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U.S. DEPARTMENT OF
ENERGY

Prepared for the U.S. Department of Energy
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Pacific Northwest National Laboratory
Richland, Washington 99352

Executive Summary

Low-activity waste (LAW) stored in underground tanks on the Hanford Site in Washington State is planned to be filtered for solids removal and processed through ion exchange columns for cesium removal. These pretreatment steps will allow the waste to be transferred to the Hanford Tank Waste Treatment and Immobilization Plant's LAW Facility for immobilization into glass. The liquid waste will be combined with glass-forming chemicals (GFCs) to form a waste feed slurry that can be fed to electric melters for vitrification.

The process of continuously converting the aqueous feed slurry into a melt is dynamic and includes multiple reactions, degassing, and dissolution processes that depend on heat from the melt below. In this conversion process, waste components are partitioned into one of two streams: glass and off-gas. Washington River Protection Solutions has requested processing information and chemical information associated with these waste products for actual waste from Hanford tank 241-AP-107 (referred to herein as AP-107). To acquire this type of information, a small-scale melter system was designed that would not require high volumes of input waste or the large resource commitment of a full-scale melter system, while also providing dynamic information that would be difficult to determine from batch reactions in a crucible system.

A continuous laboratory-scale melter (CLSM) has been designed to operate with a continuous feeding process while periodically pouring glass product and collecting off-gas. The CLSM vessel has been sized to collect the relevant process and chemical information from obtainable volumes of AP-107 waste samples. A total 8.6 L of actual AP-107 tank waste were received after filtering for solids removal and ion exchange for cesium removal. This volume of AP-107 waste was mixed with GFCs to form an estimated 11.8 L of melter feed slurry. A mass of 7.01 kg of glass product were poured from the CLSM vessel during 10.07 hours of charging the AP-107 melter feed, indicating that about 15.0 kg of melter feed was vitrified into glass. These production values and other processing results from the AP-107 waste vitrification in the CLSM system are shown in Table ES.1.

The CLSM system was also designed with the capability to fully divert the flow of off-gas produced in the CLSM vessel to a sampling line that could capture the volatile species of interest, such as technetium-99 (^{99}Tc). This novel sampling system avoided the difficulties of slipstream sampling and could be activated once the feeding reached a steady state within the CLSM vessel. Off-gas product samples captured via high-efficiency particulate air (HEPA) filters using this method, as well as selected glass product samples were sent to an analytical lab for chemical analysis. The calculated average retention of ^{99}Tc (mass of ^{99}Tc output in the glass per mass of ^{99}Tc input in the melter feed) during the off-gas sampling is shown in Table ES.1. The measured composition of the AP-107 glass product, compared to the target composition, is shown in Table ES.2, which shows that the desired glass composition was achieved from processing the AP-107 melter feed in the CLSM. The calculated recovery of ^{99}Tc in the entire CLSM system was approximately 91%.

Table ES.1. AP-107 CLSM Test Run Summary

Parameter	Result
Test Date and Feeding Duration, h	8/8/2018 10.07
Glass Produced, kg	7.01
Volume of Waste Received, L	8.6
Volume of Melter Feed Batched (estimated), L	11.8
Melter Feed Consumed (estimated), kg	15.0
Average glass production rate, kg m^{-2} d^{-1}	1477
Average bubbling rate, L m^{-2} min^{-1}	149
Average glass temperature, °C	1115
Average plenum temperature, °C	648
Average ^{99}Tc Retention, g-glass g-feed^{-1}	0.40

Table ES.2. Comparison of AP-107 Waste Glass Product with the Target Glass Composition

Metal Oxide Component	Simplified Measured Glass Composition	Simplified Target Glass Composition	% Difference Between Measured and Target Glass Composition
	wt%	wt%	%
Al_2O_3	6.1	6.2	2.2
B_2O_3	10.7	10.1	6.1
CaO	3.5	3.8	5.7
Fe_2O_3	5.9	5.6	4.4
MgO	1.5	1.5	2.8
Na_2O	19.3	17.8	8.7
SiO_2	44.9	46.9	4.2
TiO_2	1.5	1.4	3.7
ZnO	3.6	3.6	2.1
ZrO_2	2.9	3.1	6.3

Acknowledgments

The authors thank Jackie Ranger at the Southwest Research Institute for performing the chemical analysis of all radioactive and non-radioactive products from the continuous laboratory-scale melter system. We thank Renee Russell for reviewing all the data, calculations, and figures associated with this technical report and Carolyne Burns for performing the melter feed viscosity measurement and calculation. The authors gratefully acknowledge the financial support for testing of the continuous laboratory-scale melter system provided by Washington River Protection Solutions, LLC and project direction provided by Kristin Colosi.

Acronyms and Abbreviations

APEL	Applied Process Engineering Laboratory
CLSM	continuous laboratory-scale melter
DFLAW	Direct Feed Low-Activity Waste
GFCs	glass-forming chemicals
HEPA	high efficiency particulate air
HLW	high-level waste
IC	ion chromatography
ICP-AES	inductively coupled plasma atomic emission spectroscopy
ICP-MS	inductively coupled plasma mass spectrometry
ILAW	immobilized low-activity waste
LAW	low-activity waste
ORP	U.S. Department of Energy Office of River Protection
PNNL	Pacific Northwest National Laboratory
QA	quality assurance
R&D	research and development
RPL	Radiochemical Processing Laboratory
SBS	submerged bed scrubber
SwRI	Southwest Research Institute
VSL	Vitreous State Laboratory
WRPS	Washington River Protection Solutions
WTP	Hanford Tank Waste Treatment and Immobilization Plant
WWFTP	WRPS Waste Form Testing Program

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1.0 Introduction

The primary mission of the U.S. Department of Energy Office of River Protection (ORP) is to retrieve and process approximately 56 million gallons of radioactive waste from 177 underground tanks located on the Hanford Site. The Hanford waste tanks are currently operated and managed by Washington River Protection Solutions, LLC (WRPS). As part of tank farm operations, WRPS supports ORP's waste retrieval mission. An important element of the ORP mission is the construction and operation of the Hanford Tank Waste Treatment and Immobilization Plant (WTP). The WTP is tasked with separating the waste into low-activity waste (LAW) and high-level waste (HLW) fractions and immobilizing these fractions by vitrification. This requires the design, construction, and operation of large and technically complex one-of-a-kind processing, waste treatment, and vitrification facilities.

Vitrification technology was chosen to treat three types of waste: the HLW fraction of tank waste at the Hanford and Savannah River Sites, the LAW fraction of tank waste at Hanford, and potentially other defense waste streams such as the sodium-bearing tank waste or calcine HLW at Idaho National Laboratory. Joule-heated melters are being used at the Defense Waste Processing Facility and will be used at the WTP to vitrify tank waste fractions.

A tank farm pretreatment capability provides for the initial production of immobilized low-activity waste (ILAW) by feeding LAW directly from Hanford tank farms to the WTP LAW facility for immobilization of the waste into glass. Before the transfer of feed to the LAW facility, tank supernatant waste will be pretreated to meet the WTP LAW facility acceptance criteria (Bechtel 2015). The key process operations for treating the waste include solids filtration and cesium removal by ion exchange.

After pretreatment, glass-forming chemicals (GFCs) will be added to the pretreated LAW and the resulting slurry vitrified at the LAW facility to produce an ILAW product for disposal. As a result of the vitrification process, water, volatile waste components, and a portion of semi-volatile waste components are driven off into the off-gas treatment system. A large fraction of the waste components in the melter off-gas are captured in the off-gas condensate, which is then recycled to the melter after concentration in the Effluent Management Facility. Another option being evaluated for the Direct Feed Low-Activity Waste (DFLAW) flowsheet, is to grout the concentrated off-gas condensate for disposal.

A test program was established at Pacific Northwest National Laboratory (PNNL) to conduct scaled unit operation process steps with actual Hanford tank waste (Peterson et al. 2017). To facilitate this program, the Radioactive Waste Test Platform was established to allow for baseline and alternative flowsheets and unit operations to be tested in comparable tests where both the direct effect of changes and the downstream effects of changes could be evaluated. The initial waste received at the Radiochemical Processing Laboratory (RPL) was from tank 241-AP-105 (hereafter called AP-105). The AP-105 waste was filtered for the removal of solids (Geeting et al. 2018a), it underwent ion exchange for the removal of cesium (Fiskum et al. 2018), had GFCs added and was vitrified in a continuous laboratory-scale melter (CLSM) (Dixon et al. 2018), and the condensate produced from vitrification was concentrated and converted to a non-glass waste form based on Cast Stone (Cantrell et al. 2018).

A second waste portion was received at the RPL, the supernatant from Hanford tank 241-AP-107 (hereafter called AP-107). This AP-107 waste went through solids removal by filtration (Geeting et al. 2018b) and cesium removal by ion exchange (Rovira et al. 2018); this test report describes the results from the vitrification of the treated AP-107 waste in the CLSM in RPL. The vitrification test was performed in August 2018, during which glass product and off-gas effluents, both gaseous and liquid, were collected for elemental analysis.

A total 8.6 L of AP-107 waste were present after ion exchange. The Kim et al. (2012) model for WTP baseline glass formulation was used to calculate the mass of GFCs to be added to the AP-107 waste to form the AP-107 melter feed slurry. While processing the AP-107 melter feed in the CLSM, the operating parameters and average glass production rate achieved were comparable to those of the AP-105 melter feed processing in the CLSM as well as those of similar LAW melter feeds processed in a similar scaled melter system operated by the Vitreous State Laboratory (VSL) at the Catholic University of America in Washington, DC (Matlack et al. 2010, 2017, 2018).

The purpose of the test described in this report was to support the vitrification portion of the test program to simulate a complete scaled DFLAW process (Peterson et al. 2017). It also served to help establish comparison between the processing performance of AP-107 waste and that of AP-105 waste in the same CLSM system. Ultimately the CLSM has demonstrated the ability to support future WTP programmatic needs regarding cold-cap behavior, glass processing operations, and an understanding of Tc volatility into the off-gas.

1.1 Quality Assurance

The work described in this report was conducted with funding from WRPS contract 36437/212, *DFLAW Radioactive Waste Test Platform*. This contract was managed under PNNL Project 71274. All research and development (R&D) work at PNNL is performed in accordance with PNNL’s Laboratory-Level Quality Management Program, which is based on a graded application of NQA-1-2000, *Quality Assurance Requirements for Nuclear Facility Applications*, to R&D activities. To ensure that all client quality assurance (QA) expectations were addressed, the QA controls of the WRPS Waste Form Testing Program (WWFTP) QA program were also implemented for this work. The WWFTP QA program implements the requirements of NQA-1-2008, *Quality Assurance Requirements for Nuclear Facility Applications* and NQA-1a-2009, *Addenda to ASME NQA-1-2008*, and consists of the *WWFTP Quality Assurance Plan* (QA-WWFTP-001) and associated QA-NSLW-numbered procedures that provide detailed instructions for implementing NQA-1 requirements for R&D work.

The work described in this report was assigned the technology level “Applied Research” and was planned, performed, documented, and reported in accordance with procedure QA-NSLW-1102, *Scientific Investigation for Applied Research*. All staff members contributing to the work received proper technical and QA training prior to performing quality-affecting work.

2.0 Test Conditions

This section describes the CLSM system assembled in RPL, the preparation of the AP-107 waste melter feed slurry, and analysis methods for samples generated from the CLSM.

2.1 Test Objectives and Success Criteria

Testing supported programmatic objectives, functions, and requirements (Peterson et al. 2017) to vitrify waste samples into glass using the dynamic process in the CLSM to provide data on the processability of the waste and contribute towards confirming the fraction of semi-volatile waste components assumed to be partitioning into the off-gas system as well as develop lessons learned and optimization of operating parameters for testing on actual wastes. The results also aid in confirming assumptions necessary to refine flowsheet models.

The test objectives shown in Table 2.1 were satisfied with the CLSM system processing actual AP-107 tank waste melter feed slurry at RPL.

Table 2.1. Test Objective Success Criteria

Test Objective	Success Criteria	Result	Result Reference
Configure the CLSM system in the fume hood in RPL.	CLSM system modified and used for testing.	CLSM system successfully modified and used for AP-107 melter feed vitrification.	Section 2.5
Vitrify AP-107 tank waste while periodically pouring glass and collecting off-gas samples.	Operate the CLSM to vitrify as much of the AP-107 waste as possible while collecting glass and off-gas samples.	6.8 kg of glass were produced while glass was poured throughout waste feeding and 3 off-gas samples were collected.	Sections 3.1, 3.2, and 3.3
Collect process data to allow comparative studies to be performed between AP-107 and AP-105 tests.	Report the results of a comparative process study between the AP-107 and AP-105 tests.	Process data were collected and reported from both the AP-107 and AP-105 and key comparative analysis was performed.	Sections 3.2 and 4.6
Collect vitrified glass samples.	Collect vitrified glass samples during the processing of feed slurry.	Collected samples of glass product from 25 glass pours.	Section 3.2
Collect samples of melter off-gas semi-volatiles with the sampling loop filters.	After the melter has been operating at steady state, collect melter off-gas samples in the off-gas sampling loop while at steady-state feeding.	Collected 3 off-gas samples when the CLSM reached steady state feeding.	Section 3.2

Table 2.1. Test Objective Success Criteria (cont.)

