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RPT-DFTP-010, Rev. 0

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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 product for actual tank waste from tank 241-AP-105 (referred to herein as AP-105). To acquire this type of information, a small-scale melter system was desired 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-105 waste samples. Two CLSMs were constructed: one in a non-radioactive environment for processing waste simulants and another in a fume hood capable of handling radioactive material for processing actual Hanford tank waste. This dual-system setup allowed for comparison between simulated and real tank waste with essentially identical test equipment.

AP-105 waste simulant was first tested in the non-radioactive CLSM to determine the processability and desired operating window for the actual tank waste. Rhenium (Re) was added to the simulant to act as a surrogate for technetium-99 (^{99}Tc), a key waste component found in the actual tank waste. The partitioning of Re into the glass and off-gas products during the continuous feeding of simulant was determined, which allowed the Re retention, the mass flow rate of Re out of the CLSM via the glass product with respect to the mass flow rate of Re into the CLSM via the feed slurry, to be calculated. Key processing results and the average Re retention, from the CLSM test run with AP-105 simulant feed slurry are shown in Table ES.1.

Table ES.1. CLSM Test Runs Summary

	AP-105 Simulant Test Run	AP-105 Waste Test Run
Volume of Simulant/Waste Batched, L	6.2	12.4
Feeding Duration, h	5.83	15.09
Glass Produced, kg	4.3	9.5
Average Glass Production Rate, kg m^{-2} d^{-1}	1574	1330
Average Re/^{99}Tc Retention	0.39	0.18

Twelve liters of actual AP-105 tank waste were received, filtered, ion exchanged for cesium removal, and mixed with GFCs to form 16 L of waste feed slurry. This quantity of feed slurry was vitrified into 9.5 kg of glass product over the course of 15 hours of feeding to the CLSM. The processing results and average ^{99}Tc retention value from the CLSM test run with AP-105 waste feed slurry shown in Table ES.1 are similar to those from the simulant test run. Select samples of the glass product and off-gas condensate collected from the waste test run and simulant test run were analyzed for chemical composition and compared favorably to one another, as shown in Table ES.2.

Table ES.2. CLSM Waste and Simulant Test Runs Glass and Condensate Product Composition

Metal Oxide Component	Average AP-105 Simulant Glass wt%	Average AP-105 Waste Glass wt%	Metal Component	Average AP-105 Simulant Condensate Samples mg kg^{-1}	Average AP-105 Waste Condensate Samples mg kg^{-1}
Al_2O_3	6.4	6.1	Rhenium	2.40	--
B_2O_3	9.8	10.0	Technetium-99	--	2.44
CaO	2.2	2.3	Cesium	--	27.3
Fe_2O_3	5.8	6.0	Aluminum	17.8	38.0
MgO	1.4	1.4	Boron	143	222
Na_2O	20.9	21.1	Calcium	10.9	16.1
SiO_2	46.4	45.1	Chromium	6.47	6.79
TiO_2	1.3	1.4	Iron	27.1	35.1
ZnO	3.2	3.7	Molybdenum	--	1.00
ZrO_2	2.6	2.8	Nickel	--	0.468
			Potassium	38.3	72.8
			Silicon	29.7	51.4
			Sodium	694	1150
			Titanium	0.896	2.11
			Tungsten	--	1.48
			Zinc	33.2	59.2
			Zirconium	0.999	2.15
			Chloride	553	1035
			Sulfate	161	246
			Fluoride	10.4	18.4
			N (Nitrate)	1440	4470

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. The authors gratefully acknowledge the financial support for initial testing of the continuous laboratory-scale melter system provided by the U.S. Department of Energy Waste Treatment and Immobilization Plant Project and project direction provided by Dr. Albert A. Kruger.

Acronyms and Abbreviations

APEL	Applied Process Engineering Laboratory
CLSM	continuous laboratory-scale melter
DFLAW	Direct Feed Low-Activity Waste
FIO	for-information-only
GFCs	glass-forming chemicals
HEPA	high efficiency particulate air
HLW	high-level waste
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
XRD	x-ray diffraction

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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. During vitrification, 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.

Test platforms were established at Pacific Northwest National Laboratory (PNNL) to conduct scaled unit operation process steps with actual waste from tank 241-AP-105 (hereafter called AP-105) in the Radiochemical Processing Laboratory (RPL). As part of this test program, a continuous laboratory-scale melter (CLSM) and off-gas system were assembled at the Applied Process Engineering Laboratory (APEL) and at the RPL to vitrify simulated and radioactive AP-105 feeds and collect glass and off-gas gaseous and liquid effluents for elemental analysis. Tests were conducted from February 2018 to April 2018.

This test report describes the results from these small-scale vitrification tests using the CLSM test platforms at APEL and RPL, both facilities operated by PNNL.

The results described are products of testing conducted in fiscal year 2018. The first series of tests at APEL were required for system and process optimization and later testing at RPL with actual AP-105 tank waste. In all tests, the processing rate combined with bubbling rates and overall control of melter

operating parameters were comparable to each other and comparable to AP-105 simulated feed tests conducted previously with the same nonradioactive simulant slurry by the Vitreous State Laboratory (VSL) at the Catholic University of America in Washington, DC (Matlack et al. 2010, 2011, 2017).

The purpose of these tests was to support the vitrification portion of a program to simulate a complete scaled DFLAW process and establish comparisons with previous tests conducted at VSL and determine if the CLSM can provide valuable information in the future. Ultimately the CLSM could 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 69832. 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 systems assembled in APEL and RPL, the preparation of the simulated and actual waste feed slurries, 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 simulated AP-105 waste feed at APEL and actual AP-105 tank waste at RPL.

Table 2.1. Test Objective Success Criteria

Test Objective	Success Criteria	Result	Result Reference
Assemble a CLSM system in a fume canopy in APEL.	CLSM system prepared and commissioned for testing.	CLSM system successfully assembled and used for simulant feed slurry vitrification.	Section 2.2.1
Assemble a CLSM system in a fume hood at RPL.	CLSM System prepared and commissioned for testing.	CLSM system successfully assembled and used for waste feed slurry vitrification.	Section 2.2.1
Vitrify AP-105 simulant while periodically pouring glass and collecting off-gas samples.	Operate the CLSM to vitrify approximately 3 melter volumes of glass while collecting glass and off-gas samples.	4.3 kg of glass produced (2-kg melter capacity) while glass poured throughout and 3 off-gas samples collected. The amount of glass poured corresponds to 2+ melter volumes, which was less than the desired 3 volumes.	Section 3.1
Vitrify LAW AP-105 tank waste feed while periodically pouring glass and collecting off-gas samples.	Operate the CLSM to vitrify approximately 3 melter volumes of glass while collecting glass and off-gas samples.	9.5 kg of glass produced (2-kg melter capacity) while glass poured throughout and 3 off-gas samples collected. The amount of glass poured corresponds to 4.5+ melter volumes.	Section 3.2

Table 2.1. Test Objective Success Criteria (cont.)

Test Objective	Success Criteria	Result	Result Reference
Collect vitrified glass samples. (APEL & RPL)	Collect vitrified glass samples during the processing of feed slurry.	Collected samples of glass product from 18 glass pours in APEL and 27 glass pours in RPL.	Sections 3.1.1 and 3.2.1
Collect samples of melter off-gas semi-volatiles with the sample loop filters (APEL and RPL).	After the melter has been operating at steady state, collect melter off-gas samples in the off-gas sample loop while at steady-state feeding.	Collected 3 off-gas samples when the CLSM reached steady state feeding for both the APEL and RPL systems.	Sections 3.1.1 and 3.2.1
Collect melter off-gas condensate from the total run. (APEL and RPL)	Collect all off-gas condensate during the processing of feed slurry.	Collected all off-gas condensate during feeding for both the APEL and RPL systems.	Sections 3.1.3 and 3.2.3
Collect solid samples from off-gas line, post testing. (APEL and RPL)	Collect solids that have collected in the off-gas system between the CLSM vessel and submerged bed scrubber (SBS) and between the CLSM vessel and sampling high-efficiency particulate air (HEPA) filters.	Solids were collected via a wash of the APEL system after testing. The system in RPL could not be washed after testing due to radiological contamination restrictions.	Sections 3.1.2 and 3.1.3
Analyze samples of melter feed, glass, off-gas particulate, and condensate. (rad and non-rad samples)	Complete the analysis of samples identified as representing ideal test conditions	Samples of glass product, feed slurry, HEPA filters, condensate, wash liquids, and other liquids for both the APEL and RPL systems were sent for chemical analysis.	Sections 3.1.3 and 3.2.3
Collect process data to allow comparative studies to be performed between the simulated feed at APEL and actual waste feed at RPL.	Report the results of a comparative process study between the APEL and RPL test platforms.	Process data were collected and reported from both the APEL and RPL systems and key comparative analysis was performed.	Sections 3.1.1, 3.2.1, and 4

2.2 CLSM System

AP-105 simulant and actual tank waste were 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**.

