the SMRF flowrates at similar temperatures. The reported H<sub>2</sub> rates are the ratio of the H<sub>2</sub> rate to the solids feedrate, so if the QMRF were being over-fed, this value would be lower than expected, which it is. The much lower QMRF H<sub>2</sub> rate is most likely due to not being at steady state, with the feedrate exceeding the glass melting and H<sub>2</sub> generation rates.

The H<sub>2</sub> rates for the glycolic remediated data show similar trends, with good agreement at the higher temperature, but with QMRF rate at the lower temperature being too low, again probably due to not being at steady state. The agreement between the QMRF and SMRF H<sub>2</sub> rates was reasonably good at the higher VS temperatures, and if lower feedrates could be achieved, agreement at lower temperatures might be possible. However, in the QMRF, with a very short operating duration, it is difficult to determine if steady state has actually been achieved, as is described below. The major factors controlling the VS temperature were the VS heater current (amps) and the purge airflow; the feedrate would have been another factor had scaling, previously discussed, not been an issue.



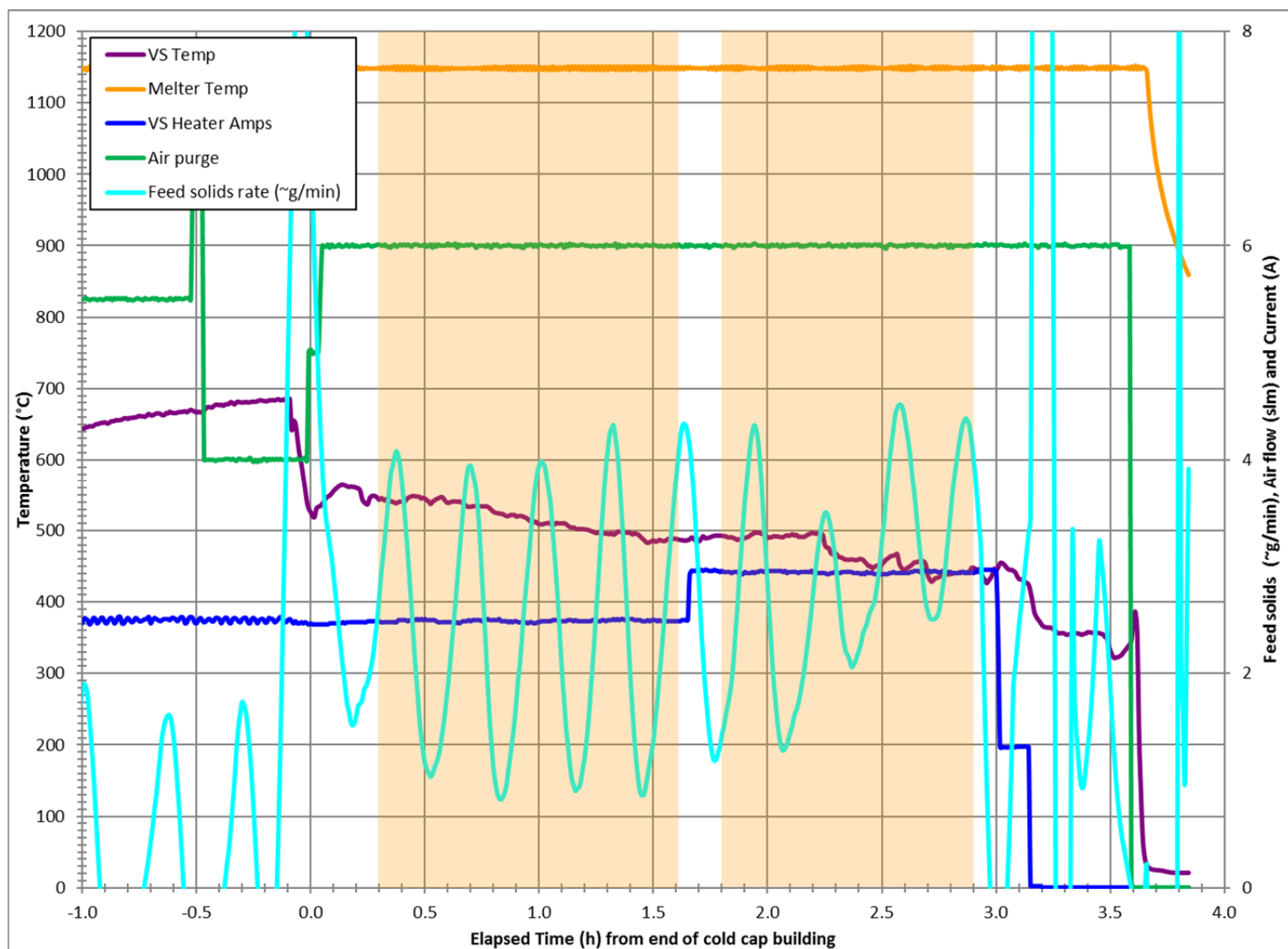


Figure 4-1. Process Condition Profiles (VS temperature, melter temperature, VS heater current, feed solids rate, and purge airflow) for Unremediated QMRF Testing.