Test Objective	Success Criteria	Result	Result Reference
Collect melter off-gas condensate from the total run.	Collect all off-gas condensate during the processing of feed slurry.	Collected all off-gas condensate during feeding.	Section3.2
Analyze samples of melter feed, glass, off-gas particulate, and condensate.	Complete the analysis of samples identified as representing ideal test conditions	Samples of glass product, feed slurry, high-efficiency particulate air (HEPA) filters, condensate, and other liquids were sent for chemical analysis; results were received, reviewed, and reported (herein).	Sections 3.4

2.2 CLSM System

AP-107 waste was processed in the CLSM system, which was designed to collect samples of glass, off-gas solids, and off-gas condensate without upsets to the operation. A simplified flow diagram of the system is shown in Figure 2.1.

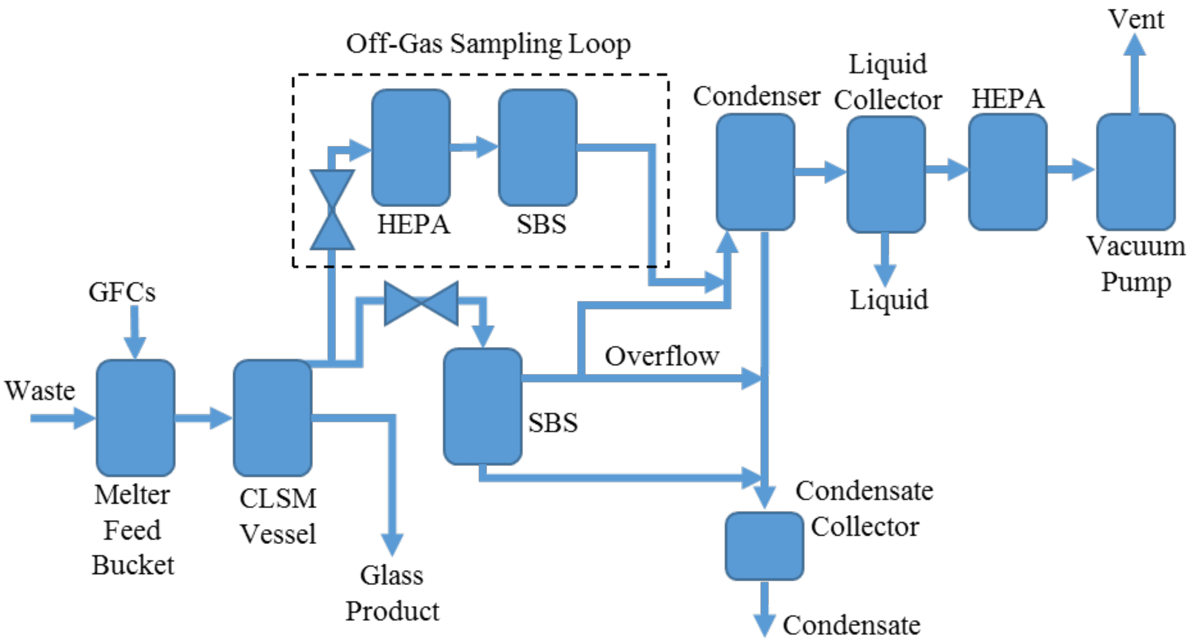

Figure 2.1. Simplified flow diagram of the CLSM system.

The total volume of AP-107 waste prepared for processing after solids and cesium removal was 8.6 L. GFCs were added to the waste, resulting in an estimated 11.8 L of AP-107 melter feed. The melter feed was mechanically agitated throughout testing and pumped to the melter using a progressing cavity pump through a water-cooled feed tube, producing a continuous dripping feed to the melter at a controlled rate.

Feed rate was adjusted based on processing conditions such as the bubbling rate but targeted between the WTP baseline processing rate of 1500 kg-glass m^{-2} d^{-1} and the optimized rate demonstrated with AP-105 simulant melter feed of 2000 kg-glass m^{-2} d^{-1} (Matlack et al. 2017).

The melter feed was converted to glass by processing in the CLSM vessel, which had a cross-sectional surface area of 0.0113 m^2. The CLSM vessel was fabricated into an octagonal cross-sectional design with an equivalent diameter of approximately 12.0 cm (4.7 inches) using an Inconel 690 plate. The glass inventory in the CLSM vessel was approximately 2.0 kg, resulting in a glass melt pool depth of ~6.4 cm (2.5 inches).

The lid of the CLSM vessel contained seven ports: two for thermocouples, one for an air bubbler, one for the feeding tube, one for a sight glass into the melter, one for the connection to the off-gas system, and one for pressure relief as seen in Figure 2.2. Heat was supplied externally to the CLSM vessel by a surrounding furnace. The hot zone of the furnace was located below and around the glass melt pool while the off-gas head space, called the plenum, area of the CLSM vessel was surrounded by insulation. The CLSM achieved continuous operation by periodically pouring glass out of the melt pool to a glass discharge box below the CLSM vessel. Pouring was achieved by lowering the vacuum maintained on the CLSM vessel by the off-gas system, which allowed glass to exit near the bottom of the melter, rise through a discharge riser, and pass over an overflow weir.

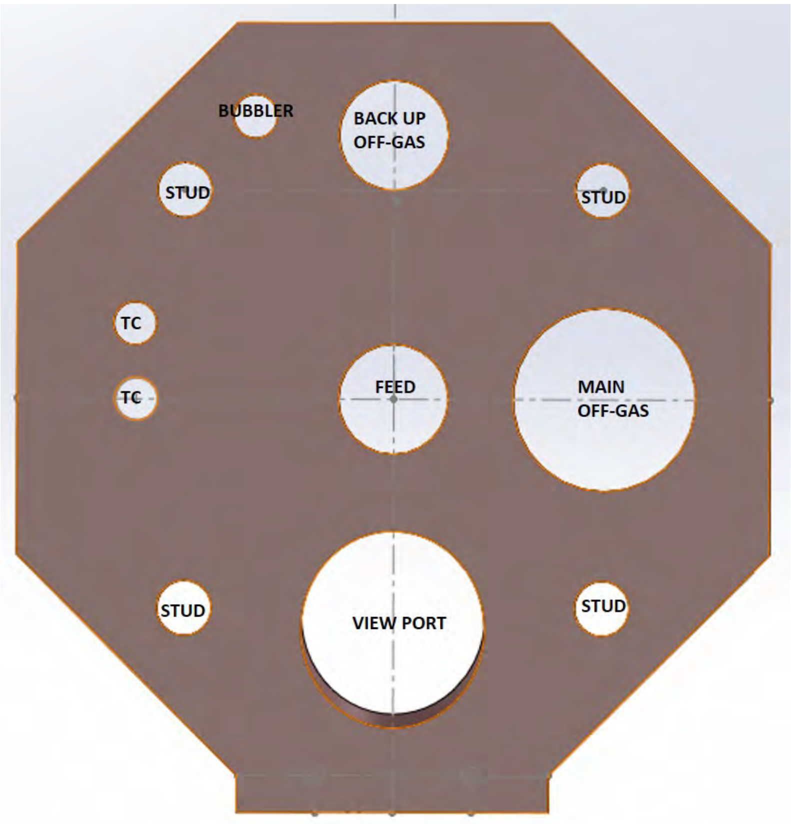

Figure 2.2. CLSM vessel lid and identified ports.

The off-gas produced by the conversion of melter feed to molten glass was drawn off from a port in the CLSM vessel lid into the off-gas system with a vacuum pump. Except when the off-gas stream was sampled, the off-gas would flow through the primary off-gas system, which consisted of a submerged bed scrubber (SBS), a condenser, a pre-HEPA liquid collector, and a HEPA filter. The SBS and the condenser worked together to both cool the off-gas, causing condensation of steam and other condensable gases, and perform scrubbing to remove the soluble gases and aerosols. The cool condensate from the condenser drained into a collector. Liquid from the SBS overflowed into the same collector where it could be drained periodically. Off-gas from the condenser passed through a collector, that allowed any remaining liquid to accumulate before the HEPA filter captured any remaining difficult-to-remove particulates. After HEPA filtration, the off-gas was released to the fume hood ventilation system.

The total off-gas stream could be sampled by diverting the full off-gas flow through a sampling loop consisting of heated HEPA filters followed by an SBS. This sampling train consisted of three parallel banks of two HEPA filters each. Each bank was available for discrete sampling evolutions. The sampled off-gas stream was then released back into the primary off-gas system before the condenser. Sampling of the total off-gas stream avoided the inherent issues with off-gas piping geometry and design with slip-stream sampling and ensured that the sample was representative. Off-gas sampling durations were 10-30 minutes until the filters became impassable.

2.2.1 System Configuration

The CLSM apparatus consisted of commercially available as well as custom parts. The system consisted of a melter feed mixing system, feed delivery system, small-scale Inconel 690 CLSM vessel with surrounding furnace, melter bubbler tube with flow meter, off-gas treatment and condensate collection system, computer-controlled data acquisition and melter control system, and associated ancillary equipment. An image of the CLSM system layout in RPL is shown in Figure 2.3.

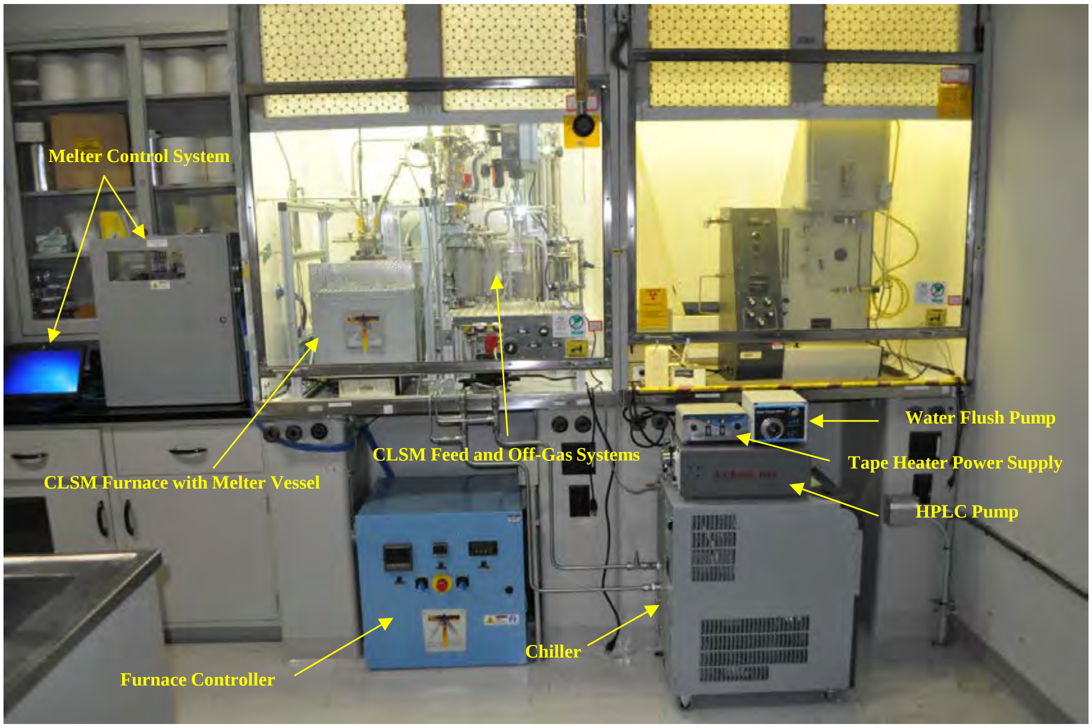

Figure 2.3. CLSM system layout in RPL fume hood.

2.2.2 Melter Operation

The CLSM was operated to maintain a glass melt pool temperature of 1150 °C (± 30 °C) by manually adjusting the control temperature of the surrounding furnace as necessary. During feeding operations, AP-107 melter feed was charged from the slurry bucket through the progressing cavity pump onto the glass melt surface in the CLSM vessel. The reacting melter feed formed a batch blanket, called a cold cap, on the glass melt surface, where the feed was heated and converted to glass (Dixon et al. 2015). The target glass production rate was between 1500 and 2000 kg-glass m^{-2} d^{-1} and was controlled by adjusting the feed rate and bubbling rate to maintain plenum temperatures of 450-650 °C and target cold-cap coverage over the glass melt surface of 75-95%. The cold-cap coverage was determined from visual observation through the CLSM vessel lid viewport. The CLSM briefly did produce glass melt pool and plenum temperatures above and below the target range. Typical of slurry-fed melters, the plenum temperature and cold-cap coverage were influenced by other factors, including feed composition and feed concentration.

2.2.3 Off-gas Operation

The condenser in the primary off-gas system was operated with chilled water and the condensate drained periodically from a collector vessel. The SBS level was maintained by overflow so that the pressure drop across the SBS remained relatively constant.

In the off-gas sampling loop, the line from the CLSM vessel to the HEPA filters was heat-traced to maintain elevated temperature and prevent/reduce condensation prior to the SBS.

The off-gas system vacuum pump was operated such that it pulled a vacuum on the CLSM vessel during feeding operation. The nominal operating vacuum pressure was 2–4 in-H_2O. The CLSM vessel vacuum was reduced periodically to pour glass. At the end of the run, the bubbler air and viewport purge were adjusted to increase the pressure in the melter, purging controlled volumes of glass from the CLSM vessel.