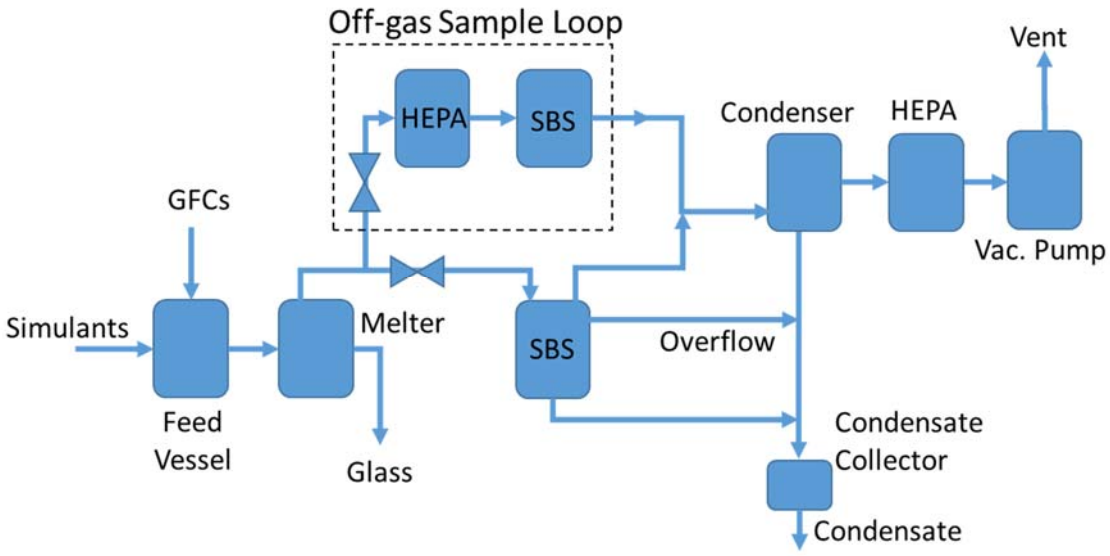

Figure 2.1. Simplified flow diagram of the CLSM system.

The total AP-105 simulated feed volume processed was ~8 L. The AP-105 actual tank waste feed volume processed during the radioactive run was ~16 L. The 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 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 0.011 m^2. The melter 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 lid of the melter 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 melter vessel by a surrounding furnace. The hot zone of the furnace was located below and around the melt pool while the cold cap and plenum areas of the melter vessel were surrounded by insulation. The test melter achieved continuous operation by periodically pouring glass out of the melt pool to a glass product tray below the melter. Pouring was achieved by lowering the vacuum maintained on the melter vessel by the off-gas system, which allowed glass to exit near the bottom of the melter, rise up through a discharge riser, and pass over an overflow weir.

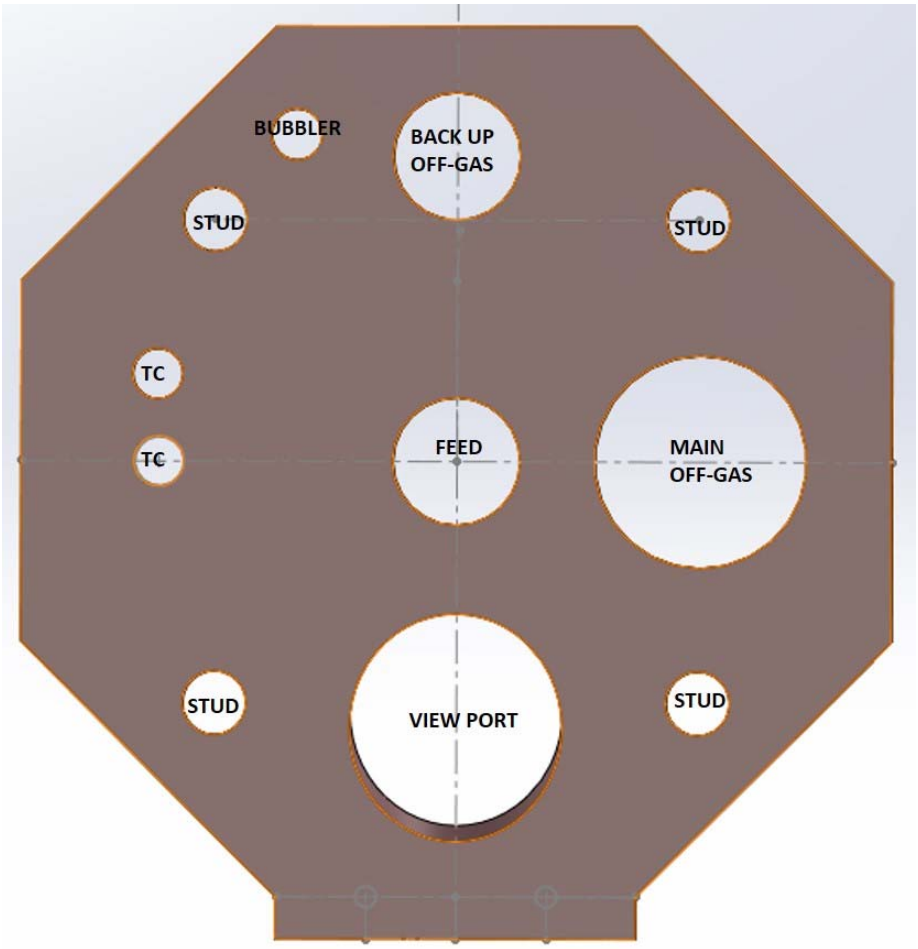

Figure 2.2. CLSM vessel lid and identified ports.

The off-gas produced by the conversion of melter feed to molten glass was drawn off from a port in the melter 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, 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. The HEPA filter captured any remaining difficult-to-remove particulates. After HEPA filtration, the off-gas was released to the fume canopy/fume hood ventilation system.

The total off-gas stream could be sampled by diverting the full off-gas flow through a sample 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 feed mixing system, feed delivery system, small-scale Inconel 690 melter 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 APEL is shown in Figure 2.3 and the CLSM system in RPL is shown in Figure 2.4.

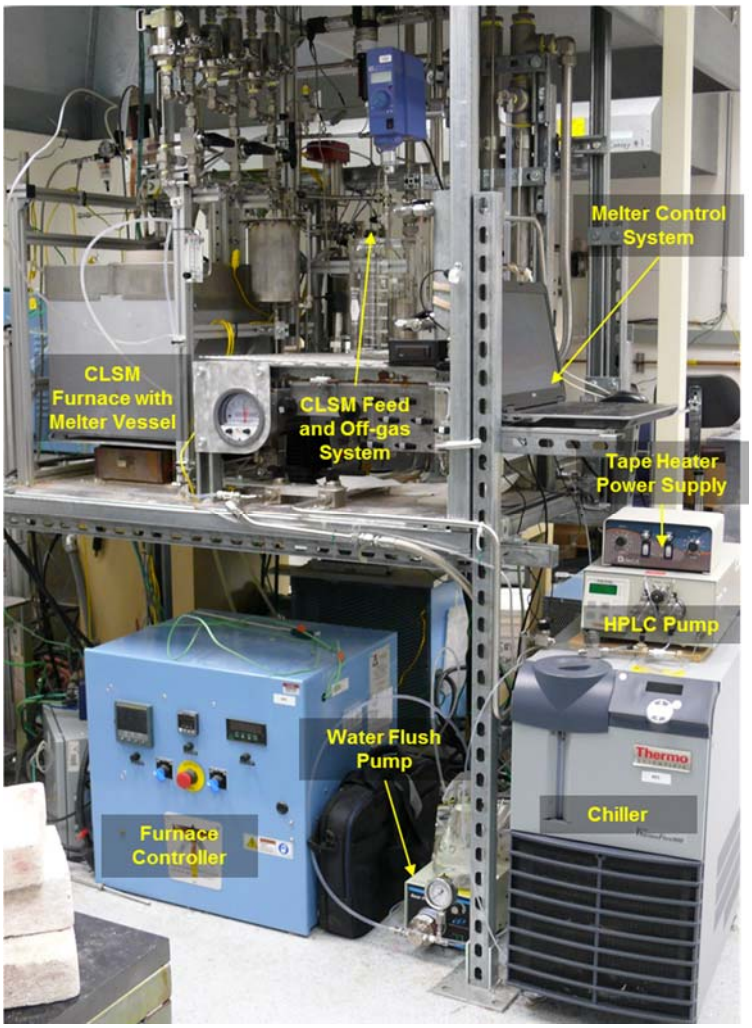

Figure 2.3. CLSM system layout in APEL ventilated space under fume canopy.