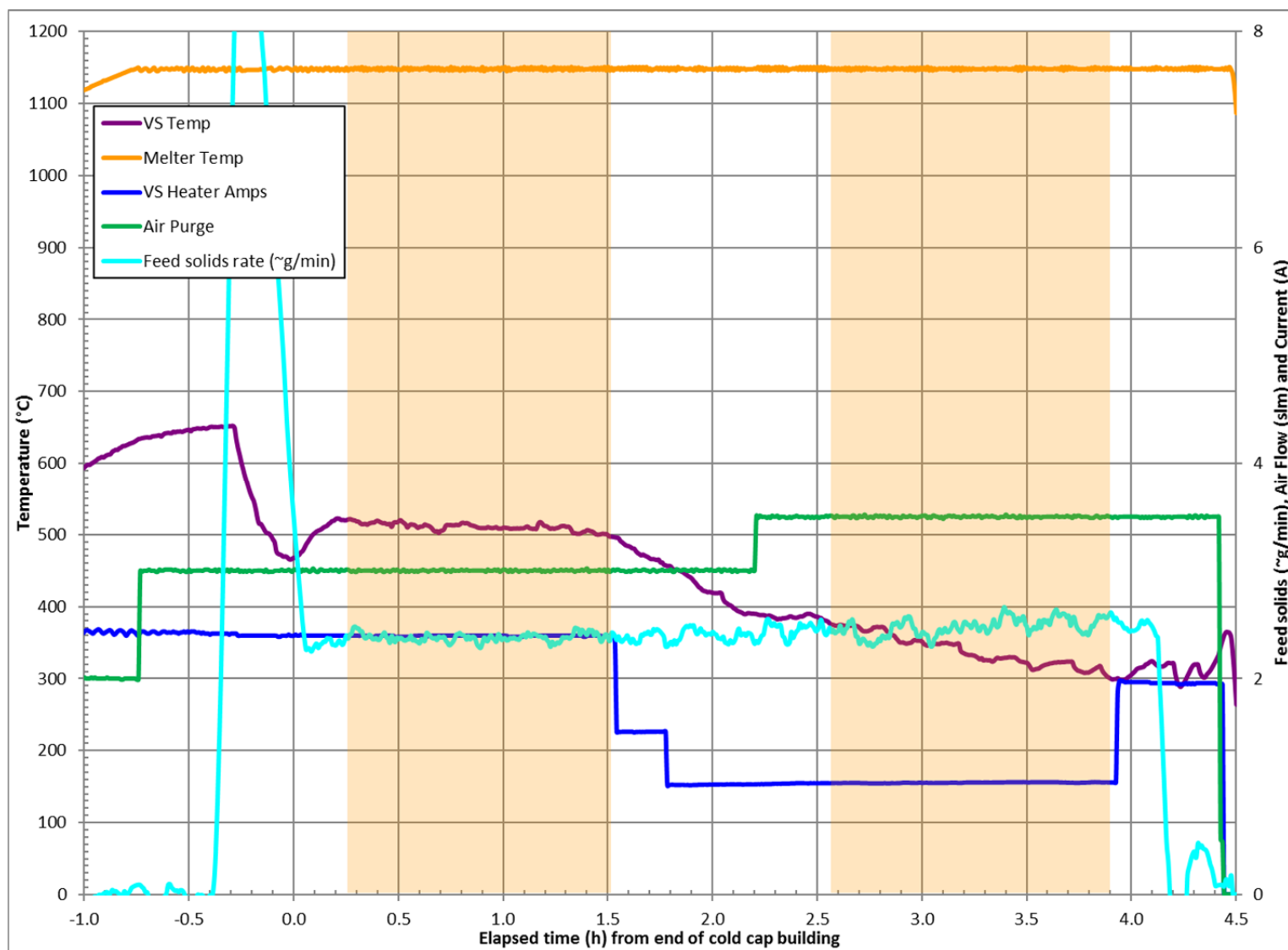


Figure 4-2. Process Condition Profiles (VS temperature, melter temperature, VS heater current, feed solids rate, and purge airflow) for Nitric-remediated QMRF Testing.