2.3 Melter Feed Preparation

The AP-107 waste was delivered in two portions, one of approximately 4.8 L and a second of approximately 3.8 L. The composition of the AP-107 waste after solids and cesium removal was analyzed by Rovira et al. (2018). The mass of each GFC to be added to the separate portions of the AP-107 waste was calculated by the glass models documented in Kim et al. (2012) to satisfy the WTP baseline requirements and the GFCs masses are given in Table 2.2. On August 7, 2018, the GFCs were batched at the Applied Process Engineering Laboratory (APEL), transported to the RPL, and combined in separate 2.5-gallon buckets with the separate portions of the AP-107 waste to make AP-107 melter feed slurry at a target glass yield of 699 g-glass L-feed^{-1}. The two buckets of the AP-107 waste were mechanically agitated in separate fume hoods during GFC addition and were continuously agitated from that time throughout CLSM operations.

The melter feed viscosity was measured with a Haake M5-RV20 (equipped with an M5 measuring head and RC20 controller) and an MV1 rotor and cup measuring system. Temperature control was achieved using a combination of the standard measuring system temperature jacket and a NESLAB Temperature-Controlled Recirculator, Model Number RTE 111. This recirculator allows heating and cooling of recirculation fluid to the rheometer over -25 to 150 ºC with a stability of ±0.01 °C. The melter feed slurry exhibited flow curve hysteresis indicative of breakdown of glass former particulate structure. The linearity of the down ramp over 0 to 500 s^{-1} suggests that the highly sheared melter feed is Newtonian with a viscosity of 10.05 mPa·s at 25 °C.

Chemical additions were performed to fulfill requirements of a Test Plan[1] prepared by PNNL and approved by WRPS and conducted according to a PNNL prepared Test Instruction[2].The target composition of the AP-107 glass is shown in Table 2.3. This AP-107 target glass composition is similar to the nominal glass formulation for AP-107 LAW without recycle used for small-scale melter tests by Matlack et al. (2018).

Table 2.2. GFCs Masses Added to Each Portion of AP-107 Waste

Component	GFCs Added to 4.8 L of Waste	GFCs Added to 3.8 L of Waste	Source
	g	g	
Kyanite	331.47	262.45	Kyanite Mineral Company
Boric Acid	821.20	650.23	Alfa Aesar
Wollastonite	371.57	294.21	NYCO Minerals
Iron Oxide	241.78	191.44	JT Baker
Lithium Carbonate	57.79	45.75	Foote Mineral Company
Olivine	136.26	107.89	Unimin Corporation
Silica	1683.42	1332.93	US Silica
Rutile Sand (TiO_2)	64.53	51.10	Chemalloy
Zinc Oxide	162.49	128.66	Zinc Corp. of America
Zircon Flour	206.76	163.71	Prince Minerals
Sucrose	288.20	228.19	C & H Sugar Company

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[1] TP-DFTP-047, Rev. 0.0, *DFLAW Test Platform Vitrification of Pretreated AP-107 Tank Waste Supernatant*. Pacific Northwest National Laboratory, Richland, Washington. (This internal PNNL document is not publicly available.)

[2] TI-DFTP-048, *DFLAW Test Platform Vitrification of AP-107 LAW Waste*. Pacific Northwest National Laboratory, Richland, Washington. (This internal PNNL document is not publicly available.)

Table 2.3. Target Composition of AP-107 Glass from AP-107 Tank Waste

Component	Wt%
Al_2O_3	6.12
B_2O_3	9.95
CaO	3.69
Cl	0.15
Cr_2O_3	0.08
Fe_2O_3	5.52
K_2O	0.47
Li_2O	0.50
MgO	1.49
Na_2O	17.49
NiO	0.01
P_2O_5	0.15
SO_3	0.37
SiO_2	46.08
TiO_2	1.40
ZnO	3.51
ZrO_2	3.01

2.4 Sample Analysis Methods

Process samples collected for analysis from the AP-107 waste run in RPL included the melter feed, glass product, off-gas condensate, HEPA filters, and SBS liquids (the SBS liquids samples contained only the liquids from the final capacity of each SBS since, during operation, the SBS liquids would overflow into the condensate collector). Table 2.4 identifies the sample matrices, size of sample to be sent for analysis, target frequency of sample collection during CLSM operation, and the desired analyses to be conducted. Table 2.5 lists the methods for the analyses listed in Table 2.4.

Table 2.4. Process Samples, Sampling Frequency, and Sample Analyses

Sample Matrix	Size	Frequency for Obtaining Samples	Analyses
Melter Feed Slurry	≥10 mL	Beginning of each feeding segment.	Cation Anion Isotopes
Glass Product	≥10 g	From every glass pour (typically every 30 minutes during steady melter feed charging).	Cation Anion Isotopes Mass
Primary SBS Liquid	≥10 mL	At the end of testing.	Cation Anion Isotopes Mass pH
Sampling Loop SBS Liquid	≥10 mL	At the end of testing.	Cation Anion Isotopes Mass pH
Off-gas Condensate	≥10 mL	In conjunction with every glass pour or more frequently as needed.	Cation Anion Isotopes Mass pH
Pre-HEPA Liquid	≥10 mL	Every four hours during melter feed charging.	Cation Anion Isotopes Mass pH
HEPA Filters	Complete filters	At the end of testing.	Cation Anion Isotopes Mass

Table 2.5. Sample Analysis Methods for Process and Off-Gas Samples

Analysis	Sample Matrix	Analysis Method	Analysis Description
Cations	Solid or Liquid	ICP-AES or ICP-MS (^{99}Tc and Cs)	Al, Ba, B, Ca, Cd, Cr, Co, Cs, Cu, Fe, La, Li, K, Mg, Mn, Mo, Na, Ni, P, Pb, Si, Sr, S, Sn, ^{99}Tc, Ti, W, V, Y, Zn, and Zr
Anions	Solid or Liquid	IC or Ion-Specific Electrode	Chloride, Chromate, Fluoride, Nitrate, Nitrite, Phosphate, and Sulfate
Isotopes	Solid or Liquid	Alpha Spectrum	Am-241, Cm-242, Cm-244, Np-237, Pu-238, Pu-239/240, and Pu-244
Mass	Solid or Liquid	---	Weigh on scale
pH	Liquid	---	Compare with pH paper

ICP-AES = inductively coupled plasma atomic emission spectroscopy
ICP-MS = inductively coupled plasma mass spectrometry
IC = ion chromatography.

Although melter feed, glass, condensate, and other liquid samples were to be collected according to the frequency and schedule indicated in Table 2.4, only those samples considered the most representative of ideal test conditions were aliquoted and analyzed. The remaining portions of each analyzed sample were archived for additional future analysis or re-evaluation if desired. As such, eight glass samples were sent for analysis: the first pour after melter feed charging began, the pours after each of the three off-gas samples, the pours associated with each glass melt inventory turnover (three samples total), and the final pour. One sample from each bucket of AP-107 melter feed was sent for analysis. One sample each from the primary SBS liquid, sampling loop SBS liquid, and the pre-HEPA liquid was sent for analysis. The condensate poured after each off-gas sample (three samples total) and the condensate poured prior to melter feed charging being stopped were sampled and sent for analysis. The remaining condensate pours were combined into three separate volumes (all the condensate pours prior to the first glass melt inventory turnover, all the condensate pours after the first turnover and prior to the end of melter feed charging, and all the condensate collected after melter feed charging stopped) and each volume was sampled for analysis. Finally, the HEPA filters from each of the three off-gas samples and the two primary HEPA filters were sent for analysis. In total, 25 samples were sent for chemical analysis. All chemical analyses (cations, anions, and istopes) of the product samples from the AP-107 CLSM run were performed by the Southwest Research Institute (SwRI). The analytical method and elements measured by each method are given in Table 2.5. The pH levels of all liquid samples sent for chemical analysis were measured with pH paper over the 0-14 range.

2.5 System Modifications

As an improvement based on the performance of the CLSM for the vitrification of the AP-105 tank waste melter feed (Dixon et al. 2018), a system was designed to ease the transfer of slurry to the bucket in the CLSM fume hood from a bucket in an adjacent fume hood. During the AP-105 CLSM test, the transfer method employed was to pour melter feed from the second bucket into the in-use bucket. The pour method resulted in a heel of high-solids-content slurry remaining in the second bucket since the melter feed was not agitated during the transfer. The modified method used for the AP-107 melter feed transfer was to pump the slurry from the bucket in the adjacent fume hood into the in-use bucket in the CLSM fume hood. An image of the system used to perform the pumping transfer method is shown in Figure 2.4.

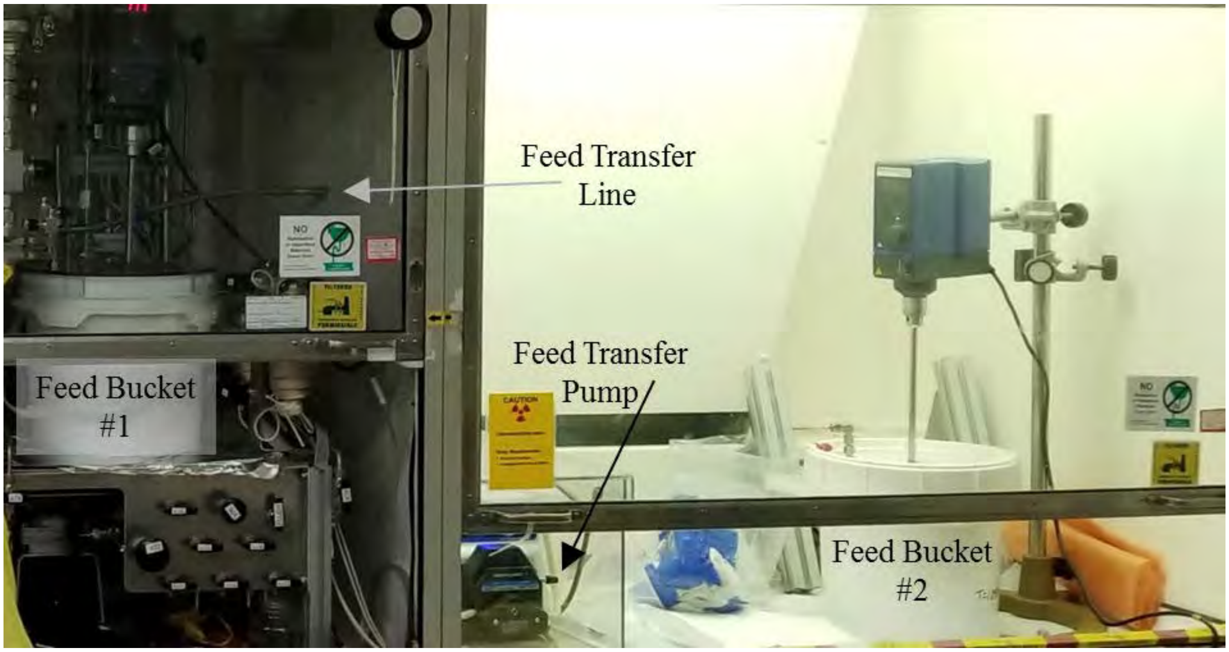

Figure 2.4. Pumping slurry system used to transfer meter feed from bucket #2 to bucket #1.

In the pumping transfer system, a flexible, braided stainless steel line connected feed bucket #2 to the feed transfer pump. This design allowed a lid to remain on the feed bucket, so it could be agitated continuously during transfer. The feed transfer pump was a peristaltic pump with the capability to adjust the transfer rate as needed based on the level of melter feed in bucket #1. An opening was created between the CLSM fume hood and the adjacent fume hood so the flexible stainless-steel line could be connected between the two hoods. The stainless-steel line allowed the slurry to drip into melter feed bucket #1. This transfer system succeeded in allowing the melter feed to be transferred, resulting in a small heel (< 2 cm) remaining in bucket #2 that did not appear to have an excess of solids content.

3.0 Run Description and Results

3.1 AP-107 Testing at RPL

The AP-107 CLSM test run was conducted at RPL on August 8, 2018. Testing was conducted to fulfill the Test Plan prepared by PNNL and approved by WRPS in association with a Test Instruction (see footnotes in Section 2.3). The target operating parameters for the run and are presented in Table 3.1.

Table 3.1. Target CLSM Operating Conditions

Parameter	Target
Target glass production rate, kg m^{-2} d^{-1}	1500-2000
Target feeding rate, kg h^{-1}	1.77-2.36
Bubbling rate, sccm	50-3000
Target glass melt temperature, °C	1150
Plenum temperature range, °C	450-650
Plenum vacuum normal operation, in-H_2O	-4
Off-gas temperature range, °C	< 500
SBS temperature, °C	15-30

The maximum steady-state processing rate was largely established by cold-cap conditions. During feeding operations, the target glass production range of 1500 to 2000 kg m^{-2} d^{-1} was controlled by adjusting the feed rate and bubbling rate to maintain the target cold-cap coverage of 75% to 95% of the surface of the glass melt. To achieve specific processing rates within the glass production range, target feeding rates were between 1.77 and 2.36 kg h^{-1}. Foaming in the high-viscosity transient connected region of the cold-cap had a significant effect on the target glass production range. Glass melt pool agitation using sub-surface air injection was employed to enhance melter feed processing rates. To accomplish this, a mass flow controller delivered air at 50-3000 sccm to a high-temperature 600 nickel alloy tube that was submerged in the CLSM vessel glass melt pool. The actual flow rate used within this range was chosen on based the operational stability and compatibility with other operational constraints such as melter plenum temperature and vacuum.