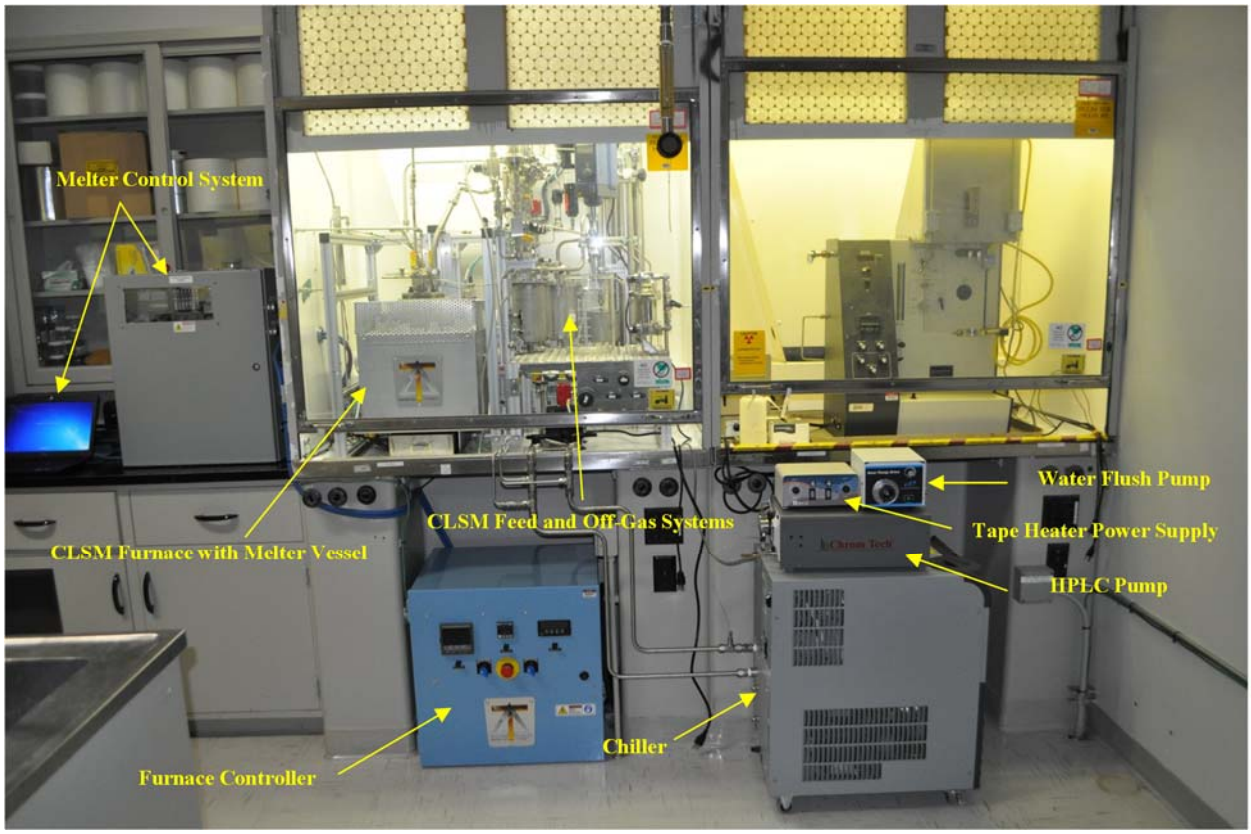

Figure 2.4. CLSM system layout in RPL fume hood

2.2.2 Melter Operation

The melter was operated to maintain a glass pool temperature of 1150°C (± 30 °C). During feeding operations, the target processing 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 of 75%-95%. The cold-cap coverage was determined from visual observation through the melter lid viewport. The CSLM briefly did produce glass pool and plenum temperatures above and below that 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 to a collector. The SBS level was maintained by overflow so that the pressure drop across the SBS remained relatively constant.

In the off-gas sample loop, the line from the melter 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 melter vessel during feeding operation. The nominal operating vacuum pressure was 2 – 4 in-H_2O. The melter vessel vacuum

was reduced periodically to pour glass. At the end of the run, the bubbler air and viewport purge was adjusted to increase the pressure in the melter, purging controlled volumes of glass from the melter vessel.

2.3 Melter Feed Preparation

The reference simulant and glass formulations for these tests were based on "AP-105 SPRN" simulant formulated to produce "WDFL1" glass referenced in Matlack et al. (2017), both satisfying the WTP baseline glass models documented in Kim et al. (2012). The target weight percent of each component in the WDFL1 glass is listed in

Table **2.2**.

Table 2.2. Target Composition of WDFL1 glass from AP-105 Tank Waste

Component	Wt%
Al_2O_3	6.10
B_2O_3	10.00
CaO	2.08
Cl	0.45
Cr_2O_3	0.05
F	0.01
Fe_2O_3	5.50
K_2O	0.41
MgO	1.48
Na_2O	21.00
NiO	0.00
P_2O_5	0.17
PbO	0.00
SO_3	0.30
SiO_2	44.55
TiO_2	1.40
ZnO	3.50
ZrO_2	3.00
Total	100.00

2.3.1 AP-105 Simulant Feed for APEL Testing

The AP-105 simulant and associated GFCs were batched and mixed into the slurry feed produced by Optima Chemicals on January 6, 2018. The mass and assay values for each chemical used by Optima to formulate the AP-105 simulant slurry feed are listed in

Table **2.3**. The AP-105 simulant slurry feed was procured by PNNL from Optima and was mixed/agitated on February 12 and 13, 2018. As a final chemical adjustment, ~11.0 kg of the total water mass, the sucrose, and Re_2O_7 (listed in

Table **2.3**) were added to the slurry to achieve a target glass yield of 635 g-glass L-slurry^{-1} with a Re concentration in the final glass of 13.9 ppm. Chemical additions were performed to fulfill requirements of a Test Plan (PNNL 2018) prepared by PNNL and approved by WRPS and conducted according to a PNNL prepared Test Instruction (Dixon 2018).

Table 2.3. Component Mass and Assay Values for 90.4 L of AP-105 Simulant Feed Slurry Acquired from Optima Chemicals

Component	Mass (g)	Assay
Water	37853.46	1.0000
$Al(NO_3)_3 \cdot 9H_2O$	19023.83	0.6111
$Ca(NO_3)_2 \cdot 4H_20$	16.74	0.9995
$Ni(OH)_2$	2.96	0.9655
PbO	1.48	0.9990
SiO_2	9.84	0.9997
NaOH	19113.14	0.5003
KOH	306.67	0.9090
$Na_2CrO_4 \cdot 4H_2O$	91.07	0.9900
$Na_3PO_4 \cdot 12H_2O$	523.26	1.0000
NaCl	428.75	0.9996
NaF	9.84	0.9864
Na_2SO_4	310.02	0.9986
$C_2H_3NaO_2$	635.99	0.9940
$NaCO_2H$	412.01	0.9842
$Na_2C_2O_4$	45.29	1.0000
$NaNO_2$	5546.38	0.9950
$NaNO_3$	2259.64	0.9850
Na_2CO_3	805.81	0.9987
Kyanite	3353.73	
Boric Acid	10271.08	
Wollastonite	2621.36	
Iron Oxide	3009.87	
Olivine	1741.62	
Silica	21212.01	0.9997
Rutile Sand (TiO_2)	743.30	
Zinc Oxide	2009.56	
Zircon Flour	2599.03	
Sucrose	3790.91	
Re_2O_7	1.04	

Solid agglomerates up to ~1 inch in diameter were discovered while adding the final chemicals to the slurry at PNNL, and these agglomerates did not dissolve into solution after a day of mixing. The agglomerates were removed from the heel of the slurry container, manually crushed to a fine paste, and returned to the bulk slurry. The AP-105 simulant slurry feed was partitioned into eleven, 2.5-gallon buckets so that each contained approximately 2 gallons or 12.5 kilograms of slurry.

2.3.2 AP-105 Waste Feed for RPL Testing

The AP-105 actual tank waste supernatant was treated for cesium removal (Fiskum et al. 2018) and 12.4 L of the resulting effluent were available for processing in the CLSM. The GFCs necessary to form "AP-105 SPRN" slurry feed from the volume of the treated AP-105 tank waste are given in Table 2.4. This formulation was also checked against WTP baseline glass models documented in Kim et al. (2012). The GFCs were batched at APEL, transported to the RPL, and combined with the pretreated AP-105 actual tank waste supernatant to make the final slurry feed at a glass yield of 635 g-glass L-slurry^{-1}. Two separate batches were produced, each approximately 8 L, and stored in 2.5-gallon buckets at room temperature while being continuously stirred in a fume hood until melting operations commenced.

Table 2.4. Component Mass Added to 12.4 L of AP-105 Actual Tank Waste

Component	Mass (g)	Source
Kyanite	596.36	Kyanite Mining
Boric Acid	1822.19	Alfa Aesar
Wollastonite	466.05	NYCO Minerals
Iron Oxide	534.85	JT Baker
Olivine	309.92	Unimin Company
Silica	3769.88	US Silica
Rutile Sand (TiO_2)	132.17	Chemalloy
Zinc Oxide	357.22	Zinc Corp. of America
Zircon Flour	461.85	Prince Minerals
Sucrose	673.32	C & H Sugar Company

2.4 Sample Analysis Methods

Process samples collected for analysis from the APEL and RPL CLSM runs included the feed slurry, glass product, off-gas condensate, HEPA filters, and off-gas line solids deposits.

Table **2.5** identifies the sample matrices, target frequency of sample collection, and desired analyses to be conducted. Table 2.6 lists the methods for the analyses listed in Table 2.5.

Table 2.5. Process Samples, Sampling Frequency, and Sample Analyses

Sample Matrix	Size	Frequency for Obtaining Samples	Analyses
Feed Slurry Blend	≥10 mL	Beginning of each feeding segment	Cation Anion Density Weight %
Glass Product	≥10 g	See Table 2.6	Cation Anion Density Crystal Mass
Off-gas Pipe Accretions	All	At the end of testing	Cation Anion Mass
Off-gas Condensate	≥10 mL	At the beginning and end of every feeding segment	Cation Anion Mass pH
SBS Sample Loop Liquid	≥10 mL	At the end of each sampling period	Cation Anion Mass pH
HEPA Filters	4 Pairs	At the end of testing	Cation Anion Mass

Table 2.6. 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, Re, or Cs)	Al, Ba, B, Ca, Cd, Cr, Co, Cs, Cu, Fe, La, Li, K, Mg, Mn, Mo, Na, Ni, P, Pb, Re, Si, Sr, S, Sn, ^{99}Tc, Ti, W, V, Y, Zn, Zr
Anions	Solid or Liquid	IC or Ion-Specific Electrode	Chloride, Chromate, Fluoride, Nitrate, Nitrite, Phosphate, and Sulfate
Mass	Solid or Liquid	---	Weigh on scale
Weight %	Solid or Liquid	---	Dry in oven and weigh on scale
Density	Solid	---	Liquid displacement, or pycnometry
pH	Liquid	---	Compare with pH paper
Density	Liquid	Mass/Volume	Weigh known volume
Crystal Evaluation	Solid	XRD	Analyses of crystal types and quantity

IC = ion chromatography; ICP-AES = inductively coupled plasma atomic emission spectroscopy; ICP-MS = inductively coupled plasma mass spectrometry; XRD = X-ray diffraction.

Although feed, glass, and off-gas samples (solids and condensate) were to be collected according to the frequency and schedule indicated in Table 2.5 and Table 2.7, only those samples considered the most representative of ideal test conditions were analyzed. However, analysis at a minimum included one sample of feed, glass, off-gas stream (HEPA), and off-gas condensate from each test. All chemical analysis of the samples produced from the APEL and RPL CLSM runs was performed by Southwest Research Institute (SwRI). The analytical method and elements measured by each method are given in

Table **2.8**, which also indicates whether the method was used to analyze the APEL or RPL samples.