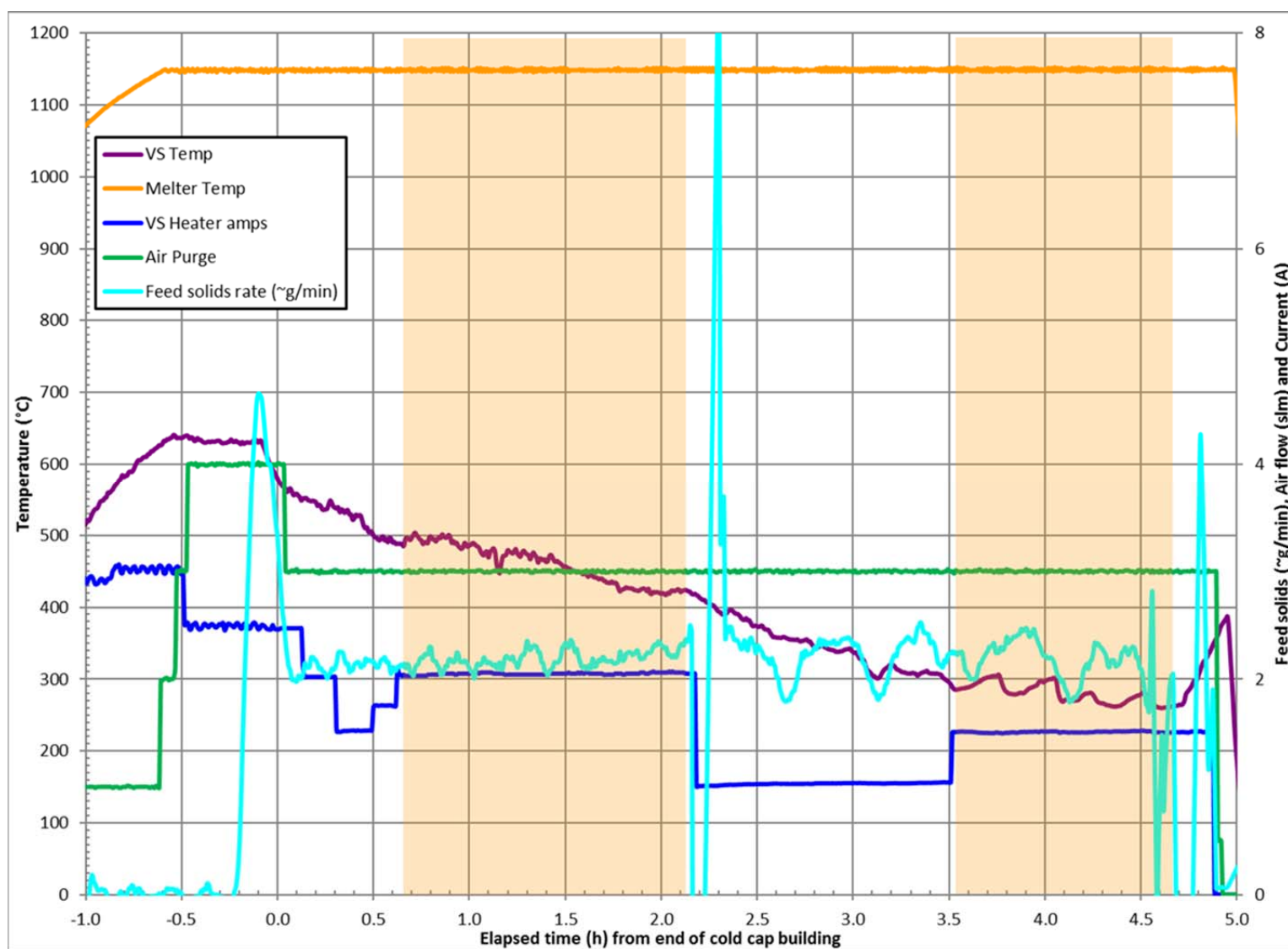
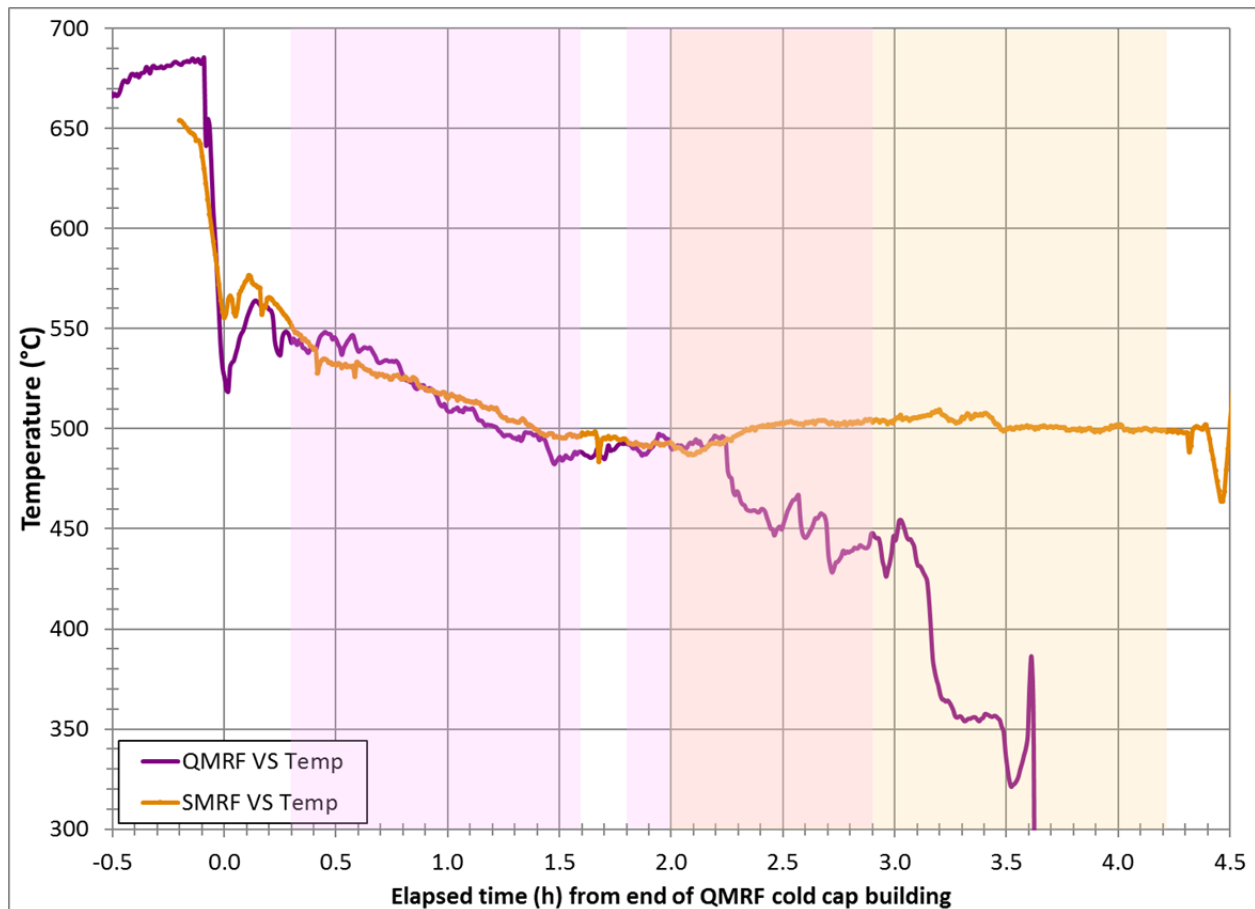


Figure 4-3. Process Condition Profiles (VS temperature, melter temperature, VS heater current, feed solids rate, and purge airflow) for Glycolic-remediated QMRF Testing.

One result of an observation that is difficult to understand is that of seemingly random drops in measured VS temperature after a period of relative stability when no variables were changed. One example can be seen in Figure 4-1 at approximately 2.2 hours; the QMRF measured VS temperature plot drops abruptly without any outside stimulus to the system. It is possible that overfeeding reached a tipping point where, due to thickness, radiation from the cold cap to the VS abruptly drops off, resulting in a drop in VS temperature, but there is no direct evidence of this phenomenon.