3.2 CLSM Processing Results

To begin the AP-107 test run, the CLSM vessel was loaded with 2.0 kg of previously prepared AP-107 glass (target composition shown in Table 2.3) pieces, prepared by processing non-radioactive AP-107 melter feed simulant in the CLSM located in APEL (substantially similar to the RPL CLSM but not radiologically contaminated). The furnace surrounding the CLSM vessel was heated from room temperature to 1250 °C at 10 °C min^{-1}. When the thermocouple located in the glass melt read 1150 ± 30 °C, feeding of the AP-107 melter feed into the CLSM vessel began. Feeding began at 11:18 AM and was concluded at 9:22 PM. The total feeding time, estimated mass of melter feed consumed, mass of glass produced, and average values over the course of feeding for several key processing components are given in Table 3.2. The mass of AP-107 melter feed could not be measured due to radioactive contamination restrictions; thus, the amount consumed during the CLSM run and the average feeding rate were estimated by calculating the mass of melter feed based on the target mass conversion ratio from AP-107 melter feed to glass (Matlack et al. 2018).

The glass temperature and plenum temperature during the test run are shown in Figure 3.1. The lower-than-expected reading of the glass temperature at the beginning of the run (from hour 0.00 to 0.16) was due to apparent electrical interference in the thermocouple wiring, since a secondary, separate reading of the glass thermocouple from a handheld device displayed glass temperatures in line with the expected trends of the values. The glass thermocouple was temporarily (~1 minute) disconnected from its lead and reconnected, after which the temperature of the computer reading aligned with the handheld device measurement. The temperatures at the start of the off-gas system, at the sampling switch, and of the primary SBS during the test run are shown in Figure 3.2. The effective glass production rate during the test run was calculated based on the total mass of glass produced and total feeding time reported in Table 3.2 and is shown in Figure 3.3. The melter vacuum values during the test run are shown in Figure 3.4. The bubbling rate during the test run is shown in Figure 3.5.

Table 3.2. CLSM Production Values for AP-107 Melter Feed Run

Parameter	Result
Test Date and Feeding Duration, h	8/8/2018 10.07
Glass Produced, kg	7.01
Volume of Waste Received, L	8.6
Volume of Melter Feed Batched (estimated), L	11.8
Melter Feed Consumed (estimated), kg	15.0
Average glass production rate, kg m^{-2} d^{-1}	1477
Average feeding rate (estimated), kg h^{-1}	1.49
Average bubbling rate, L m^{-2} min^{-1}	149
Average glass temperature, °C	1115
Average plenum temperature, °C	648
Plenum temperature range, °C	530–875
Plenum vacuum range, in-H_2O	0–5
SBS temperature range, °C	15–36

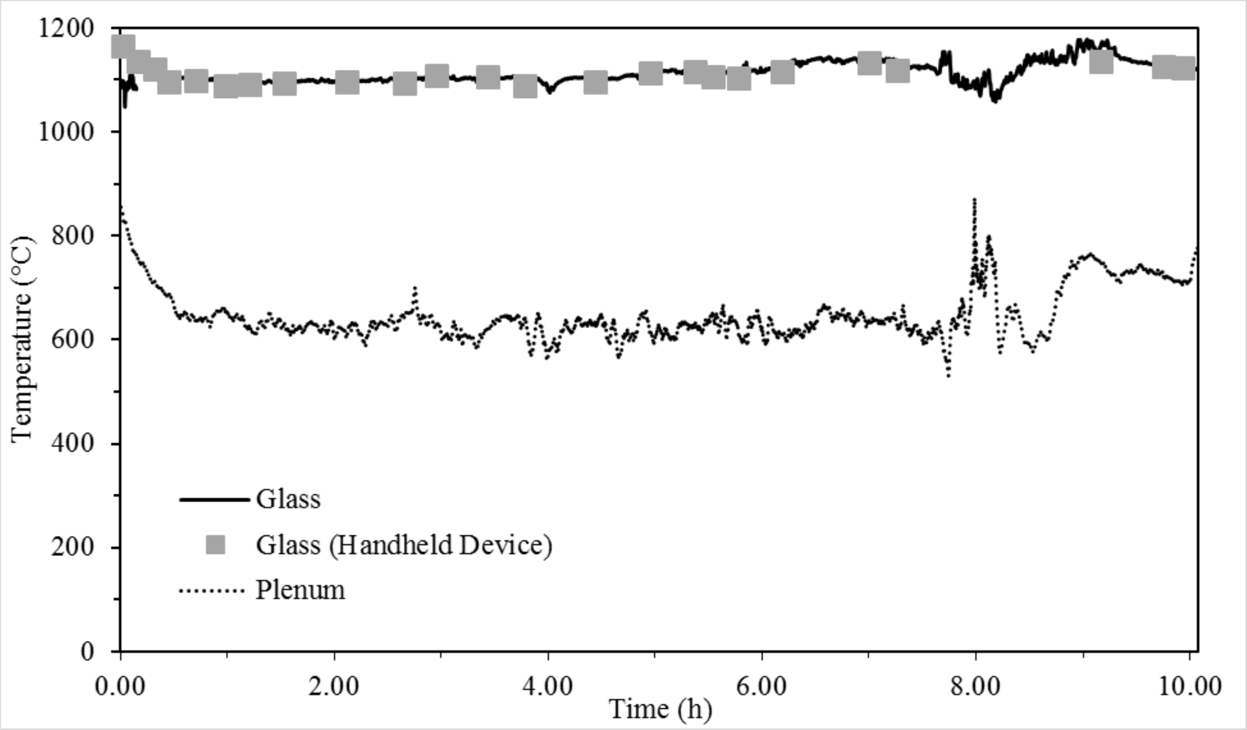

Figure 3.1. Glass and plenum temperature for AP-107 CLSM run.

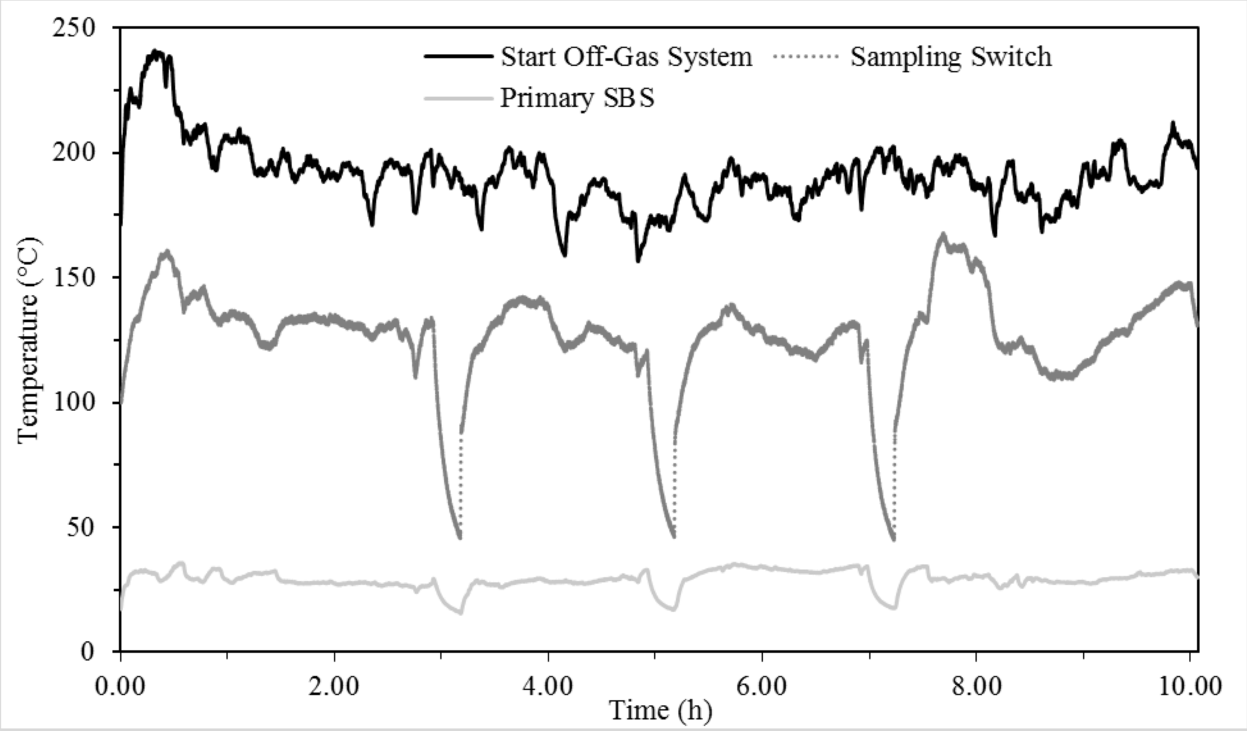

Figure 3.2. Start of off-gas system, sampling switch, and primary SBS temperature for AP-107 CLSM run.

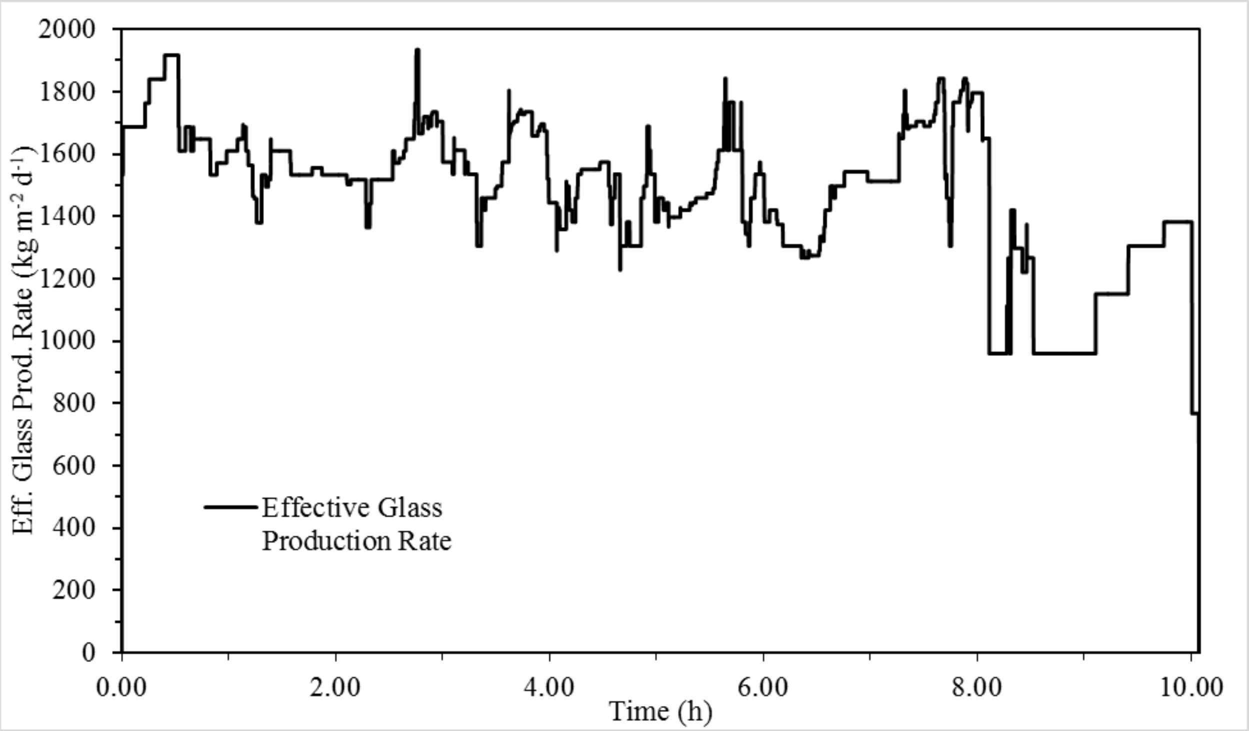

Figure 3.3. Effective glass production rate for AP-107 CLSM run.