Table 2.7. Sampling Schedule During Feeding Operations

	Sample Frequency	Size
Glass Samples	Sample all glass during the pressure-assisted pouring period. Planned pouring periods should occur approximately every 30 minutes.	Contents of each pour placed in small metal canisters with lid.
Condensate Samples	Collect condensate during each pouring period or whenever the condensate collector is full.	All of accumulated melter off-gas condensate.
Sample Loop SBS Liquid Samples	Collect liquid from sample loop SBS at the end of each sampling period.	All of accumulated SBS liquid.
HEPA Filter Samples	At the end of each test.	Up to eight HEPA filters.
Off-Gas Line Wash	At the end of each test.	Accumulated liquids/solids from a wash out of the off-gas line from the melter vessel lid to sample HEPA filters and primary SBS.

Table 2.8. Chemical Analyses of Product Samples to be Performed by SwRI

Analysis Method	Elements Measured	APEL	RPL
ICP-AES	Al, Ba, B, Ca, Cd, Cr, Co, Cu, Fe, La, Li, K, Mg, Mn, Mo, Na, Ni, P, Pb, Si, Sr, S, Sn, Ti, W, V, Y, Zn, Zr	X	X
ICP-MS	Re	X	
ICP-MS	^{99}Tc and Cs		X
IC	Chloride, Chromate, Fluoride, Nitrate, Nitrite, Phosphate, and Sulfate	X	X
Alpha Spectrum	Am-241, Cm-242, Cm-244, Np-237, Pu-238, Pu-239/240, and Pu-244		X

Additional analysis of the simulant feed slurry included feed density measurement via a graduated cylinder and water content measurement performed by drying two aliquots of slurry in an oven at 105 °C overnight. The pH levels of all condensate samples sent for chemical analysis was measured with pH paper over the 0-14 range. The crystal fraction of glass products from the APEL runs was measured by x-XRD using a Bruker D8 Advance diffractometer (Cu Kα) equipped with a LynxEye™ position-sensitive

detector with a collection window of 3° 2θ. The scan parameters used were 5-70° 2-θ with a step size of 0.009° 2-θ and a 1 second dwell time at each step.

2.5 System Modifications

The CLSM system at APEL was used as the proof-of-process test bed. System and process modifications were made at APEL first and transferred to the CLSM at RPL before radioactive processing commenced. This allowed optimization in a non-radioactive laboratory environment so that changes at RPL could be implemented with higher confidence. Commissioning tests were performed in the fall 2017 and winter 2017/2018 with feed slurry simulant compositions AP-105, AN-102, and AN-105, which had been used in previous melter testing (Matlack et al. 2011 and 2017). Feed system plugging was evident early in these informational runs, resulting in termination of most of the tests. As a result, feed slurry pump tests commenced with only the feed system operational to determine the cause of the plugging and develop solutions for this issue. Numerous configuration changes were made in addition to enhancements to system operation and flushing procedures. The most significant changes were elimination of tight 90° bends, increasing slurry line sizes, optimizing line slopes, optimizing the feed suction tip, and removing a pressure transducer, that offered high flow resistance. These changes, in addition to optimized flushing points and flush procedures, resulted in the ability to pump 8 L of feed slurry into the CLSM vessel without plugging.

After feed system troubleshooting was complete, hot operations resumed to further troubleshoot the CLSM. During these runs, only AP-105 simulant feed slurries were used and the tests conducted again under for-information-only (FIO) laboratory quality procedures. The majority of hardware changes required during these tests were related to solving plugging in the off-gas line between the melter and the SBS. This was solved with a larger off-gas pipe size and a design modification to allow a mechanical scraper to be manually actuated in the case of a plug in the off-gas piping around the melter lid or sampling switch. Other minor changes to the placement of lid components were necessary to either optimize cold-cap behavior or reduce cold-cap bridging. During this time, the staff also gained valuable operations experience and developed formal operating procedures and Test Instructions to prepare for formal simulant testing.

3.0 Run Descriptions and Results

3.1 Non-radioactive Simulant Testing at APEL

Four CLSM test runs were completed at APEL between February 22 and March 14, 2018. Testing was conducted to implement a Test Plan prepared by PNNL and approved by WRPS (PNNL 2018). During each run, glass product, off-gas particulate, off-gas condensate, and feed slurry were collected for analysis. After each run, the system was cleaned by washing out the solid particulate that had collected on the off-gas piping and these washes were collected for analysis. The target operating parameters were the same for all of the APEL CLSM runs 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, ^{o}C	1150
Plenum temperature range, ^{o}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 rates were largely established by cold-cap conditions. During feeding operations, the target glass production range of between 1500 to 2000 kg m^{-2} d^{-1} was controlled by adjusting 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 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 tube that was submerged in the melter vessel glass pool. The actual flow rate used within this range was chosen on the basis of operational stability and compatibility with other operational constraints such as melter plenum temperature and vacuum.

The three initial CLSM test runs contributed to the system modifications described in Section 2.5 and were performed according to the associated Test Instructions (Dixon 2018a,b,c). During these test runs, feed processing was intermittent and target production rates were not achieved. As a result, the samples collected from these test runs were not sent for analysis. The following results are from the fourth CLSM test run, performed on March 14, 2018.

3.1.1 CLSM Processing Results

To begin the fourth CLSM test run (Dixon 2018d), the CLSM vessel was loaded with 1.8 kg of previously prepared glass pieces designed after the WDFL1 formulation and the furnace surrounding the CLSM vessel was heated to 1250 °C. When the thermocouple located in the glass melt read 1150 ± 30 °C, feeding of the AP-105 feed slurry into the melter was allowed to begin. Feeding began at 11:38 AM and was concluded at 6:09 PM with intermittent interruptions as will be described. The total feeding time, mass of slurry 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 glass temperature and plenum temperature during the test run are shown in Figure 3.1. 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-105 Simulant Feed Slurry

Parameter	**Result**
Test Date and Feeding Duration, h	3/14/18 5.83
Glass Produced, kg	4.322
Slurry Consumed, kg	10.840
Average glass production rate, kg m^{-2} d^{-1}	1574
Average feeding rate, kg h^{-1}	1.86
Average bubbling rate, L m^{-2} min^{-1}	101
Average glass temperature, ^{o}C	1137
Plenum temperature range, ^{o}C	350 – 850
Plenum vacuum range, in-H_2O	0 – 5
SBS temperature range, °C	14 – 38

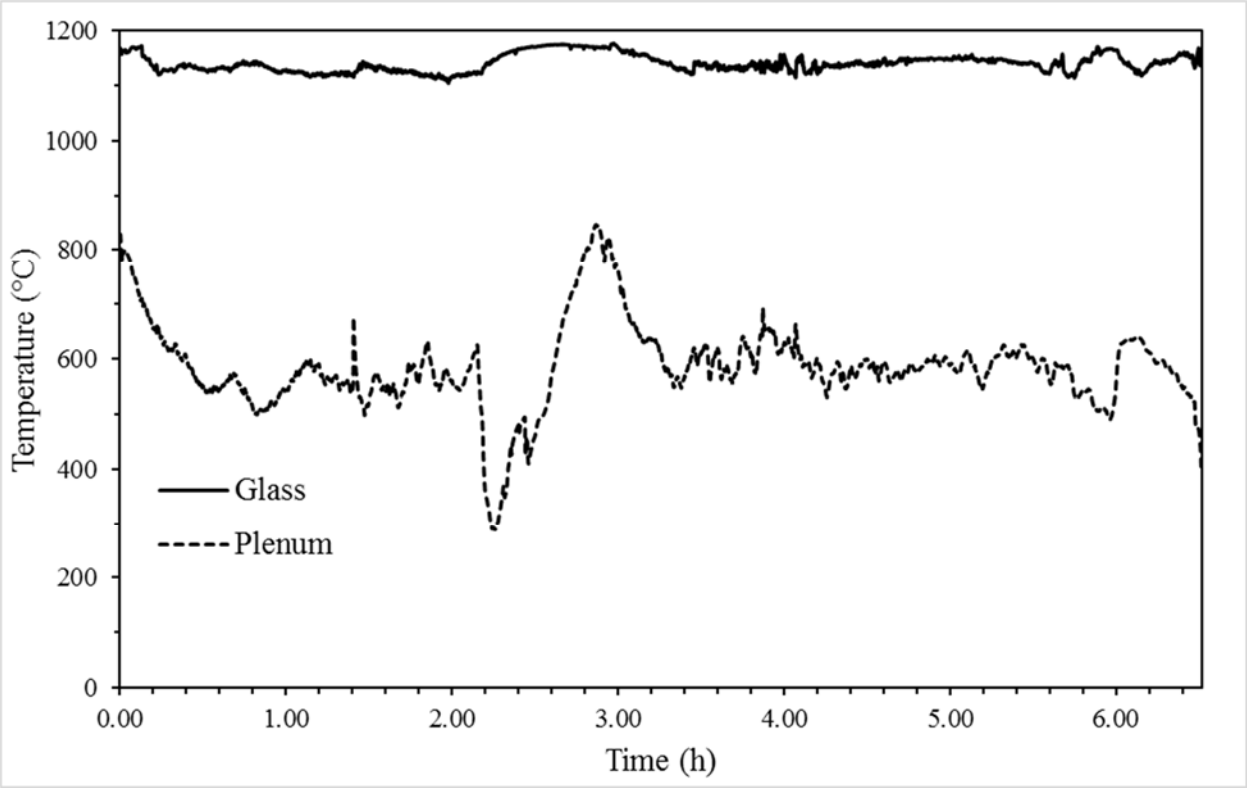

Figure 3.1. Glass and plenum temperature from CLSM run with AP-105 simulant feed slurry.