Comparing the QMRF measured VS temperatures of the unremediated feed test to that in the SMRF at similar measured VS temperatures, Figure 4-4 shows the QMRF has more overall fluctuation in the measured VS temperature as well as the unexpected drop. The two steady state regions for the QMRF test are highlighted in purple (from 0.3 to 1.6 hours and 1.8 to 2.9 hours, respectively) while the one displayed steady state region from the SMRF test is highlighted in orange (from 2.0 to 4.2 hours). The steady state regions overlap from the 2.0 to the 2.9 hour marks and feeding to the QMRF was stopped at the 3.5 hour mark. The time on the x-axis only represents duration for the SMRF data; it is not correlated to the time of the overall test.



**Figure 4-4. QMRF and SMRF VS Temperature Stability Comparison.**

In general, the measured VS temperatures in the SMRF were steadier than in the QMRF at similar conditions. This could be explained by the scaling effect in that with a smaller system, changes are less dampened and appear sharper. This scaling effect could also explain the temperature drop if some sudden internal change occurred independent of the external controls (for example, a sudden melt pool temperature inversion, the collapse of a bubble under the cold cap, etc.).

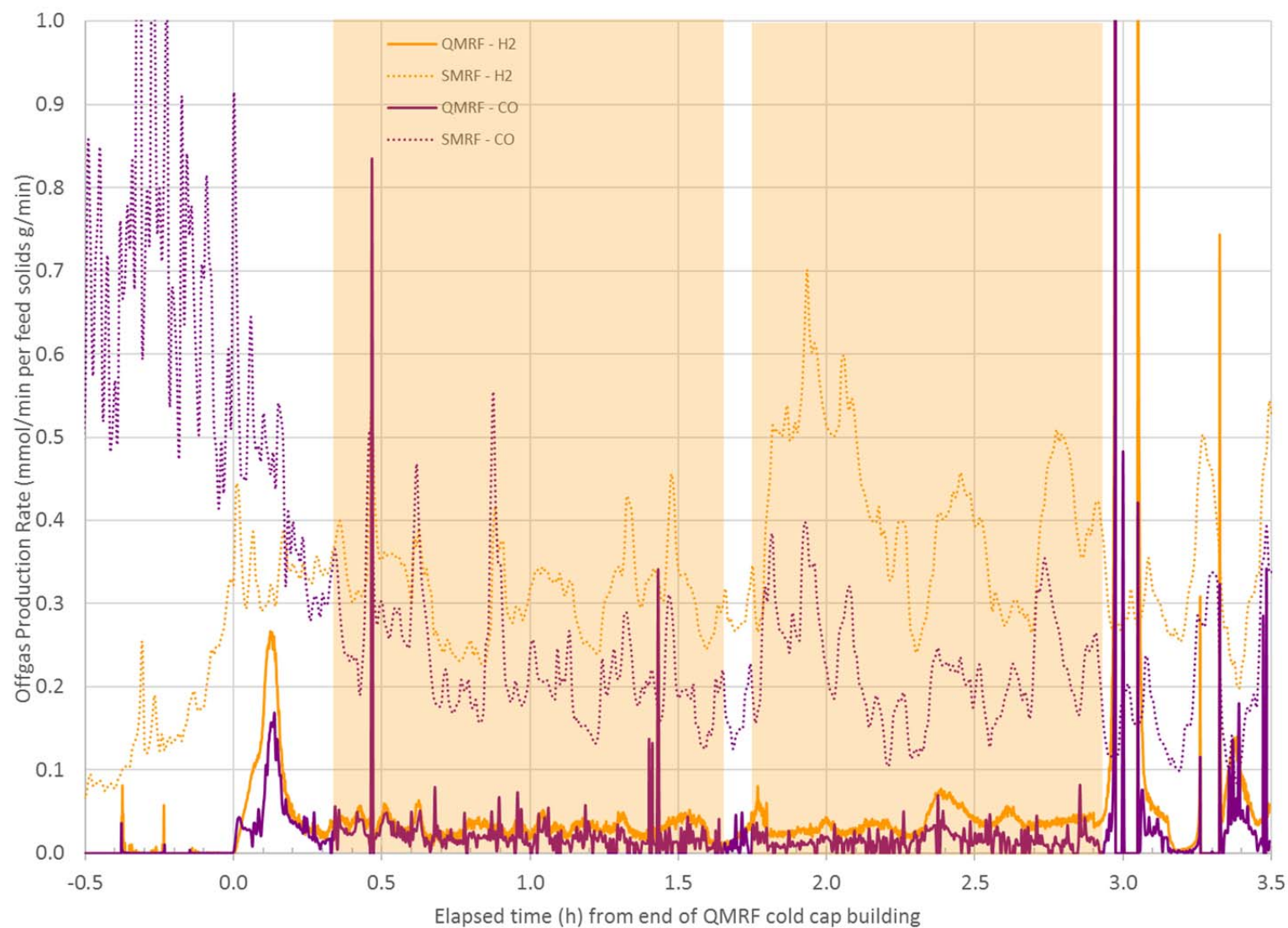
It is important to state that these VS temperatures are the measured values from the thermocouple located in the vessel and not the actual offgas temperatures ( $T_{\text{actual}}$ ). The VS thermocouple registers both the temperature of the gas in the vapor space as well as any radiant shine from the melt pool or vessel. This situation applies to all melters and corrections to  $T_{\text{actual}}$  are geometry dependent and require an energy balance calculation. To be able to perform the energy balance calculation, a second purge and thermocouple located in the offgas stream just off the melter (i.e., isolated from any radiant shine) would be required. Therefore, the melter offgas flammability correlations for  $T_{\text{actual}}$  vs. measured vapor space temperature ( $T_{\text{measured}}$ ) could not be performed for the QMRF and it is unknown if the  $\text{H}_2$  or CO rates vs  $T_{\text{measured}}$  would be the same.