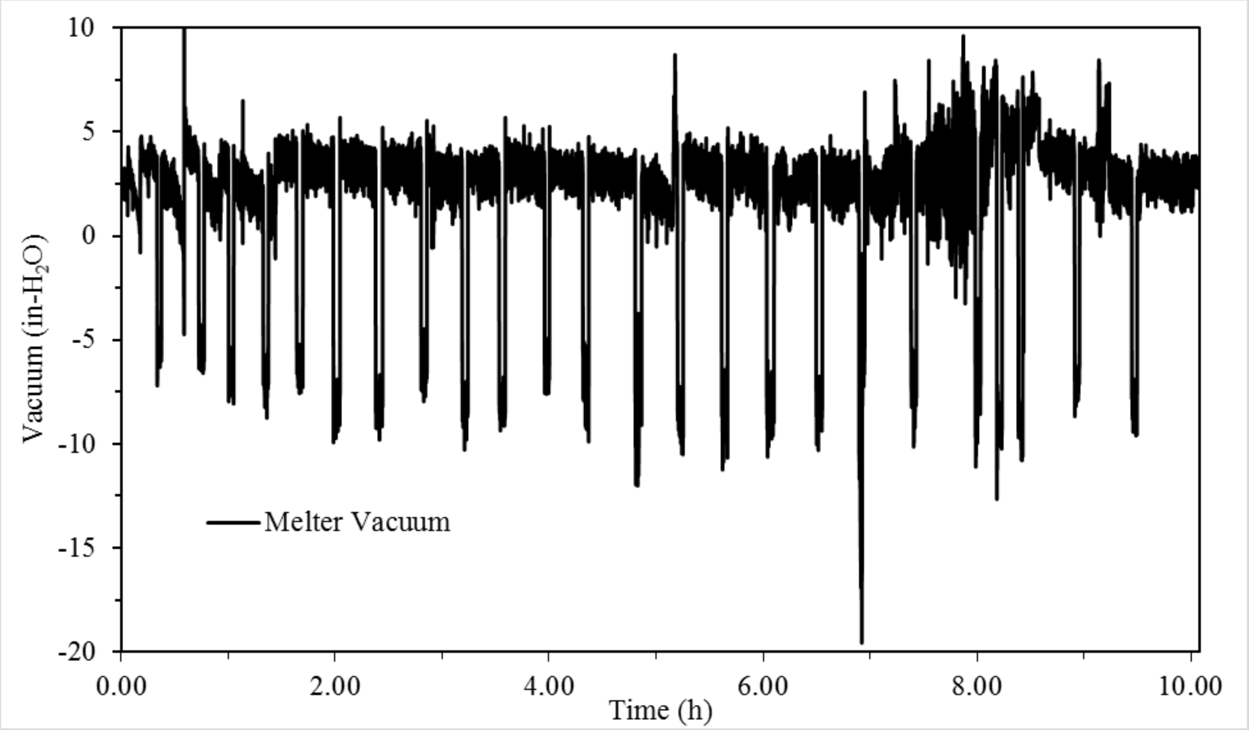

Figure 3.4. Melter vacuum for AP-107 CLSM run.

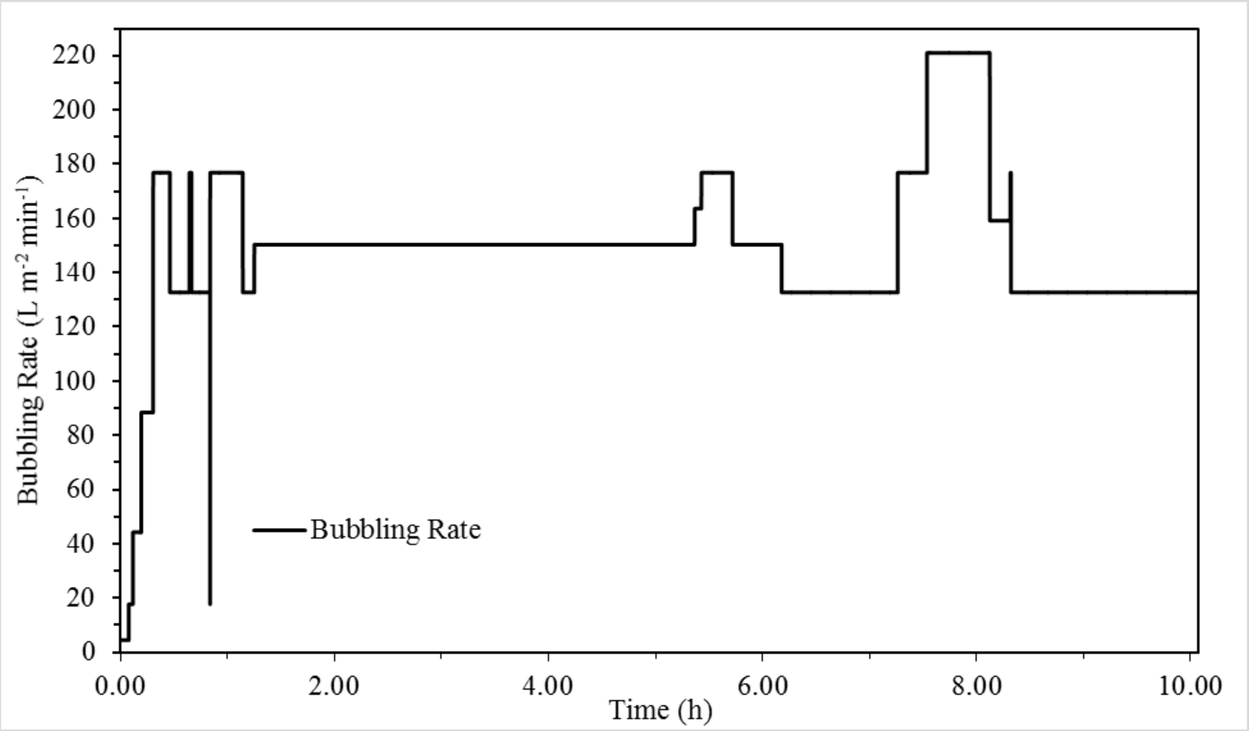

Figure 3.5. Bubbling rate for AP-107 CLSM run.

Three off-gas samples were collected during feeding. The timing and duration of each sample are listed in Table 3.3 and their occurrence in the timeline of the plenum temperature is shown in Figure 3.6. During these three times, the temperature at the off-gas sampling switch and primary SBS (Figure 3.2) decreased since off-gas was no longer flowing through them, but the temperature at the start of the off-gas system did not decrease because off-gas was still flowing past that spot in the off-gas system.

Table 3.3. Timing of Off-Gas Samples for AP-107 CLSM Run

Off-gas Sample Number	Date	Off-Gas Sample Start Time	Off-Gas Sample Start on Test Run Timeline	Off-Gas Sample End Time	Off-Gas Sample End on Test Run Timeline	Total Sampling Time
		Time of Day	h	Time of Day	h	min
1	8/8/2018	14:13	2.92	14:28	3.18	15.15
2	8/8/2018	16:13	4.93	16:28	5.18	15.08
3	8/8/2018	18:16	6.98	18:31	7.23	15.13

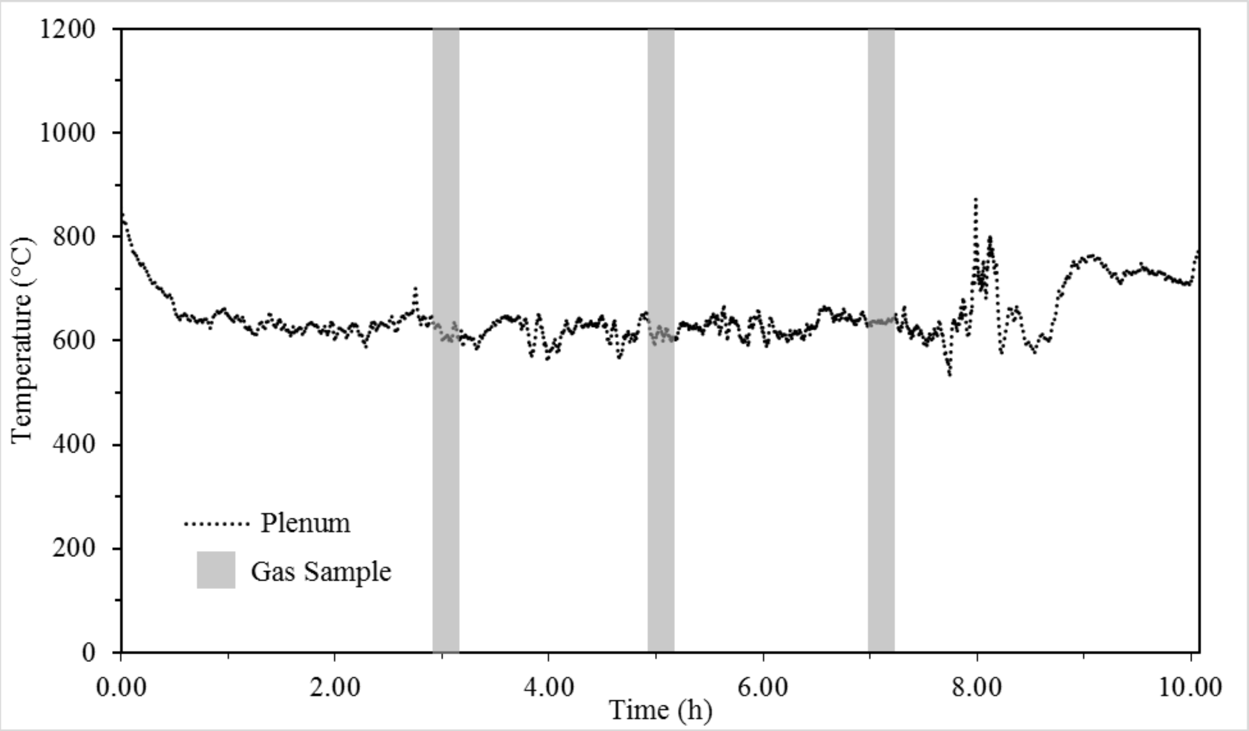

Figure 3.6. Plenum temperature as a function of time for AP-107 CLSM run, showing timing and durations of gas-sampling events.

The timing and mass of each periodic glass pour are shown in Table 3.4, which correspond with the times that the CLSM vacuum was set in the range of -5 to -10 in-H_2O in Figure 3.4. The cumulative mass of glass poured from the CLSM vessel revealed that the glass melt inventory (2.0 kg) was turned-over three times during the melter feed charging.

Table 3.4. Timing and Mass of Glass Pours from CLSM Run with AP-107 Simulant Feed Slurry

Glass Pour Time	Glass Mass	Cumulative Glass Mass
h	g	g
0.00	94.06	94.06
0.33	146.00	240.06
0.72	149.64	389.70
1.00	153.17	542.87
1.33	181.73	724.60
1.64	198.14	922.74
1.99	261.23	1183.97
2.38	279.50	1463.47
2.80	231.00	1694.47
3.19	286.50	1980.97
3.54	296.00	2276.97
3.90	219.50	2496.47
4.32	322.35	2818.82
4.81	385.50	3204.32
5.20	333.00	3537.32
5.61	287.50	3824.82
6.04	419.50	4244.32
6.50	357.29	4601.61
6.90	331.50	4933.11
7.38	265.00	5198.11
7.98	226.00	5424.11
8.18	251.00	5675.11
8.38	207.01	5882.12
8.90	262.50	6144.62
9.45	381.50	6526.12
10.07	2482.50	9008.62

3.3 Feed Processing Characteristics

From hour 0.00 to 0.56, the temperature of the plenum steadily dropped from around 850 °C to 650 °C, shown in Figure 3.1, while the cold cap was formed and observed to spread out to cover ~90% of the glass melt surface. After this time, from hour 0.56 to 7.68, the cold-cap coverage and plenum temperature were maintained at relative steady state by varying the feeding and bubbling rates as necessary. Similar small-scale melter systems, like the DM10 system (0.021 m^2 melter surface area and ~8 kg glass inventory) operated by the VSL at the Catholic University of America, reached this cold-cap steady state period after ~5 hours of melter feed charging (Matlack et al. 2017 and 2018). The relatively short period of time with which the CLSM can reach a cold-cap steady state may lead to less volatility of desired components from the glass melt during an equivalent time frame compared to other melter systems.

At around hour 7.68, the plenum temperature began to rapidly drop below ~630 °C, shown in Figure 3.1, necessitating an increase in the bubbling rate to 221 L m^{-2} min^{-1}. This increase in bubbling rate resulted in a decrease of the glass temperature and, for the remainder of the run from hour 7.68 to 10.07, the glass temperature fluctuated. The feeding rate had to be decreased to counter the glass temperature fluctuations and to attempt to maintain the glass temperature in the desired range of 1150±30 °C. The average glass temperature for the entire run was 1115 °C, which was ~5 °C outside of the desired range. It is recommended to procure a furnace with a higher heating capacity so that the external heating of the CLSM vessel can be increased to counteract the effect of the bubbling cooling the glass melt. At hour 10.07 (August 8, 2018, at 9:22 PM), the feeding line was flushed with water, the cold cap was allowed to burn off, all of the glass melt inventory was poured from the CLSM vessel, and the test was terminated. An estimated ~1 L heel of melter feed remained in each melter feed bucket after processing ended.

3.4 Sample Chemical Analysis Results

All the samples described in Section 2.4 were sent for chemical analysis. The pH level of each analyzed liquid sample was measured with pH paper and the results are listed in Table 3.5. The value for each sample mass and the resulting concentration of each analyzed cation and anion are given in Table 3.6. Where no numerical data are reported, the values were below the analysis detection limit.