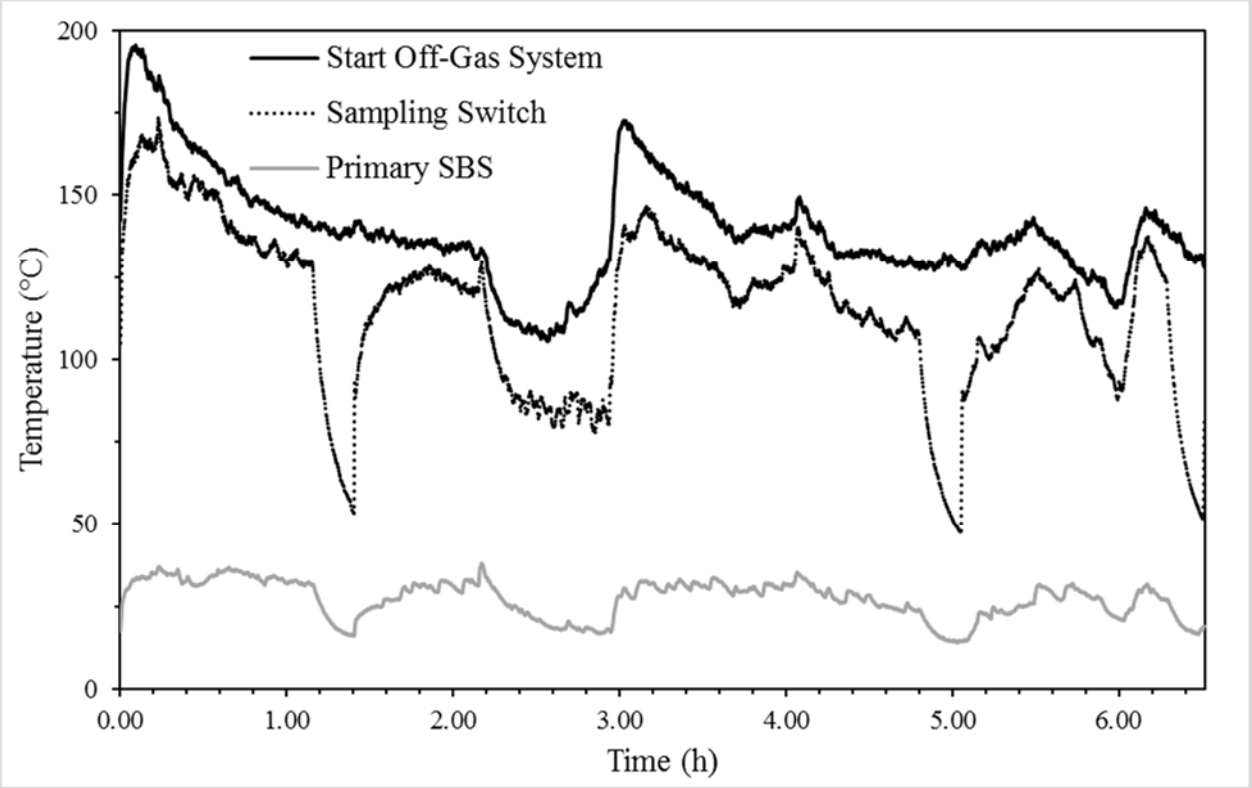

Figure 3.2. Start of off-gas system, sampling switch, and primary SBS temperature from CLSM run with AP-105 simulant feed slurry.

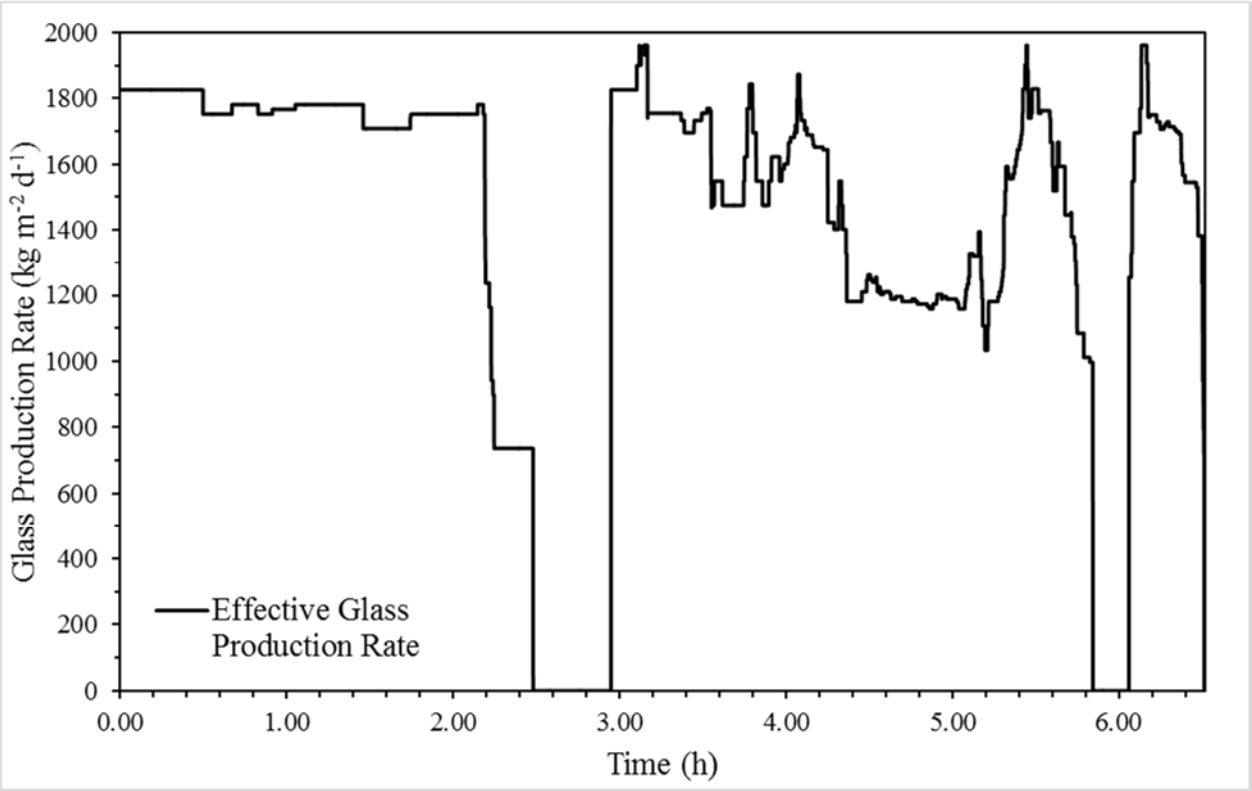

Figure 3.3. Effective glass production rate from CLSM run with AP-105 simulant feed slurry.

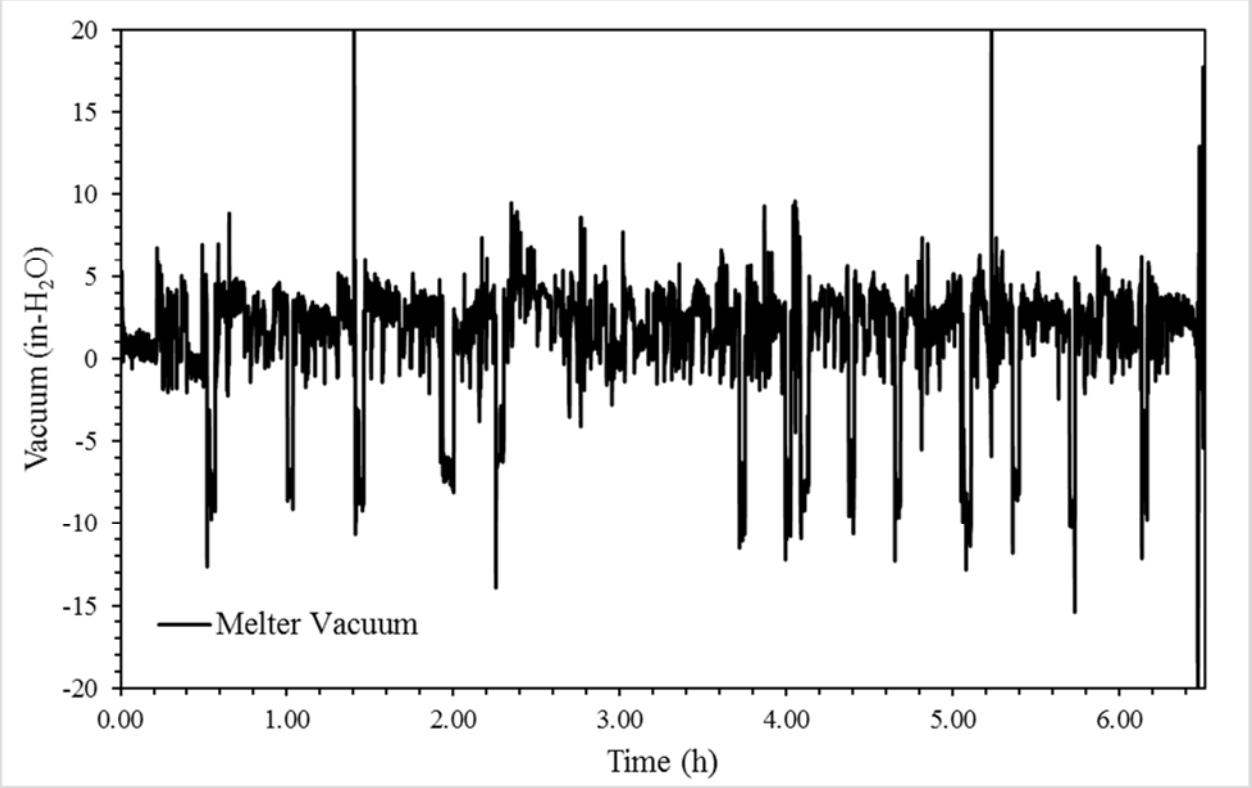

Figure 3.4. Melter vacuum from CLSM run with AP-105 simulant feed slurry.