#### 4.2 Offgas Analyses

Offgas concentrations were corrected for the total offgas flow tracked by the included He tracer gas. The helium flows set on the mass flow controller and the helium concentration reading from the MS were used to calculate the total flow of offgas from the vessel. From the total offgas flow, the millimolar flow of each offgas component could be calculated based on the concentrations measured by either the MS or FTIR.

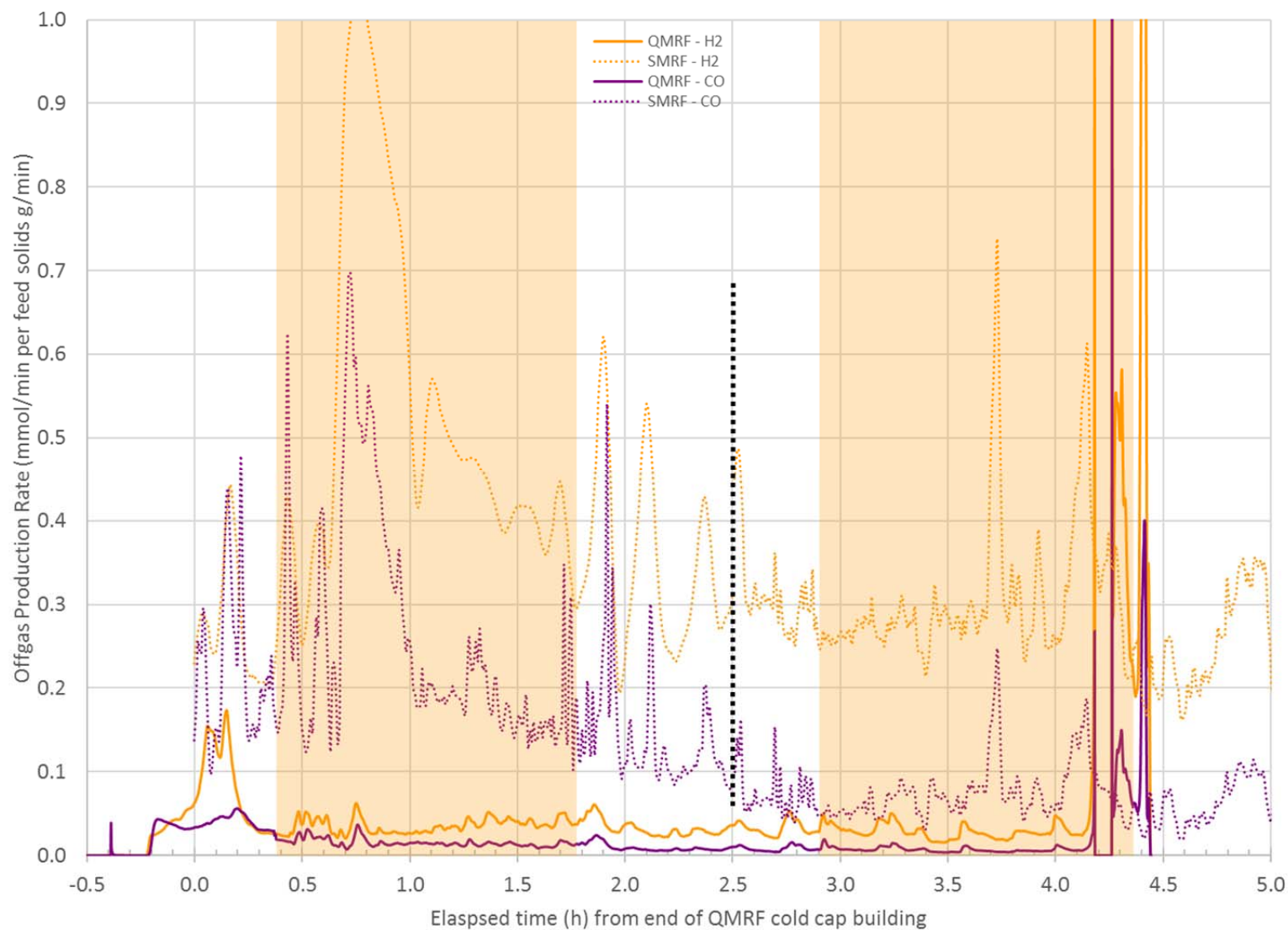
In typical melter offgas flammability studies, two of the major offgas components of concern are hydrogen and carbon monoxide. Figure 4-5 thru Figure 4-7 display the production rates (mmol gas / g feed solids) of hydrogen and carbon monoxide in the offgas streams as a function of time during each test. The periods of time best representing steady states in the QMRF are highlighted; the majority of the SMRF data plotted is associated with steady states.

The stability of the offgas concentrations varied due to periodic fluctuations in the feedrate. Since the periodicity of the pumping fluctuations was so much longer than any variation in the offgas rates, the offgas rates plotted during the steady states was done per the average feedrates for those periods. The rates of production for the more reduced gases, hydrogen and carbon monoxide, were slightly higher for the more reduced feeds than the more oxidized nitric-remediated feed in both the QMRF and SMRF. The increase in  $\text{H}_2$  and CO for the glycolic-remediated feed is related to the reducing nature of the feed; the more reduced a feed is, the more the chemical reactions in the cold cap are shifted towards production of reduced species, such as  $\text{H}_2$  and CO.