Table 3.5. pH of Analyzed Liquid Samples from the AP-107 CLSM Run

Liquid Sample Names	pH
Condensate after HEPA 1	~1
Condensate after HEPA 2	~1
Condensate after HEPA 3	~1
Condensate End	~1
Condensate Combined 1	~1
Condensate Combined 2	~1
Condensate Combined 3	~1
Primary SBS Fluid	~5
Sampling SBS Fluid	~8
Pre-HEPA Collected Fluid	~7

Table 3.6. Chemical Analysis of Selected Samples for AP-107 CLSM Run

Sample Name	Sample Type	Sample Mass	Technetium-99	Cesium	Aluminum	Barium	Boron	Cadmium	Calcium	Chromium	Cobalt	Copper	Iron
		g	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}
Glass Pour 0.33	Glass	146.00	0.499	771	29400	422	28900	34.9	21100	2690	3.15	7.29	41300
Glass Pour 3.19	Glass	286.50	2.04	331	31200	196	31800	16.9	24200	2020	3.09	5.15	40500
Glass Pour 3.54	Glass	296.50	4.00	294	30700	178	31300	15.7	23800	1470	3.10	4.17	39200
Glass Pour 5.20	Glass	333.00	2.31	170	31800	125	31000	11.4	25000	1230	2.78	3.43	39500
Glass Pour 6.04	Glass	419.50	2.53	131	30800	100	30900	9.99	24700	1330	2.93	3.33	38700
Glass Pour 7.38	Glass	265.00	2.26	73.9	30900	79.7	30250	8.56	24900	929	2.94	3.40	38250
Glass Pour 9.45	Glass	381.50	2.48	39.2	30600	64.2	31600	7.52	24600	934	2.75	2.97	37400
Glass Pour 10.07	Glass	2482.50	2.53	109	29800	94	37900	9.88	24000	1160	2.59	3.16	37000
Melter Feed Bucket 1	Feed	n/a	2.64	--	5505	16.4	13950	2.60	11650	276	1.54	0.88	17400
Melter Feed Bucket 2	Feed	n/a	2.72	--	5500	15.4	14000	2.80	11800	267	1.60	1.01	17900
Sampling HEPA Filter 1	HEPA	16.50	20.5	287	11500	15150	12200	0.599	4570	91.2	--	1.10	222
Sampling HEPA Filter 2	HEPA	17.00	36.3	175	12000	15600	11000	--	4740	100	--	1.10	263
Sampling HEPA Filter 3	HEPA	16.50	30.8	85.0	11300	14600	10900	--	4420	70.1	--	0.689	209
Primary HEPA Filter 1	HEPA	10.50	9.31	513	12400	16000	11300	1.47	4860	107	--	1.06	176
Primary HEPA Filter 2	HEPA	22.00	46.6	153	3830	5080	4600	0.303	1520	156	--	0.694	121
Condensate after HEPA 1	Liquid	192.50	0.912	17.5	11.3	--	134	--	8.94	7.57	--	--	19.3
Condensate after HEPA 2	Liquid	175.50	1.26	16.0	13.4	--	168	--	11.3	6.65	--	--	27.8
Condensate after HEPA 3	Liquid	306.50	1.57	12.3	12.4	--	170	--	11.6	5.25	--	0.455	30.3
Condensate End	Liquid	191.00	1.72	8.41	10.1	--	168	--	10.9	3.95	--	--	8.34
Condensate Combined 1	Liquid	1791.50	0.786	16.8	14.2	--	155	--	9.6	13.0	--	--	16.5
Condensate Combined 2	Liquid	3026.00	1.52	12.5	11.5	--	165	--	11.3	5.12	--	--	13.4
Condensate Combined 3	Liquid	1391.00	2.01	7.54	15.1	--	205	--	16	4.99	--	--	15.8
Primary SBS Fluid	Liquid	1150.50	2.64	9.11	40.8	--	324	--	37	8.8	--	--	22.6
Sampling SBS Fluid	Liquid	1286.00	0.123	0.489	--	--	10.8	--	--	--	--	--	--
Pre-HEPA Collected Fluid	Liquid	561.00	1.67	11.6	13.4	--	182	--	12.5	5.41	--	0.388	15.4

Values marked with ‘--‘ denote that the metal concentration was below the analysis detection limit.

Table 3.6. Chemical Analysis of Selected Samples for AP-107 CLSM Run (cont.)

Sample Name	Lanthanum	Lead	Lithium	Magnesium	Manganese	Molybdenum	Nickel	Phosphorus	Potassium	Silicon	Sodium	Strontium	Sulfur
	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}
Glass Pour 0.33	3000	24.8	1490	8130	107	1070	3020	881	3420	200000	133000	316	1320
Glass Pour 3.19	1240	12.8	2000	8740	84	471	2340	832	3480	202000	136000	150	1660
Glass Pour 3.54	1100	12.3	2020	8480	80	420	2220	784	3480	200000	136000	137	1620
Glass Pour 5.20	687	9.57	2110	8770	76.3	279	1810	783	3450	201000	135000	98	1680
Glass Pour 6.04	503	8.69	2150	8660	73.1	219	1540	716	3480	195000	134000	79.5	1740
Glass Pour 7.38	333	7.54	2205	8635	69.8	159	1165	666	3470	198500	135000	63.5	1705
Glass Pour 9.45	189	6.63	2300	8200	66.9	109	792	679	3540	197000	138000	49.8	1760
Glass Pour 10.07	463	8.4	2260	7870	68.9	204	1200	722	3640	204000	144000	75.3	1740
Melter Feed Bucket 1	1.10	2.26	1030	4120	29.4	20.2	45.8	293	1540	97400	60300	14.7	866
Melter Feed Bucket 2	--	2.17	1010	4270	28.5	19.7	45.0	323	1530	95000	59500	14.3	837
Sampling HEPA Filter 1	--	2.71	38.5	657	7.67	35.2	7.42	--	9635	361000	36150	173	870
Sampling HEPA Filter 2	--	2.72	48.7	679	30.1	28.1	9.31	--	8790	357000	33600	178	841
Sampling HEPA Filter 3	--	2.48	44.6	633	7.47	23.3	4.92	--	8670	344000	32100	167	853
Primary HEPA Filter 1	--	2.72	22.6	696	4.47	49.9	3.52	--	9760	378000	32400	182	918
Primary HEPA Filter 2	--	1.03	59.8	220	9.29	13	4.01	--	4170	128000	22800	56.7	1480
Condensate after HEPA 1	--	--	2.95	--	--	0.512	0.747	--	28.0	33.5	484	--	31.3
Condensate after HEPA 2	--	--	3.99	--	--	0.74	0.582	--	33.2	39.8	598	--	38.3
Condensate after HEPA 3	--	--	4.29	--	--	0.726	0.470	--	36.1	35.3	623	--	36.7
Condensate End	--	--	4.46	--	--	0.629	0.283	--	37.5	26.2	625	--	40.1
Condensate Combined 1	--	--	2.85	--	--	0.760	1.41	--	31.5	38.0	577	--	35.1
Condensate Combined 2	--	--	4.69	--	--	0.594	0.473	--	38.7	36.9	672	--	37.0
Condensate Combined 3	--	--	5.77	--	--	0.708	0.297	--	46.1	48.7	778	--	52.5
Primary SBS Fluid	--	--	10.7	--	--	--	0.289	--	77.2	82.9	1280	--	96.6
Sampling SBS Fluid	--	--	--	--	--	--	1.51	--	--	--	16.2	--	--
Pre-HEPA Collected Fluid	--	--	4.77	--	--	0.673	0.539	--	39.9	30.2	673	--	40.6

Values marked with '--' denote that the metal concentration was below the analysis detection limit.

Table 3.6. Chemical Analysis of Selected Samples for AP-107 CLSM Run (cont.)

Sample Name	Tin	Titanium	Tungsten	Vanadium	Yttrium	Zinc	Zirconium	Chloride	Sulfate	Chromate	Fluoride	N (Nitrate)	N (Nitrite)
	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}
Glass Pour 0.33	10.2	9000	230	55.4	44.2	27700	20200	2.24	4.85	--	--	2.05	--
Glass Pour 3.19	13.9	8790	272	57.1	41.7	28300	20500	2.07	3.63	--	--	--	--
Glass Pour 3.54	15.3	8550	267	56.3	41.7	28000	20300	1.83	2.03	--	--	--	--
Glass Pour 5.20	15.3	8710	296	57.4	40.6	27900	20500	1.90	2.16	--	--	--	--
Glass Pour 6.04	16.1	8430	285	56.6	40.5	27800	20600	1.75	2.14	--	--	--	--
Glass Pour 7.38	16.4	8345	282	55.6	41.3	27900	20450	2.21	2.41	--	--	--	--
Glass Pour 9.45	18.9	7910	281	53	40.5	26600	19900	9.31	2.72	--	--	--	--
Glass Pour 10.07	15.2	7800	283	51.3	39.3	28500	19400	1.97	2.63	--	--	--	--
Melter Feed Bucket 1	5.11	475	118	3.20	3.30	11950	287	1250	2120	515	85.8	11500	7090
Melter Feed Bucket 2	5.24	511	116	3.22	3.34	12500	295	1250	2125	503	71.4	11550	7135
Sampling HEPA Filter 1	1.94	20.8	87.0	--	2.48	11200	115	8835	1580	52.8	174	617	82.4
Sampling HEPA Filter 2	--	21.1	92.7	--	2.49	11600	116	12700	2000	101	286	894	135
Sampling HEPA Filter 3	--	20.2	82.7	--	2.38	10800	111	10300	2310	97.5	283	463	344
Primary HEPA Filter 1	1.73	20.2	94.9	--	2.6	11800	119	27.9	3200	3.51	4.74	3610	--
Primary HEPA Filter 2	--	7.80	27.7	--	0.834	3710	40.1	4360	6720	--	86.1	38000	--
Condensate after HEPA 1	--	0.644	--	--	--	25.2	--	390	194	--	17.3	2000	--
Condensate after HEPA 2	--	0.773	--	--	--	31.5	0.795	468	200	--	20.6	2220	--
Condensate after HEPA 3	--	0.896	--	--	--	31.0	1.03	542	206	--	24.3	2630	--
Condensate End	--	0.790	--	--	--	26.3	0.915	590	206	--	28.0	2430	--
Condensate Combined 1	--	0.786	--	--	--	28.0	1.20	389	207	--	15.8	1710	--
Condensate Combined 2	--	0.750	--	--	--	28.8	0.881	529	213	--	25.0	2280	--
Condensate Combined 3	--	1.37	--	--	--	37.2	1.61	684	259	--	41.1	2400	--
Primary SBS Fluid	--	1.19	--	--	--	94.2	2.64	980	407	--	87.7	1530	--
Sampling SBS Fluid	--	--	--	--	--	--	--	17.6	5.78	--	--	107	480
Pre-HEPA Collected Fluid	--	0.795	--	--	--	32.9	0.828	683	236	--	22.4	6770	--

Values marked with '--' denote that the concentration was below the analysis detection limit.

Table 3.6. Chemical Analysis of Selected Samples for AP-107 CLSM Run (cont.)

Sample Name	P (Phosphate)	Np-237	Cm-242	Cm-244	Am-241	Pu-238	Pu-239/240
	mg kg^{-1}	pCi g^{-1}	pCi g^{-1}	pCi g^{-1}	pCi g^{-1}	pCi g^{-1}	pCi g^{-1}
Glass Pour 0.33	--	1.40E+00	--	4.71E+00	7.68E+01	4.55E+00	2.86E+01
Glass Pour 3.19	--	5.28E+00	--	9.90E+00	2.83E+02	1.70E+01	1.20E+02
Glass Pour 3.54	--	5.40E+00	2.01E+00	9.92E+00	2.97E+02	2.00E+01	1.67E+02
Glass Pour 5.20	--	7.86E+00	--	1.12E+01	3.33E+02	2.35E+01	2.83E+02
Glass Pour 6.04	--	1.09E+01	2.01E+00	1.34E+01	3.60E+02	2.10E+01	1.69E+02
Glass Pour 7.38	--	9.63E+00	1.66E+00	1.64E+01	3.69E+02	2.46E+01	1.85E+02
Glass Pour 9.45	--	1.44E+01	--	1.59E+01	3.97E+02	2.38E+01	1.96E+02
Glass Pour 10.07	--	1.08E+01	--	1.43E+01	3.79E+02	3.53E+01	2.19E+02
Melter Feed Bucket 1	--	5.26E+00	--	7.18E+00	1.86E+02	1.03E+01	7.85E+01
Melter Feed Bucket 2	--	4.26E+00	--	8.79E+00	1.87E+02	1.06E+01	7.40E+01
Sampling HEPA Filter 1	--	--	--	--	2.33E+00	--	1.73E+00
Sampling HEPA Filter 2	--	1.71E+00	--	--	1.85E+00	--	2.02E+00
Sampling HEPA Filter 3	--	--	--	--	2.08E+00	--	--
Primary HEPA Filter 1	--	--	--	--	--	--	2.79E+00
Primary HEPA Filter 2	36.8	--	--	--	1.40E+00	2.00E+00	8.65E+00
Condensate after HEPA 1	--	--	--	--	9.82E-01	1.19E+00	5.35E+00
Condensate after HEPA 2	--	--	--	--	--	--	--
Condensate after HEPA 3	--	--	--	--	--	--	--
Condensate End	--	--	--	--	7.58E-01	--	--
Condensate Combined 1	--	--	--	--	1.06E+00	--	--
Condensate Combined 2	--	--	--	--	--	--	--
Condensate Combined 3	--	--	--	--	--	--	--
Primary SBS Fluid	--	--	--	--	1.38E+00	--	--
Sampling SBS Fluid	--	--	--	--	--	--	--
Pre-HEPA Collected Fluid	--	--	--	--	8.03E-01	--	--

Values marked with '--' denote that the metal concentration was below the analysis detection limit.