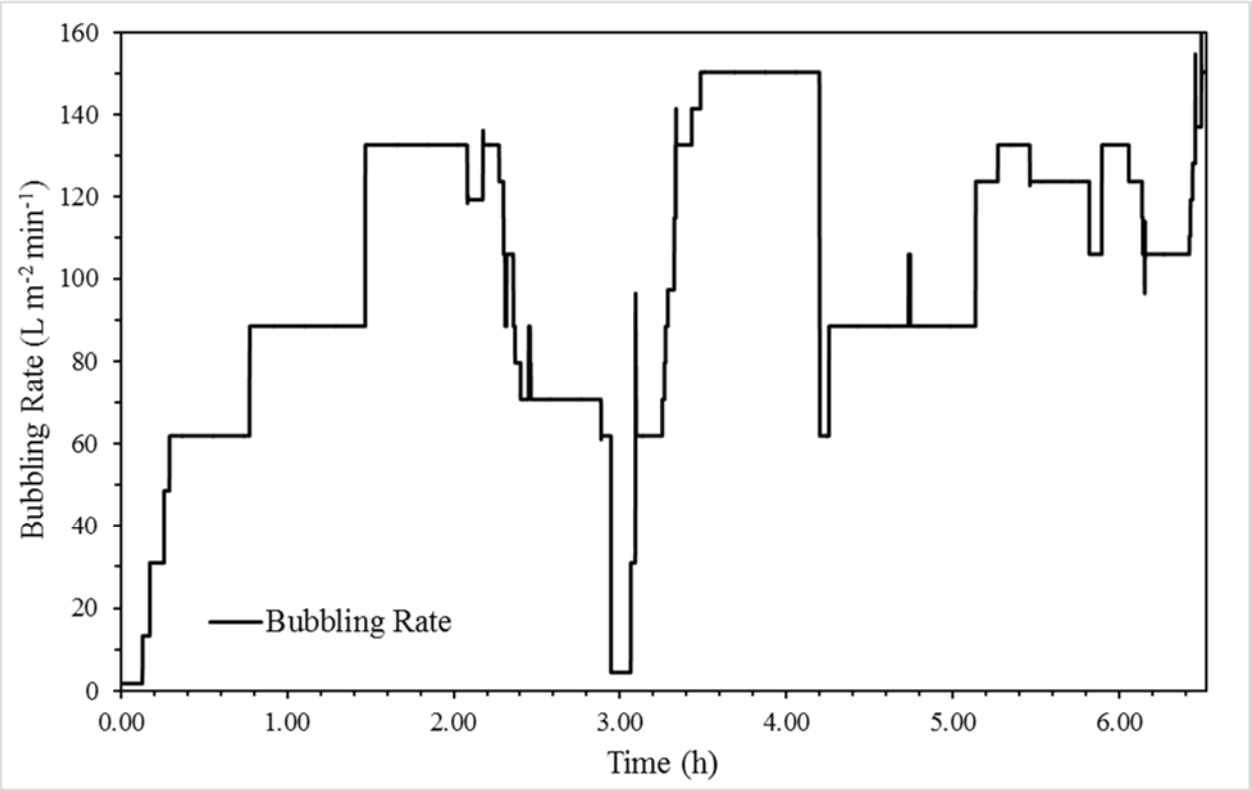

Figure 3.5. Bubbling rate from CLSM run with AP-105 simulant feed slurry.

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 from CLSM Run with AP-105 Simulant Feed Slurry

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	3/14/2018	12:47:38 PM	1.16	1:02:32 PM	1.40	14.90
2	3/14/2018	4:26:09 PM	4.80	4:41:22 PM	5.05	15.22
3	3/14/2018	5:55:30 PM	6.29	6:08:36 PM	6.51	13.10

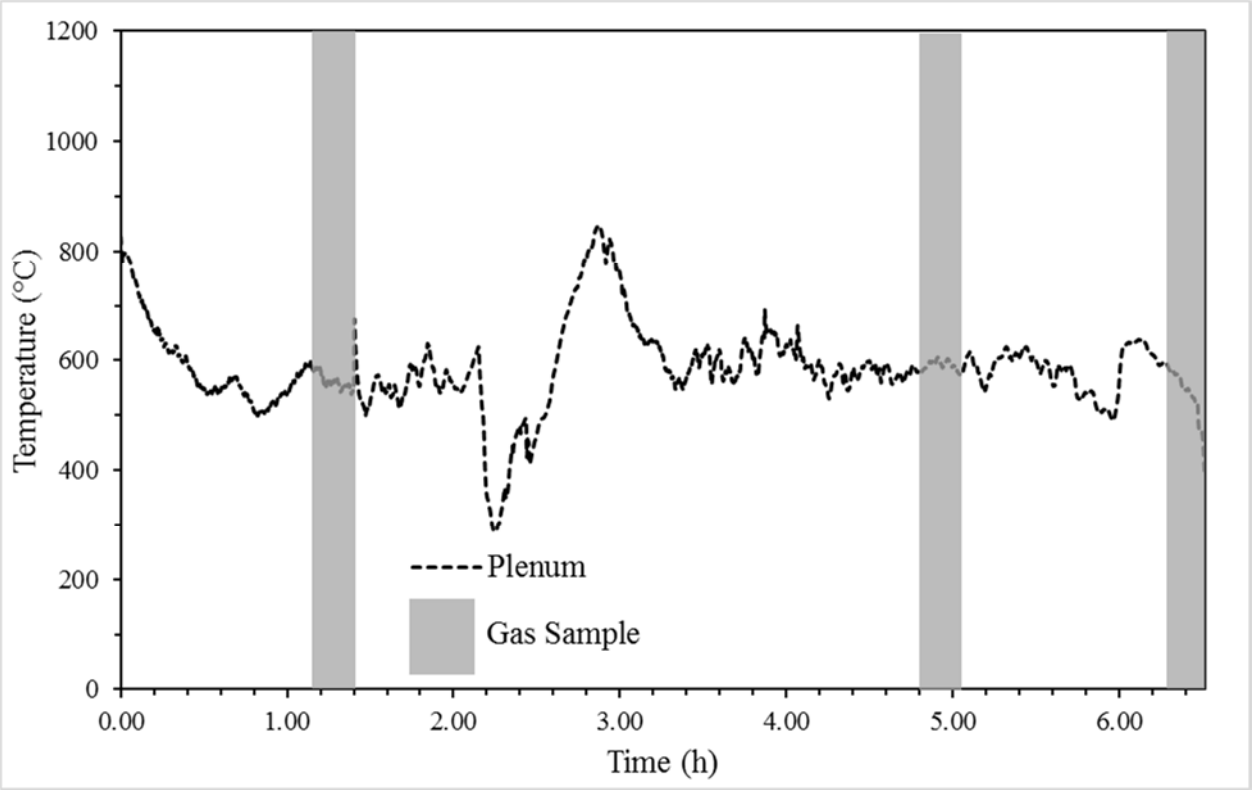

Figure 3.6. Plenum temperature as a function of time for the CLSM run with AP-105 simulant feed slurry, showing timing and durations of gas-sampling events.

The timing and mass of each periodic glass pour are shown in Table 3.4, which correspond with the times that the melter vacuum was set in the range of -5 to -10 in-H_2O in Figure 3.4. In some instances, glass was poured from the melter before the glass from the previous pour could be cleared from the glass catch bin, resulting in both glass pours being handled together.

Table 3.4. Timing and Mass of Glass Pours from CLSM Run with AP-105 Simulant Feed Slurry

Glass Pour Time			Glass Pour Mass
h			g
		0.00	180.20
		0.51	148.53
		0.99	128.00
		1.41	219.91
		1.93	284.53
2.24	&	2.38	723.64
		3.71	201.10
		3.99	180.96
		4.08	210.88
		4.36	197.38
		4.65	204.64
5.06	&	5.36	547.18
		5.70	221.26
6.13	&	6.51	524.19
		6.51	2149.95

3.1.2 Feed Processing Characteristics

During the initial feeding period from hour 0.00 to 2.48, the foam layer in the cold cap was steadily growing, causing the reacting feed and boiling slurry of the cold cap moving away from the glass melt surface to a steadily higher level in the melter vessel plenum space. Beginning at hour 2.15, the reacting feed and boiling slurry portion of the cold cap came into contact with the thermocouple in the plenum space of the melter vessel, resulting in a sharp decrease in plenum temperature from 624.5 °C to 289.0 °C over the period from hour 2.15 to 2.26, shown in Figure 3.1 and Figure 3.6. To reduce the height of the cold cap, the feeding rate was reduced and eventually stopped at hour 2.48, shown in Figure 3.3, which caused the cold cap to completely burn off, resulting in melter idling conditions.

At hour 2.95, slurry feeding to the melter vessel was restarted. When visual inspection of the cold cap at hour 5.84 indicated that the cold cap height might again reach the plenum thermocouple, slurry feeding was stopped to prevent such an occurrence. The period of no feeding lasted from hour 5.84 to 6.06, during which time the cold-cap coverage was reduced to ~40%. The final feeding segment lasted from hour 6.06 to 6.51, after which the feeding line was flushed with water, the cold cap was allowed to burn off, all of the glass inventory was poured from the melter vessel, and the test was terminated.