\* Superimposed SMRF data displayed from 500°C steady state test; QMRF data from 517 and 468°C steady state tests (left to right)

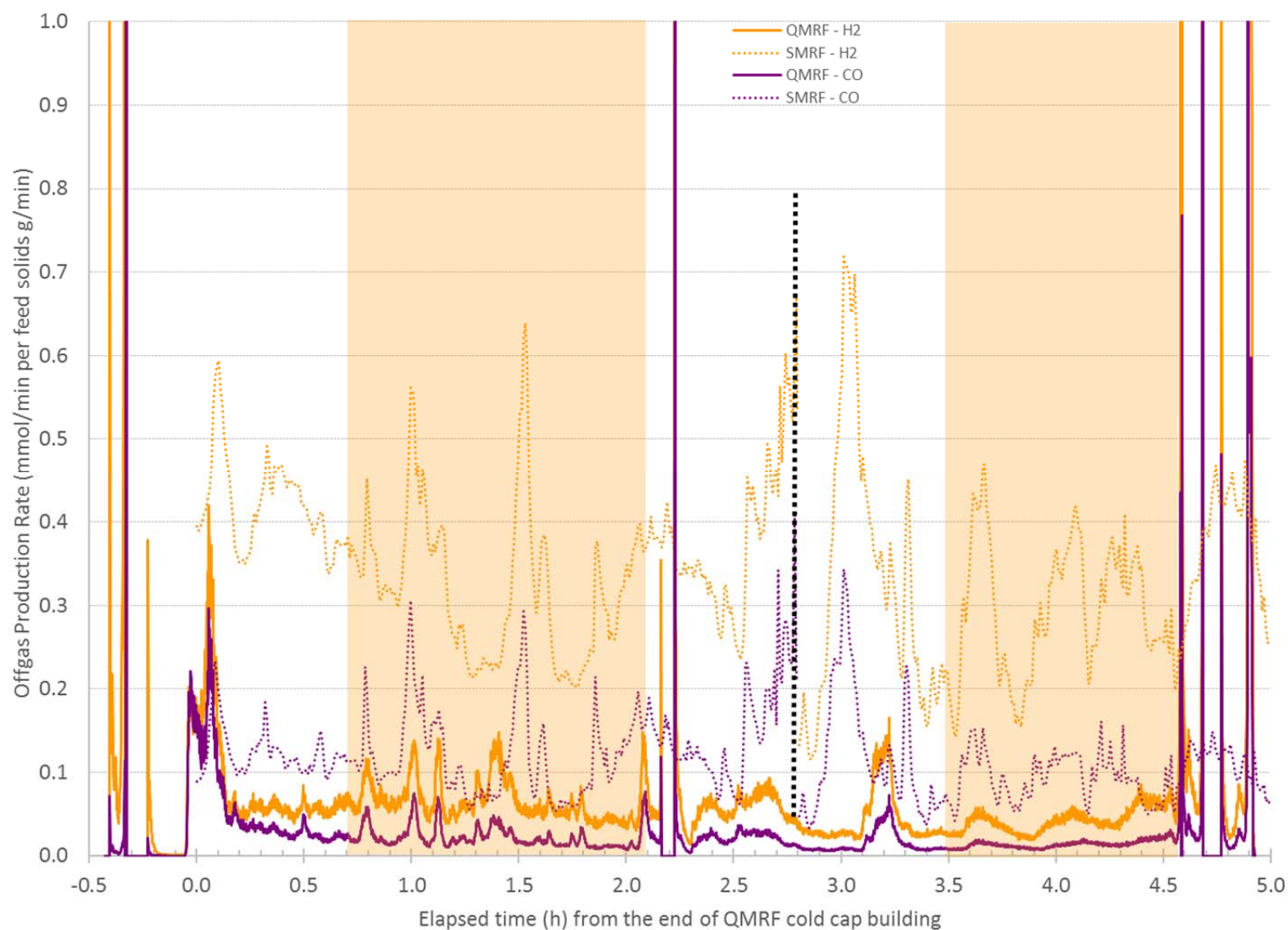
**Figure 4-5. Offgas Production Rates of H<sub>2</sub> and CO for QMRF and SMRF Testing with Unremediated Feed.**



\* Superimposed SMRF data displayed from separate 496 and 361°C steady state tests and separated by a vertical dashed line; QMRF data from 509 and 339°C steady state tests (left to right)

**Figure 4-6. Offgas Production Rates of H<sub>2</sub> and CO for QMRF and SMRF Testing with Nitric-remediated Feed.**





\* Superimposed SMRF data displayed from separate 421 and 324°C steady state tests and separated by a vertical dashed line; QMRF data from 459 and 282°C steady state tests (left to right)

**Figure 4-7. Offgas Production Rates of H<sub>2</sub> and CO for QMRF and SMRF Testing with Glycolic-remediated Feed.**



### 4.3 Glass Composition and REDOX Analyses

Measured glass compositions were compared to the compositions calculated from the feed and frit added masses.<sup>2</sup> Comparisons of these compositions are in Appendix B. The majority of deviations from the target composition were within acceptable ranges ( $\pm 10$ -15%) and suggest that feed segregation during testing did not occur.

Glass REDOX measurements for each test are compared to the model predicted values and values from the MRF and SMRF testing in Table 4-2.<sup>2</sup> The cold cap isolated the bulk molten glass from the oxidizing purge air, allowing reactions to occur at the cold cap-melt pool interface just as they would in an actual melter, a MRF beaker, or the conventional closed (sealed) crucible REDOX testing method.

**Table 4-2. Glass REDOX Measurements**

Feed remediation	Fe <sup>2+</sup> /ΣFe			
	QMRF measured	Avg. MRF measured	Avg. SMRF measured	Model predicted <sup>10</sup>
Unremediated	0.16 – 0.17	0.20	0.0	0.16
Nitric Remediated	0.05	0.07	0.0	0.03
Glycolic Remediated	0.30 – 0.41	0.59	0.0 – 0.12	0.27

The variability between sample measurements of the same test is small with the samples from the unremediated and nitric-remediated samples. The variability in the measurements from the glycolic-remediated samples is possibly due to sampling from a less reduced region of glass. A more oxidized region of glass in contact with one of the gaseous void spaces between the cold cap and the melt pool would result in a lower REDOX value compared to a volume not in contact with the void spaces.

The visual quality of each glass was acceptable according to the same criteria utilized to select samples from closed (sealed) crucibles for REDOX modeling. Each glass sample appeared macroscopically homogeneous with no visible crystallization. The colors were all dark brown-black with little visible tinting.