4.0 Discussion

4.1 Technetium-99 in AP-107 Glass Pours

The effective glass production rate and plenum temperature for the AP-107 waste run in RPL (Figure 3.3 and Figure 3.1, respectively) have been plotted together with the concentration of technetium-99 (^{99}Tc) in each analyzed glass product sample (Table 3.6) from the glass pours with respect to time of the glass pour during the test run and are shown in Figure 4.1. The ^{99}Tc concentration in the glass remained relatively constant after the first glass pour, except for the glass pour at hour 3.53. The high concentration of ^{99}Tc in the hour 3.53 glass pour did not correspond with a feeding problem of any kind, so the cause for one anomalous, high ^{99}Tc concentration measurement will require further investigation such as re-analysis of the hour 3.53 glass pour and analysis of the subsequent, non-evaluated pours.

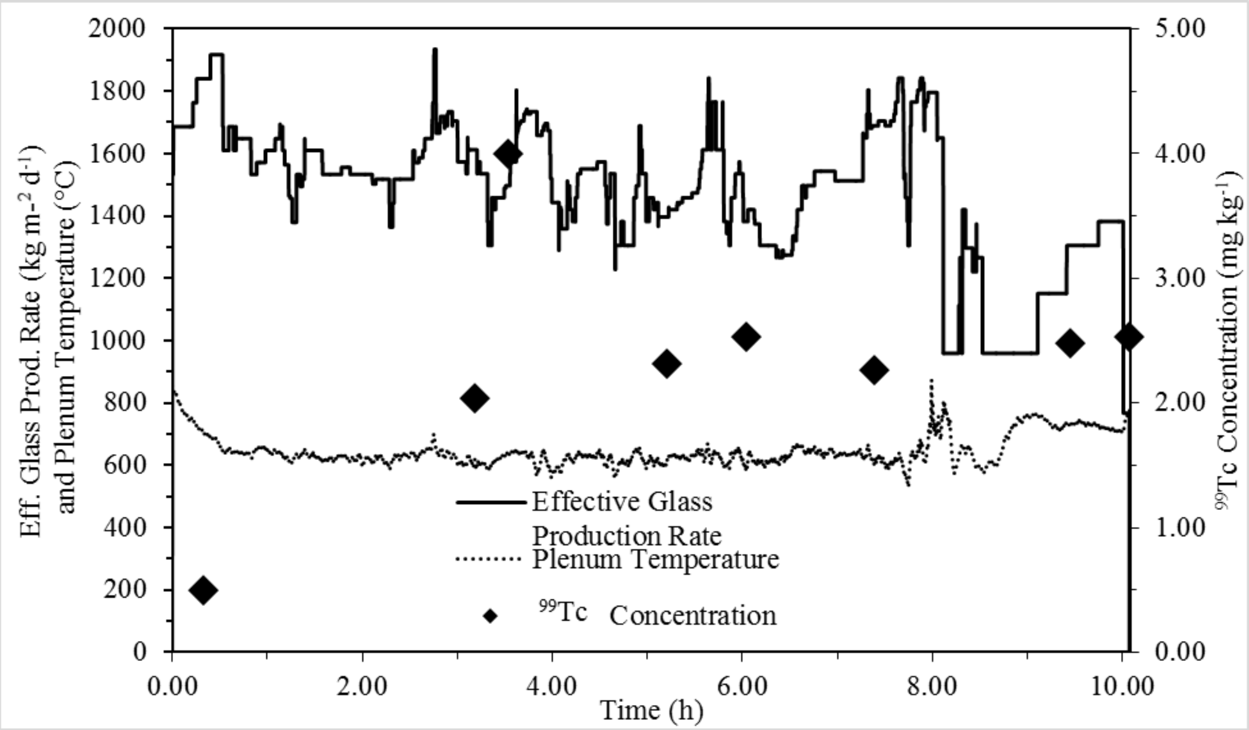

Figure 4.1. Effective glass production rate, plenum temperature, and ^{99}Tc concentration in analyzed glass pour samples for AP-107 CLSM run.

4.2 AP-107 Glass Product

Table 4.1 compares the average composition (determined by converting the metals concentration in the glass product in Table 3.6 to their associated oxides) of the samples of glass product from the AP-107 test run to the target glass composition from Table 2.3. For this comparison, minor components (target values <1.0 wt%) were removed and the glass composition renormalized. The percent difference between the measured glass product and the AP-107 target glass composition in Table 4.1 shows that the weight percent for the major glass-forming oxides are within 10% of each other, which demonstrates that the appropriate amount of GFCs were added to the AP-107 waste and that the CLSM system can convert melter feed to the desired glass product. In addition, the order of magnitude of the concentration of sulfur in the glass product from all of the glass pours in the AP-107 waste run (Table 3.6) remained consistent throughout the test run, indicating that the sulfur had reached a steady-state concentration in the glass and that a separated sulfate salt was not formed during the burn off of the cold cap and subsequent emptying of the glass melt inventory.

Table 4.1. Comparison of AP-107 Waste Glass Product with the Target Glass Composition

Metal Oxide Component	Simplified Measured Glass Composition	Simplified Target Glass Composition	% Difference Between Measured and Target Glass Composition
	wt%	wt%	%
Al_2O_3	6.1	6.2	2.2
B_2O_3	10.7	10.1	6.1
CaO	3.5	3.8	5.7
Fe_2O_3	5.9	5.6	4.4
MgO	1.5	1.5	2.8
Na_2O	19.3	17.8	8.7
SiO_2	44.9	46.9	4.2
TiO_2	1.5	1.4	3.7
ZnO	3.6	3.6	2.1
ZrO_2	2.9	3.1	6.3

4.3 Technetium-99 Retention and Recovery in AP-107 Waste Run

The retention of the ^{99}Tc in the in the AP-107 glass product ($R_{^{99}Tc}$) is given in Eq. (4.1):

$$R_{^{99}Tc} = \frac{\dot{m}_{^{99}Tc,glass}}{\dot{m}_{^{99}Tc,feed}} \tag{4.1}$$

where $\dot{m}_{^{99}Tc,glass}$ is the mass flow rate of ^{99}Tc out of the melter via the glass and $\dot{m}_{^{99}Tc,feed}$ is the mass flow rate of ^{99}Tc into the melter via the melter feed. If $R_{^{99}Tc}$ is calculated for a fixed amount of time during the off-gas sampling periods (Table 3.3), the mass flow rates ($\dot{m}_{^{99}Tc}$) become total mass values ($m_{^{99}Tc}$), and Eq. (4.1) can be rewritten as:

$$R_{^{99}Tc} = \frac{m_{^{99}Tc,glass}}{m_{^{99}Tc,feed}} \tag{4.2}$$

Since the feeding rate of AP-107 melter feed could not be measured for the test run, the feeding rate during each off-gas sampling period was determined from the effective glass production rate (Figure 3.3) and the value for glass yield per kilogram of melter feed (466 g-glass kg-feed^{-1}) as calculated by Matlack et al. (2018). The average concentration of ^{99}Tc in the two melter feed buckets (Table 3.6) was used to calculate $m_{^{99}Tc,feed}$ since the melter feed from bucket 2 was being transferred to bucket 1 throughout the run as needed. Thus the masses of ^{99}Tc input into the system during collection of off-gas samples 1, 2, and 3 were calculated from the calculated melter feed input, the time duration of the off-gas sampling periods (Table 3.3), and the average ^{99}Tc concentration in the melter feed (Table 3.6). The results of this calculation are shown in Table 4.2. The mass of ^{99}Tc output from the system during these periods was calculated from the effective glass production rate of AP-107 glass product (Figure 3.3) and the concentration of ^{99}Tc in the glass product (Table 3.6) that was poured following each off-gas sampling period. Glass pours 3.19, 5.20, and 7.38 were after off-gas samples 1, 2, and 3, respectively, and the concentrations of ^{99}Tc in each resultant glass product is given in Table 3.6. The retention of ^{99}Tc during each off-gas sampling period was then calculated and the results are listed in Table 4.2.

Table 4.2. Technetium-99 Retention and Recovery during Off-Gas Sampling Periods

Off-gas Sample Number	^{99}Tc Mass Input Feed	^{99}Tc Mass Output Glass	^{99}Tc Mass Output Sampling HEPA	^{99}Tc Mass Output Sampling SBS	$R_{^{99}Tc}$	^{99}Tc Recovery
	mg	mg	mg	mg		%
1	1.112	0.394	0.338	0.056	0.35	~ 71
2	0.990	0.398	0.617	0.050	0.40	~ 108
3	1.034	0.406	0.508	0.052	0.39	~ 94

The percent recovery of ^{99}Tc in the CLSM system during collection of off-gas samples 1, 2, and 3 (^{99}Tc Recovery) is given in Eq. (4.3):

$$^{99}\text{Tc Recovery} = \frac{m_{^{99}Tc,glass} + m_{^{99}Tc,off-gas}}{m_{^{99}Tc,feed}} \times 100 \tag{4.3}$$

where $m_{^{99}Tc,off-gas}$ is the total mass of ^{99}Tc captured in the off-gas system. Throughout each sampling period, ^{99}Tc was collected in three different locations: the sampling HEPA filters (located as the first unit of the sampling loop, not the primary HEPA filters located as the end of the off-gas system), the sampling SBS, and the walls of the off-gas system leading up to the sampling HEPA filters. While the sample HEPA filters and sample SBS fluid were able to be collected, washing the walls of the off-gas sampling system was not performed due to radioactive contamination restrictions in the CLSM system fume hood. The masses of ^{99}Tc on each set of sampling HEPA filters were calculated from the mass of each filter (Table 3.6) and concentration of ^{99}Tc on each filter (Table 3.6) and are given in Table 4.2. Sampling SBS liquid collection was performed once the CLSM system was shut down, thus the ^{99}Tc collected from the liquid was deposited during the totality of all three sampling events. As a result, the total mass of ^{99}Tc in the liquid was partitioned to each off-gas sample based on the duration of each sampling time (Table 3.3). The resulting masses of ^{99}Tc captured by the sampling SBS during off-gas samples 1, 2, and 3 (calculated from the mass of each solution and concentration of ^{99}Tc in each solution, both shown in Table 3.6) are given in Table 4.2 as well as the calculated ^{99}Tc recovery for each sample.

The ^{99}Tc recovery was also calculated throughout the entire CLSM system. The ^{99}Tc concentration of each measured glass pour, shown in Table 3.6, was assumed to be the concentration present in each previous, unmeasured pour. The total mass of ^{99}Tc recovered in each section of the CLSM system is shown in Table 4.3 and the total ^{99}Tc recovery throughout the CLSM system was 91%, the balance of which was assumed to remain in the inaccessible sections of the CLSM system or be the result of measurement tolerance. This 91% recovery of ^{99}Tc in the CLSM system is also in the range (80-100%) of the total technetium recovered in the DM10 system operated by the VSL at the Catholic University of America (Matlack et al. 2017) for a variety of melter feed compositions.

Table 4.3. Technetium-99 Recovery Throughout CLSM System

Total ^{99}Tc in Feed	Total ^{99}Tc in Glass	Total ^{99}Tc in Sampling HEPA filters	Total ^{99}Tc in Primary HEPA filters	Total ^{99}Tc in Condensate	Total ^{99}Tc in Sampling SBS	Total ^{99}Tc in Primary SBS	Total ^{99}Tc in pre-HEPA Fluid	Total ^{99}Tc Recovery
mg	mg	mg	mg	mg	mg	mg	mg	%
40.307	21.285	1.464	1.123	8.803	0.158	3.037	0.937	91

Due to the unsteady nature of ^{99}Tc incorporation into glass and volatilization from glass during cold-cap build-up or burn-off and melter idling (Pegg 2015), this total system balance method was not used to calculate ^{99}Tc retention in the AP-107 glass product. The lower ^{99}Tc recovery during the first off-gas sampling period (71%) compared to the recovery throughout the entire CLSM system (91%) indicated that the incorporation of ^{99}Tc into glass had not reached a chemical steady state by that time of processing. In the subsequent off-gas sampling periods, the ^{99}Tc recovery was roughly equivalent to the recovery throughout the entire CLSM system indicating that the ^{99}Tc incorporation had reached a chemical steady state. Thus the $R_{^{99}Tc}$ values reported in Table 4.2 during the second and third off-gas sampling time frames best capture the steady state CLSM conditions and the resulting average $R_{^{99}Tc}$ for the AP-107 waste CLSM run was 0.40. This $R_{^{99}Tc}$ value for AP-107 melter feed processing in the CLSM is within the range (18-66%) of ^{99m}Tc single pass retention in the DM10 system operated by the VSL at the Catholic University of America (Matlack et al. 2011) for a variety of melter feed compositions.

4.4 Silicon Recovery in CLSM System

The recovery of silicon (Si) throughout the CLSM system was calculated in the same manner as the ^{99}Tc recovery in Equation 4.3. The mass of Si in the starter glass present in the CLSM vessel had to be deducted from the total mass of Si recovered in the AP-107 glass product since it was not a result of the vitrification of the AP-107 melter feed. In addition, the mass of Si in a blank HEPA filter (measured in Dixon et al. 2018) was subtracted from the total mass of Si recovered in the six sampling HEPA filters and two primary HEPA filters. The total mass of Si recovered in each section of the CLSM system is shown in Table 4.4 and the total Si recovery throughout the CLSM system was 98%. This near-100% Si recovery indicated that the AP-107 melter feed-to-glass conversion value used to estimate the mass of melter feed consumed was an accurate value to use for the ^{99}Tc retention and recovery calculations.