3.1.3 Sample Analysis Results

A set of 20 samples that represented the entire CLSM run was sent for chemical analysis. These samples included six glass samples from pours throughout the run, one sample of the feed slurry, five HEPA filter samples (two sets of sample HEPA filters, two primary HEPA filters, and one blank HEPA filter), and eight liquid samples (two condensate samples, one primary SBS liquid sample, one sample SBS liquid sample, and four liquid samples from washing the off-gas system). The value for each sample mass and the resulting concentration of each analyzed cation and anion are given in Table 3.5. Where values were left blank, the values were below the analysis detection limit.

Table 3.5. Chemical Analysis of Selected Samples from the CLSM Run with AP-105 Simulant Feed Slurry

Sample Name	Sample Type	Sample Mass	Rhenium	Aluminum	Barium	Boron	Calcium	Chromium	Cobalt	Copper	Iron	Lanthanum	Lead	Lithium
		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}	mg kg^{-1}
Glass Pour 0.51	Glass	148.53	5.12	32900	8.98	32000	15600	2090	7.02	6.34	39600	8.91	27.6	336
Glass Pour 1.41	Glass	219.91	5.62	32700	8.42	28200	15350	1735	7.43	6.25	39450	7.20	27.4	224
Glass Pour 3.99	Glass	180.96	5.54	31900	7.76	25700	14700	1350	7.24	6.50	38100	4.59	26.1	114
Glass Pour 5.36	Glass	547.18	5.53	33000	7.62	31600	15100	1190	7.31	6.67	39200	3.94	27.8	88.8
Glass Pour 6.13	Glass	524.19	5.91	32300	7.53	30400	14900	1070	7.09	6.42	38600	3.52	26.7	66.2
Glass Pour 6.51	Glass	2149.95	6.55	32500	7.35	26400	15200	1050	6.82	6.26	39100	3.34	26.8	59.4
Feed Slurry	Feed	10840.00	5.73	6900	2.98	14400	6320	225		1.98	15900		11.1	
Blank HEPA Filter	HEPA	5.06		12000	15600	10600	4540				158		2.45	11.2
Sample HEPA filter 1	HEPA	10.51	80.5	11600	15200	11500	4385	72.4		1.18	288		3.10	6.20
Sample HEPA filter 2	HEPA	14.15	52.1	8710	11300	9970	3250	67.4		1.13	238		1.91	
Primary HEPA filter 1	HEPA	5.43	42.1	11000	14500	12200	4170	55.4			164		2.38	
Primary HEPA filter 2	HEPA	6.57	128	9580	12000	10300	3660	102			160		3.45	
Condensate Sample 1	Liquid	6024.24	2.38	17.5		151	10.8	6.46			27.2			
Condensate Sample 2	Liquid	6024.24	2.42	18.1		135	11.0	6.48			26.9			
Primary SBS Liquid	Liquid	976.61	3.76			190	6.89	7.72			7.45			
Sample SBS Liquid	Liquid	658.62	3.79			189	6.82	7.77			7.38			
Total Line Wash	Liquid	92.98	7.96	62.9		307	47.9	40.8		0.256	155			
Primary Line Wash	Liquid	172.81	8.88	174		518	179	31.2			400			
Sample Line Wash	Liquid	170.71	2.75	61.3		243	38.2	6.76			148			
Pre-Primary HEPA Liquid	Liquid	322.18	0.893	13.8		52.7	7.74	4.17			36.5			

Table 3.5. Chemical Analysis of Selected Samples from the CLSM Run with AP-105 Simulant Feed Slurry (cont.)

Sample Name	Magnesium	Manganese	Molybdenum	Nickel	Phosphorus	Potassium	Silicon	Sodium	Strontium	Sulfur	Tin	Titanium	Tungsten	Vanadium
	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}	mg kg^{-1}
Glass Pour 0.51	8150	114	6.10	2690	68.7	3570	207000	147000	13.1	1020	10.6	7850	255	60.8
Glass Pour 1.41	8255	111	4.63	2500	60.6	3495	206000	145500	12.9	958	11.4	7800	263	60.6
Glass Pour 3.99	8190	108	4.22	2040	83.6	3480	206000	145000	12.4	1140	11.1	7600	270	60.6
Glass Pour 5.36	8340	106	3.12	1700	32.5	3600	209000	151000	12.6	1120	10.3	7840	269	60.4
Glass Pour 6.13	7920	105	3.32	1420	52.3	3590	208000	149000	12.5	1100	11.4	7670	272	59.8
Glass Pour 6.51	7450	103	3.79	1310	65.2	3620	209000	151000	12.4	1170	10.6	7640	258	59.1
Feed Slurry	1770	36.4		30.8	153	1590	117000	64100	5.10	607		847	120	5.21
Blank HEPA Filter	742	3.61	17.6	2.89		8270	398000	23800	168	158		17.9	127	
Sample HEPA filter 1	710	5.29	18.7	8.06		9280	381000	37150	160	675		20.0	127	
Sample HEPA filter 2	524	12.2	12.6	5.97		6320	303000	24900	118	447		16.2	79.5	
Primary HEPA filter 1	681	3.32	16.5	2.81		7820	361000	24700	155	386		16.5	118	
Primary HEPA filter 2	594	3.65	13.8	7.46		7950	300000	38000	126	1030		13.8	103	
Condensate Sample 1						38.3	29.5	695		32.5		0.878		
Condensate Sample 2						38.2	29.8	692				0.913		
Primary SBS Liquid						62.5	9.74	1060		40.6				
Sample SBS Liquid					6.70	62.2	9.92	1080		43.1				
Total Line Wash		0.903	1.70	5.05	10.2	117	134	2540		131		4.48		
Primary Line Wash	5.95	0.856		1.00	11.0	161	271	3080		119		15.9	1.83	
Sample Line Wash		0.794	0.882	3.07		45.7	77.9	1040		24.4		2.98		
Pre-Primary HEPA Liquid		0.282		0.928		15.2	39.4	274				1.13		

Table 3.5. Chemical Analysis of Selected Samples from the CLSM Run with AP-105 Simulant Feed Slurry (cont.)

Sample Name	Yttrium	Zinc	Zirconium	Chloride	Sulfate	Chromate	Fluoride	N (Nitrate)	N (Nitrite)	P (Phosphate)
	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 0.51	46.1	24700	18200	11.4	13.8	4.36				
Glass Pour 1.41	46.1	24900	18150	14.5	18.7	4.36				
Glass Pour 3.99	46.1	24100	18000	14.6	19.5	6.51				
Glass Pour 5.36	46.4	24800	18600	15.7	17.2	6.12				
Glass Pour 6.13	46.3	24400	18000	14.9	15.2	5.43				
Glass Pour 6.51	45.7	24800	17900	15.5	19.2	5.78				
Feed Slurry	3.31	10100	33.9	1950	1730	427	21.5	12100	8760	54.5
Blank HEPA Filter	2.74	12000	134	4.02	34.2					
Sample HEPA filter 1	2.70	11600	132	14550	1260	89.3	69.6	651	341	
Sample HEPA filter 2	1.96	8540	101	12900	1090	37.9	49.9	1430	788	
Primary HEPA filter 1	2.55	10900	124	80.6	1130		1.96	10900		
Primary HEPA filter 2	2.03	9510	100	246	3050		1.99	38000	2.86	3.27
Condensate Sample 1		33.1	1.00	539	169		9.97	1440		
Condensate Sample 2		33.3	0.998	567	153		10.8	1440		
Primary SBS Liquid		8.32		921	137	19.3	15.5	371	966	
Sample SBS Liquid		8.37		928	138	19.2	16.1	374	958	
Total Line Wash		85.2	4.53	2600	402	73.6	72.9	80.4	14.3	
Primary Line Wash		273	14.0	2280	334	49.3	76.6	100	135	
Sample Line Wash		105	2.59	541	70.1	8.47	20.9	80.7	114	
Pre-Primary HEPA Liquid		18.1	1.59	452	116		8.52	12700	14.8	

The measured solids content of the AP-105 simulant feed slurry was 53% and the density was 1.55 g mL^{-1}. For comparison, measured values for AP-105 simulant feed slurry prepared by Matlack et al. (2017) were solids content of 51% and density of 1.49 g mL^{-1}. The pH levels of each analyzed liquid sample was measured with pH paper and the results are listed in Table 3.6. The XRD spectrum for all the analyzed glass samples demonstrated an amorphous structure with no crystals present with a representative spectrum shown in

Figure **3.7**.