## 5.0 Conclusions

Evaluation of the QMRF platform as a lab-scale melter for comparison to the larger scale CEF and SMRF was performed on three separate REDOX remediations of the SB6I NG flowsheet feed. The data were compared to that acquired during 2016 SMRF testing.<sup>2</sup> Promising results from operation of the QMRF as compared to the operation of the SMRF and CEF included the ability to achieve similar vapor space temperatures across platforms, observation of comparable periods of operational stability while targeting a steady state, and production of glass that displayed REDOX values that corresponded much closer to those values predicted via the REDOX model equation. For the QMRF to act as a functional replacement for the SMRF or CEF, operation at steady states would need to be extended and additional temperature monitoring points would need to be added so that the data may be fully incorporated into the melter offgas flammability calculations.

The results of this testing demonstrated that the modified QMRF is operable in a continuously fed mode for the purpose of producing melter offgas data. Each QMRF experiment was performed during the

normal working hours of one day each and was supported by a crew significantly smaller than that required for operation of either the SMRF or CEF. Comparable operating conditions to the larger scale tests were achieved even in the shorter time frames. Similar steady state VS temperatures were examined with the QMRF as were examined in the 2016 SMRF testing with scaled purge air rates.

The feedrates, being near the lower operational and physical limits of the pumping system and the feed utilized, were not capable of being scaled relative to the SMRF. A set feedrate as close to scaled as physically feasible (i.e., sludge and frit did not segregate during feeding) with the current configuration was chosen and shown to operate sufficiently for this experimentation. However, at the lower VS temperatures tested, the feedrates could not be decreased such that steady state melting occurred, resulting in the rate of H<sub>2</sub> generation being lower than expected for a given feedrate (mmol H<sub>2</sub> / g feed solids is too low because feedrate is higher than melting and H<sub>2</sub> generation rate).

The VS temperatures were easily adjusted through manipulation of the VS heater power output and the purge airflow though the temperature was unstable at times. The seeming instability was not entirely unexpected as smaller volumes demonstrate more rapid responses to fluctuations in conditions. Where the larger SMRF and CEF have dilution effects in the vapor space to depress sharp changes in temperature, the smaller volume of the QMRF headspace is much more responsive giving the appearance of instability. Extending the operating time at each steady state could improve the overall observation of the temperature measurements even with the sharper responses.

QMRF testing provided sufficient data to perform preliminary comparisons of offgas speciation utilized for melter offgas flammability modeling efforts. Continuing the observed trend from SMRF and CEF testing, analysis of the QMRF offgas data showed lower concentrations of flammable gases (i.e., hydrogen and CO) produced from more oxidizing NG flowsheet feeds. However, counter to the trend in the SMRF, as the temperature for a given feed was lowered, the concentration of H<sub>2</sub> also dropped. The best current explanation for this observation is that the steady feedrate resulted in overfeeding to the cold cap, since it could not be scaled down any further with the current peristaltic pump configuration.

A significant difference from larger scale testing was in the measured REDOX values of the glass. The glass REDOX from the QMRF was significantly higher than from the SMRF and CEF melters, but was comparable to glass from MRF and crucible melts. The glass produced from the QMRF was sampled directly from the solidified melt rather than from a pour stream as in the SMRF and CEF. Had the SMRF and CEF been sampled from the melt pool, it is possible those glass samples will have shown similar REDOX values.

## **6.0 Recommendations, Path Forward or Future Work**

For additional or future testing, the following recommendations are suggested to improve the operation of the QMRF and its utility as a scoping melter tool:

- Operation of the QMRF at DWPF nominal conditions for a single test (i.e., not manually varying the VS temperature) to achieve a single steady state at typical operating conditions for a longer period of time. Extended single steady state testing could ensure that true steady state conditions have been achieved as well as provide true melt rate analyses for the NG flowsheet feed.
- Develop a feed system that can operate at low feedrates of about 1 g/min so that steady state operation can be achieved at lower temperatures. Optimization of a slurry feed pump system that can operate at benchtop scales without segregation of the sludge and feed solids would

allow for more accurate comparisons to larger scale platforms in terms of offgas production as a function of feed solids added to the melter.

- Modification of the QMRF vessel to allow for a deeper melt pool, glass pouring, or both to extend testing durations. Currently, the runtime of the QMRF is limited by the volume within the vessel to receive feed and convert to cold cap and glass. With a deeper vessel or the ability to pour the glass during testing, the operating limit can be extended to allow additional time to observe multiple steady states. It would also then be possible to compare the pour stream glass to the solidified melt pool glass in terms of REDOX and more directly compare to glasses produced in the SMRF and CEF.
- The QMRF platform would be an ideal system for testing and implementation of an *in situ* melt pool REDOX probe to track the progression of REDOX in the melt as a function of VS temperature, melt pool location, and residence time in the melter that could then be applied to larger melter systems.

## 7.0 References

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## **8.0 Appendices**

Appendix A. Supplemental Equipment Diagrams  
Appendix B. Supplemental Glass Analytical Data