Table 4.4. Silicon Recovery Throughout CLSM System

Total Si in Feed	Total Si in Glass	Total Si in Sampling HEPA filters	Total Si in Primary HEPA filters	Total Si in Condensate	Total Si in Sampling SBS	Total Si in Primary SBS	Total Si in pre-HEPA Fluid	Total Si Recovery
g	g	g	g	g	g	g	g	%
1446.8	1407.4	5.6	2.8	0.2	0.0	0.1	0.0	98

4.5 Cesium Recovery

Despite the concentration of Cs in the AP-107 melter feed being less than the chemical analysis limit of detection for the inductively coupled plasma-mass spectrometry method, Cs was detected in quantifiable concentrations in all output product samples. Similar results were observed during the AP-105 melter feed run and it was postulated that a Cs spike was a result of the CLSM vessel change mid-run (Dixon et al. 2018). While the same CLSM vessel was used during the AP-107 processing, another source of Cs contamination may have been the 2.0 kg of previously prepared AP-107 glass pieces added to the CLSM vessel prior to melter feed charging. Chemical analysis of the impurities present in the raw materials used to fabricate the AP-107 glass pieces would be required to further investigate this phenomena.

4.6 Comparison between AP-105 and AP-107 Waste CLSM Runs

The AP-105 and AP-107 actual tank waste samples pre-treated by PNNL, as described in Sections 1.0 and 2.3, were both processed in the CLSM system in RPL. A comparison between the processing results from the AP-105 and AP-107 CLSM runs is given in Table 4.5. The CLSM operations team noted when the feeding rate or bubbling rate were adjusted, the corresponding response of the cold-cap coverage/thickness and plenum temperature happened quicker during the AP-107 melter feed processing compared to the AP-105 melter feed processing. The average bubbling rate during the AP-107 test run was ~15% higher than during the AP-105 test run, resulting in a slightly lower average glass temperature for the AP-107 run. To optimize the glass production rate while maintaining cold-cap steady state, the CLSM operations team determined that the higher bubbling rate was necessary for the AP-107 processing. Given those influences, the glass production rate was ~10% higher for the AP-107 run compared to the AP-105 run even with the cold-cap coverage appearing to be ~90% during both runs. The measured ^{99}Tc retention during the AP-107 processing was also ~50% greater than during AP-105 processing, although the ^{99}Tc retention measured during AP-105 processing may have been artificially low due to the anomalous liquid found in two of the sampling HEPA filter housings as described in Dixon et al. (2018).

Table 4.5. Performance Results Comparison between AP-105 and AP-107 Processing in the CLSM

Parameter	AP-105	AP-107
Test Duration, h	15.09	10.07
Volume of Waste Received, L	12.4	8.6
Melter Feed Consumed (estimate), kg	22.2	15.0
Glass Produced, kg	9.45	7.01
Average Glass Production Rate, kg m^{-2} d^{-1}	1330	1477
Average Bubbling Rate, L m^{-2} min^{-1}	125	149
Average Glass Temperature, °C	1122	1115
Average Plenum Temperature, °C	673	648
Average ^{99}Tc Retention	0.18	0.40

The average measured composition of the glass product generated from the AP-105 and AP-107 waste vitrification is shown in Table 4.6. The AP-107 glass product composition was ~2.0 wt% greater in B_2O_3 + CaO concentration while the AP-105 glass product composition made up the balance with a ~2.0 wt% greater Na_2O concentration. All other components were ≤ ~0.2 wt% different between the AP-105 and AP-107 glass product. The predicted viscosities of AP-105 and AP-107 simulant glasses at 1150 °C were 5.5 Pa s (Matlack et al. 2017) and 5.4 Pa s (Matlack et al. 2018), respectively. The similarity in glass composition and viscosity indicated that the difference in production rate between AP-105 and AP-107 processing was not due to the glass properties. With similar, observable cold-cap coverage and thickness during the steady-state processing, the production difference between AP-105 and AP-107 appeared to be due to properties of the cold cap such as melter feed thermal conductivity or melting rate.

Table 4.6. Glass Product Composition Comparison between AP-105 and AP-107 Processed in the CLSM

Metal Oxide Component	Measured AP-105 Glass Composition	Measured AP-107 Glass Composition
	wt%	wt%
Al_2O_3	6.1	6.1
B_2O_3	10.0	10.7
CaO	2.3	3.5
Fe_2O_3	6.0	5.9
MgO	1.4	1.5
Na_2O	21.1	19.3
SiO_2	45.1	44.9
TiO_2	1.4	1.5
ZnO	3.7	3.6
ZrO_2	2.8	2.9

The average concentrations of metals found in the condensate samples generated from the AP-105 and AP-107 vitrification are shown in Table 4.7. The concentrations of most metals was found at the same order of magnitude, or within 50%, in both the AP-105 and AP-107 condensate samples. As seen with the AP-105 samples (Dixon et al. 2018), the levels of Cr and Ni in the AP-107 condensate (and glass product) samples were greater than expected, likely due to the corrosion of those components from the walls of the Inconel-690 CLSM vessel.

Table 4.7. Condensate Sample Concentration Comparison between AP-105 and AP-107 Processed in the CLSM

Metal Component	Average AP-105 Condensate	Average AP-107 Condensate
	mg kg^{-1}	mg kg^{-1}
Technetium-99	2.44	1.44
Cesium	27.3	12.3
Aluminum	38.0	13.6
Boron	222	175
Calcium	16.1	12.3
Chromium	6.79	7.70
Iron	35.1	15.2
Lithium	--	4.44
Molybdenum	1.00	0.687
Nickel	0.468	0.727
Potassium	72.8	38.8
Silicon	51.4	41.2
Sodium	1150	676
Titanium	2.11	0.969
Tungsten	1.48	--
Zinc	59.2	31.3
Zirconium	2.15	1.23
Chloride	1035	534
Sulfate	246	226
Fluoride	18.4	27.3
N (Nitrate)	4470	2130

Values marked with '--' denote that the metal concentration was below the analysis detection limit.

5.0 Conclusions

The CLSM system was designed to convert obtainable volumes of waste from Hanford tanks to glass while collecting process data and product samples for analysis. For this test run, waste from tank 241-AP-107, called AP-107 waste, was obtained and subjected to both solids removal by filtration and cesium removal by ion exchange. The resultant 8.6 L of AP-107 waste were combined with GFCs to form approximately 11.8 L of melter feed. Roughly 15.0 kg of AP-107 melter feed was charged to the CLSM vessel for 10.07 hours, producing 7.01 kg of glass product, resulting in an average glass production rate of 1477 kg m^{-2} d^{-1}. Given the CLSM vessel glass melt inventory of 2.0 kg, this processing resulted in more than three inventory turnovers. For three, ~15-minute segments during melter feed charging, when the processing characteristics were observed to approach steady state, the off-gas stream was diverted to a sampling loop and the volatiles were captured on a set of HEPA filters. Selected samples of melter feed, glass product, off-gas HEPA filters, and cooled off-gas condensate were sent for chemical analysis.

Results of the chemical analysis of the glass product samples indicated that the measured composition was similar to the desired, target AP-107 glass composition. The average retention fraction of ^{99}Tc, a radionuclide of interest, from the AP-107 melter feed into the glass product during the latter two of the three off-gas sampling time periods, when the CLSM conditions and chemical recovery for ^{99}Tc were near steady state, was calculated to be 0.40. The total recovery of ^{99}Tc in the CLSM system during the entire AP-107 waste test run was ~91%, a near complete recovery given analytical error and the fact that the off-gas system was not washed after the run.

Prior to the AP-107 waste CLSM test run, waste from Hanford tank 241-AP-105, called AP-105 waste, was vitrified in the CLSM system. The average glass production rate during the AP-105 waste vitrification was 1330 kg m^{-2} d^{-1}, a lower rate than the AP-107 processing, with slightly higher average glass melt operating temperature and slightly lower bubbling rate. Given the similarity in composition between AP-105 and AP-107 glass products, these production differences are hypothesized to be influenced primarily by the cold-cap properties. The ^{99}Tc retention in the AP-105 glass product was calculated to be 0.18, but due to test run anomalies, this value may be artificially low, thus the ^{99}Tc retention difference during AP-105 and AP-107 processing may be due to chemical behavior of the ^{99}Tc in the respective LAW glasses or to the CLSM conduct that was corrected between runs. Finally, the condensate collected from both the AP-105 and AP-107 test runs had similar concentrations of metals present and may be studied for alternative disposal methods if desired.

The observed dynamic behavior from the AP-107 waste test run in the CLSM system confirmed the viability of the platform to produce data that are representative of full-scale WTP melter operations. The test objectives for the CLSM system were successfully achieved and additional testing is encouraged to further elucidate production changes amongst desired additional wastes or ^{99}Tc spiked simulants.

6.0 References

Bechtel. 2015. *ICD 30 – Interface Control Document for Direct LAW Feed.* Report No. 24590-WTP-ICD-MG-01-030, Rev 0, Bechtel National, Inc., Richland, Washington.

Cantrell KJ, II Leavy, MM Snyder, SR Baum, SD Shen, BD Williams, SA Saslow, and EJ Krogstad. 2018. *Off-Gas Condensate and Cast Stone Analysis Results*. PNNL-27765 (RPT-DFTP-012, Rev. 0). Pacific Northwest National Laboratory, Richland, Washington.

Dixon DR, MJ Schweiger, BJ Riley, R Pokorny, and P Hrma. 2015. *Temperature Distribution within a Cold Cap during Nuclear Waste Vitrification*. Environmental Science and Technology, 49: 8856-8863.

Dixon DR, CM Stewart, JJ Venarsky, JA Peterson, GB Hall, TG Levitskaia, JR Allred, WC Eaton, JB Lang, MA Hall, DA Cutforth, AM Rovira, and RA Peterson. 2018. *Vitrification of Hanford Tank Waste 241-AP-105 in a Continuous Laboratory-Scale Melter*. PNNL-27775 (RPT-DFTP-010, Rev. 0). Pacific Northwest National Laboratory, Richland, Washington.

Fiskum SK, JR Allred, HA Colburn, AM Rovira, MR Smoot, and RA Peterson. 2018. *Multi-Cycle Cesium Ion Exchange Testing Using Spherical Resorcinol-Formaldehyde Resin with Diluted Hanford Tank Waste 241-AP-105*. PNNL-27432 (RPT-DFTP-006, Rev. 0). Pacific Northwest National Laboratory, Richland, Washington.

Geeting JGH, JR Allred, AM Rovira, RW Shimskey, CA Burns, and RA Peterson. 2018a. *Crossflow Filtration of Hanford Tank AP-105 Supernatant*. PNNL-27085 (RPT-DFTP-005, Rev. 0). Pacific Northwest National Laboratory, Richland, Washington.

Geeting JGH, AM Rovira, JR Allred, RW Shimskey, CA Burns, and RA Peterson. 2018b. *Filtration of Hanford Tank AP-107 Supernatant*. PNNL-27638 (RPT-DFTP-009, Rev. 0). Pacific Northwest National Laboratory, Richland, Washington.

Kim DS, JD Vienna, and AA Kruger. 2012. *Preliminary ILAW Formulation Algorithm Description,* 24590 LAW RPT-RT-04-0003, Rev. 1. ORP-56321, Revision 0. U. S. Department of Energy Office of River Protection, Richland, Washington.

Matlack KS, IS Muller, I Joseph, and IL Pegg. 2010. *Improving Technetium Retention in Hanford LAW Glass – Phase 1,* VSL-10R1920-1, Rev. 0, Vitreous State Laboratory, The Catholic University of America, Washington, DC and Atkins Energy Federal EPC, Inc., Calverton, Maryland.

Matlack KS, H Abramowitz, IS Muller, I Joseph, and IL Pegg. 2017. *DFLAW Glass and Feed Qualifications to Support WTP Start-Up and Flow-Sheet Development,* VSL-17R4330-1, Rev. 0. Vitreous State Laboratory, The Catholic University of America, Washington, DC and Atkins Energy Federal EPC, Inc., Calverton, Maryland.

Matlack KS, H Abramowitz, IS Muller, I Joseph, and IL Pegg. 2018. *DFLAW Glass and Feed Qualifications for AP-107 to Support WTP Start-Up and Flow-Sheet Development,* VSL-18R4500-1, Rev. A. Vitreous State Laboratory, The Catholic University of America, Washington, DC and Atkins Energy Federal EPC, Inc., Columbia, Maryland.

Pegg IL. 2015. *Behavior of technetium in nuclear waste vitrification processes*. Journal of Radioanalytical Nuclear Chemistry, 305: 287-292.

Peterson RA, SK Fiskum, WC Eaton, and J Geeting. 2017. *Functions and Requirements of the Radioactive Waste Test Platform*, PNNL-26322 (RPT-DFTP-002, Rev. 0). Pacific Northwest National Laboratory, Richland, Washington.

Rovira AM, SK Fiskum, HA Colburn, JR Allred, MR Smoot, and RA Peterson. 2018. *Cesium Ion Exchange Testing Using Crystalline Silicotitanate with Hanford Tank Waste 241-AP-107*. PNNL-27706 (RPT-DFTP-011, Rev. 0). Pacific Northwest National Laboratory, Richland, Washington.

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