## Appendix A. Supplemental Equipment Diagrams

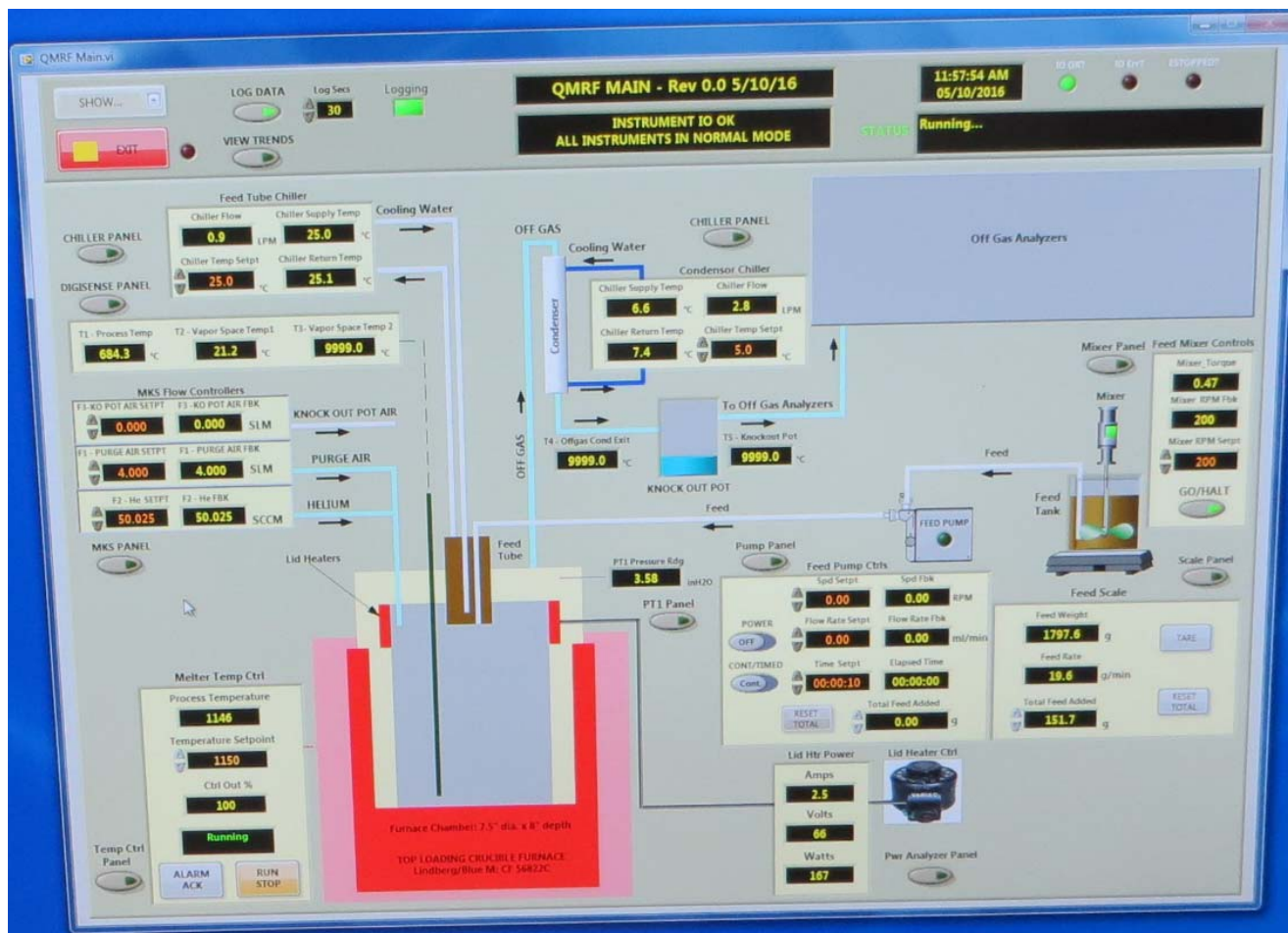


Figure A-1. QMRF Data Acquisition System (DAS) Display

**Table A-1. QMRF Instrumentation**

<b>Equipment description</b>	<b>M&amp;TE #</b>	<b>Cal. Date</b>	<b>Exp. date</b>	<b>Comment</b>
Digi Sense Temp Controller	TC DC6900	6/25/2014	6/25/2017	
Digi Sense Scanning Thermometer	GT3-T006	2/3/2016	2/3/2017	
Knock Out Pot Purge Air MKS	FC5K-13	4/11/2012	4/11/2017	5 slm; used in May 2016 run only
	FC500-4	10/17/2016	10/17/2018	500 sccm; used in Dec 2016 and Jan 2017 runs
Helium Flow MKS	FC500-12	3/23/2015	3/23/2017	500 sccm
Vessel Purge Air MKS	FC10K-11	4/10/2012	4/10/2017	10 slm
Melter Vapor Pressure	ITS PT001	4/11/2016	4/11/2018	
Feed Scale	ITS BL012	4/24/2014	4/24/2017	
Melter Vapor Space Thermocouple (TC#1)	ITS TC0062	3/15/2016	3/15/2019	
Knock Out Pot Thermocouple (TC#2)	ITS TC0063	3/15/2016	3/15/2019	

**Appendix B. Supplemental Glass Analytical Data**  
**Table B-1. Average of Analyzed Glass Compositions**

Major Glass Components Elements (Oxides)	Unremediated		Nitric-Remediated		Glycolic-Remediated		Predicted based on composition of sludge and frit	
	Elemental wt %	Oxide wt %	Elemental wt %	Oxide wt %	Elemental wt. %	Oxide wt %	Elemental wt %	Oxide wt %
Al (Al <sub>2</sub> O <sub>3</sub> )	4.86	9.18	4.82	9.11	5.19	9.80	5.04	9.53
B (B <sub>2</sub> O <sub>3</sub> )	1.45	4.66	1.31	4.22	1.30	4.17	1.59	5.12
Fe (Fe <sub>2</sub> O <sub>3</sub> )	7.22	10.32	7.36	10.52	7.79	11.14	8.04	11.49
Li (Li <sub>2</sub> O)	2.13	4.59	2.17	4.66	2.05	4.41	2.19	5.12
Mn (MnO)	2.17	2.80	2.26	2.92	2.52	3.26	2.44	3.15
Na (Na <sub>2</sub> O)	8.71	11.74	8.53	11.49	9.61	12.96	8.65	11.67
Ni (NiO)	0.75	0.95	0.81	1.03	0.80	1.02	1.08	1.37
Si (SiO <sub>2</sub> )	23.93	51.19	23.58	50.44	22.10	47.28	23.30	49.85



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