Table 3.6. pH of Analyzed Liquid Samples from the CLSM Run with AP-105 Simulant Feed Slurry

Liquid Sample Names	pH
Condensate Sample 1	~2
Condensate Sample 2	~2
Primary SBS Liquid	~7
Sample SBS Liquid	~9
Total Line Wash	~10
Primary Line Wash	~10
Sample Line Wash	~9
Pre-Primary HEPA Liquid	<0.5

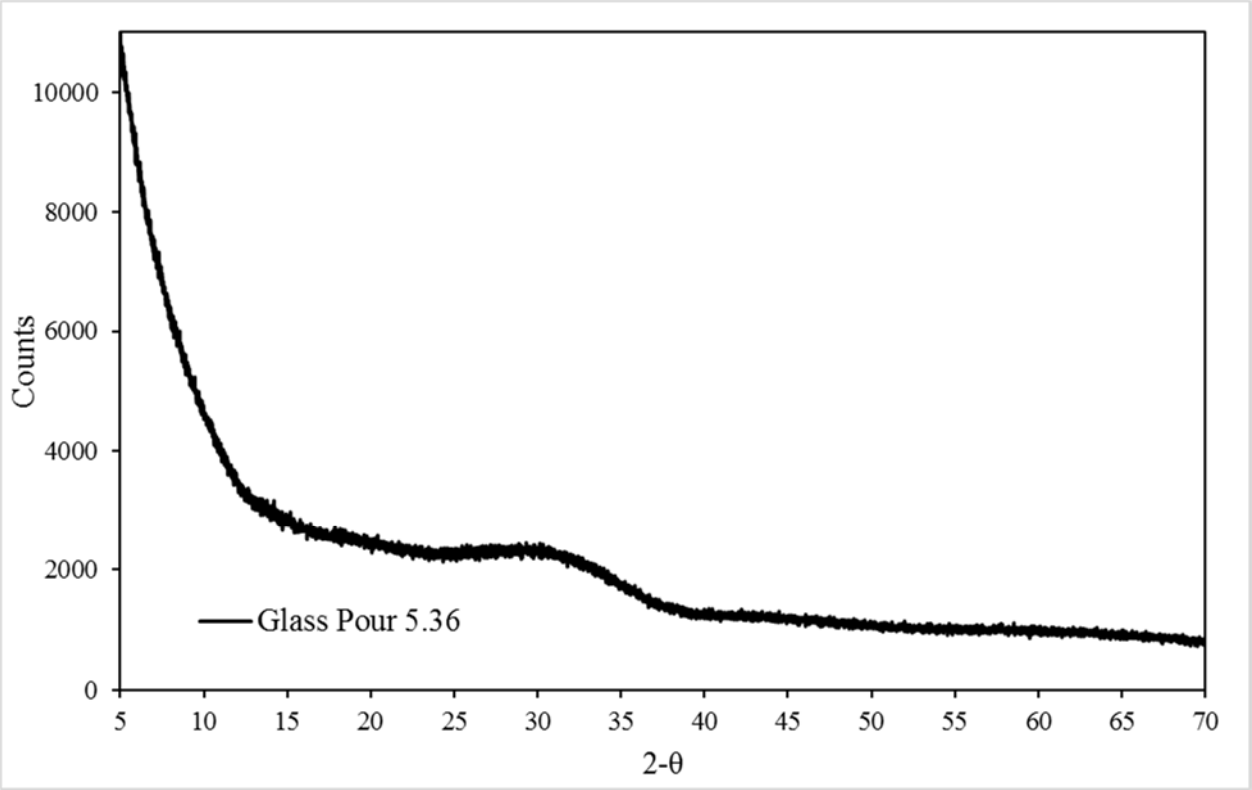

Figure 3.7. XRD spectrum of glass pour 5.36 showing an amorphous glass structure without crystals.

3.2 Radioactive Waste Testing at RPL

Two CLSM test runs were conducted at RPL between March 28 and April 12, 2018. In each run, glass product, off-gas particulate, off-gas condensate, and feed slurry were collected for analysis. The target operating conditions were the same for both RPL CLSM runs and are presented in Table 3.1. As with the CLSM system at APEL, the maximum steady-state processing rates were largely dictated 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 feed rate and bubbling rate to maintain the target cold-cap coverage of 75% to 95%. To achieve specific processing rates within this 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 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 to 3000 sccm to a high-temperature 600 nickel tube that was submerged in the melter vessel glass pool. The actual flow rate used within this range was chosen on the basis of operational stability and compatibility with other operational constraints such as melter plenum temperature and vacuum.

The initial test at RPL was conducted on March 28, 2018 with simulated feed slurry to fulfill requirements of a Test Plan prepared by PNNL and approved by WRPS (PNNL 2018a). The test run was performed FIO, and as such, the samples collected during feeding were not sent for analysis. The primary purpose of this test was to conduct system and process shakedown/troubleshooting. The feed slurry used for this test was a part of the AP-105 feed simulant that was prepared as described in Section 2.3.1. The

test duration was approximately 9 hours, including initial system checks and the operational shutdown steps. The total mass of glass produced was approximately 1.4 kg over 2.44 hours of feeding. Throughout testing, the glass temperature was lower than the target goals and required frequent adjustments of the newly installed furnace. The test was terminated due to a feed line plug on the discharge side of the pump. It was later determined that solids had accumulated in a flush line, preventing flushing of the feed line. The flush line was re-routed after the test to eliminate low spots where solids collected.

The following results are from the second CLSM test run performed on April 5, 11, and 12, 2018 to fulfill a Test Plan prepared by PNNL and approved by WRPS (PNNL 2018b) in association with a test instruction (Venarsky 2018).

3.2.1 CLSM Processing Results

To begin the CLSM test run, the CLSM vessel was loaded with 1.8 kg of previously prepared glass pieces designed after the WDFL1 formulation and the furnace surrounding the CLSM vessel was heated to 1250 °C. When the thermocouple located in the glass melt read 1150 ± 30 °C, feeding of the AP-105 feed slurry into the melter was allowed to begin. Feeding began at 5:23 PM on April 5, 2018, and was concluded at 8:42 AM on April 12, 2018, with intermittent interruptions as described in Section 3.2.2. The total feeding time, mass of glass produced, and average values over the course of feeding for several key processing components are given in

Table **3.7**. The mass of slurry could not be measured due to radioactive contamination restrictions; thus, the amount of slurry consumed and the average feeding rate could not be calculated. The glass temperature and plenum temperature during the test run are shown in Figure 3.8. The occasional, sharp decreases and increases in the glass temperature (from hour 3.18 to 3.54 and from hour 14.88 to 17.99) were due to apparent electrical interference in the thermocouple wiring, as a secondary, separate reading of the glass thermocouple from a handheld device displayed glass temperatures in line with the expected trends of the values. During the time range from hour 1.91 to 2.84, the processing data were set to 0 due to a CLSM system shutdown, as described in Section 3.2.2. The temperature 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 3.9. 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 3.7 and is shown in Figure 3.10. The melter vacuum values during the test run are shown in Figure 3.11. The anomalous vacuum readings from hour 13.54 to 17.33 were a result of feed particulate blocking the pressure sensor, but not the vacuum flow, as described in Section 3.2.2. The bubbling rate during the test run is shown in Figure 3.12.

Table 3.7. CLSM Production Values for AP-105 Waste Feed Slurry

Parameter	Result
Test Date and Feeding Duration, h	4/5/18 4/11/18 4/12/18 15.09
Glass Produced, kg	9.45
Slurry Consumed, kg	NC
Average glass production rate, kg m^{-2} d^{-1}	1330
Average feeding rate, kg h^{-1}	NC
Average bubbling rate, L m^{-2} min^{-1}	125
Average glass temperature, °C	1122
Plenum temperature range, °C	400 – 900
Plenum vacuum range, in-H_2O	0 – 10
SBS temperature range, °C	13 – 38

NC = not calculated

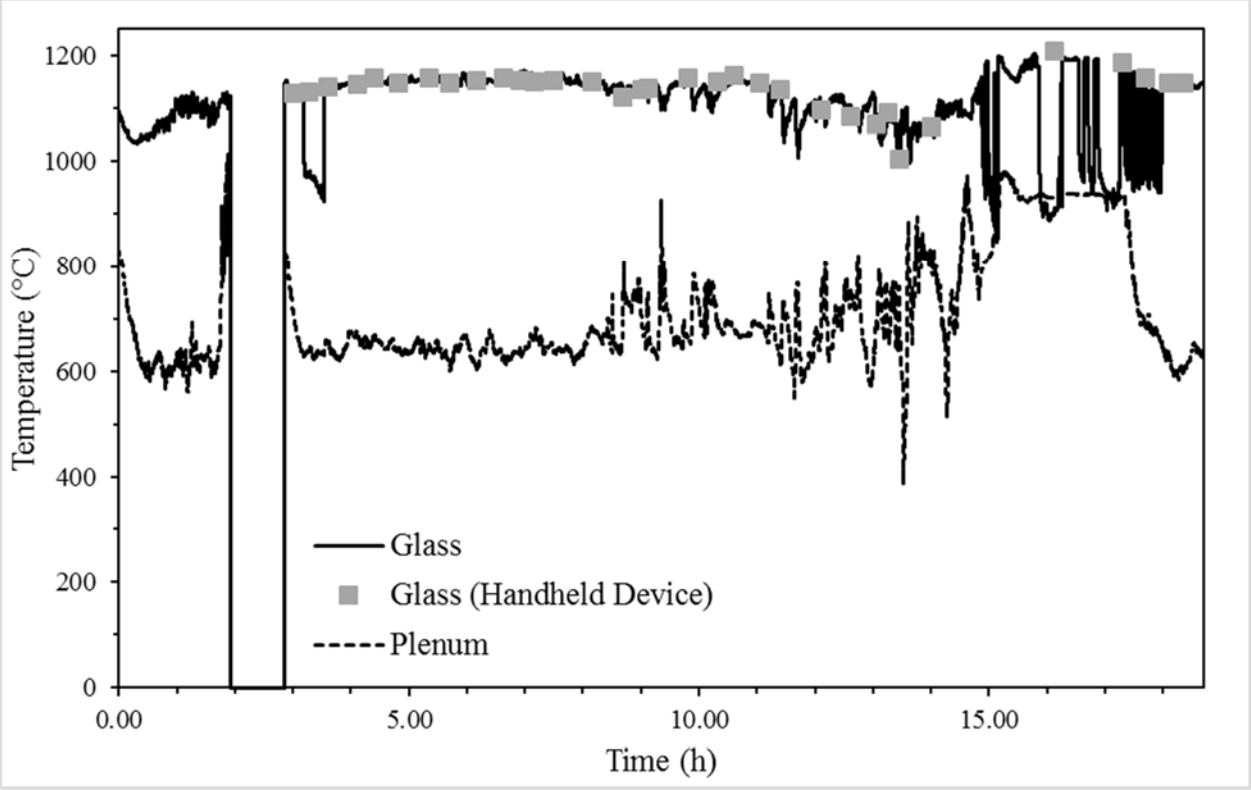

Figure 3.8. Glass and plenum temperature from CLSM run with AP-105 waste feed slurry.

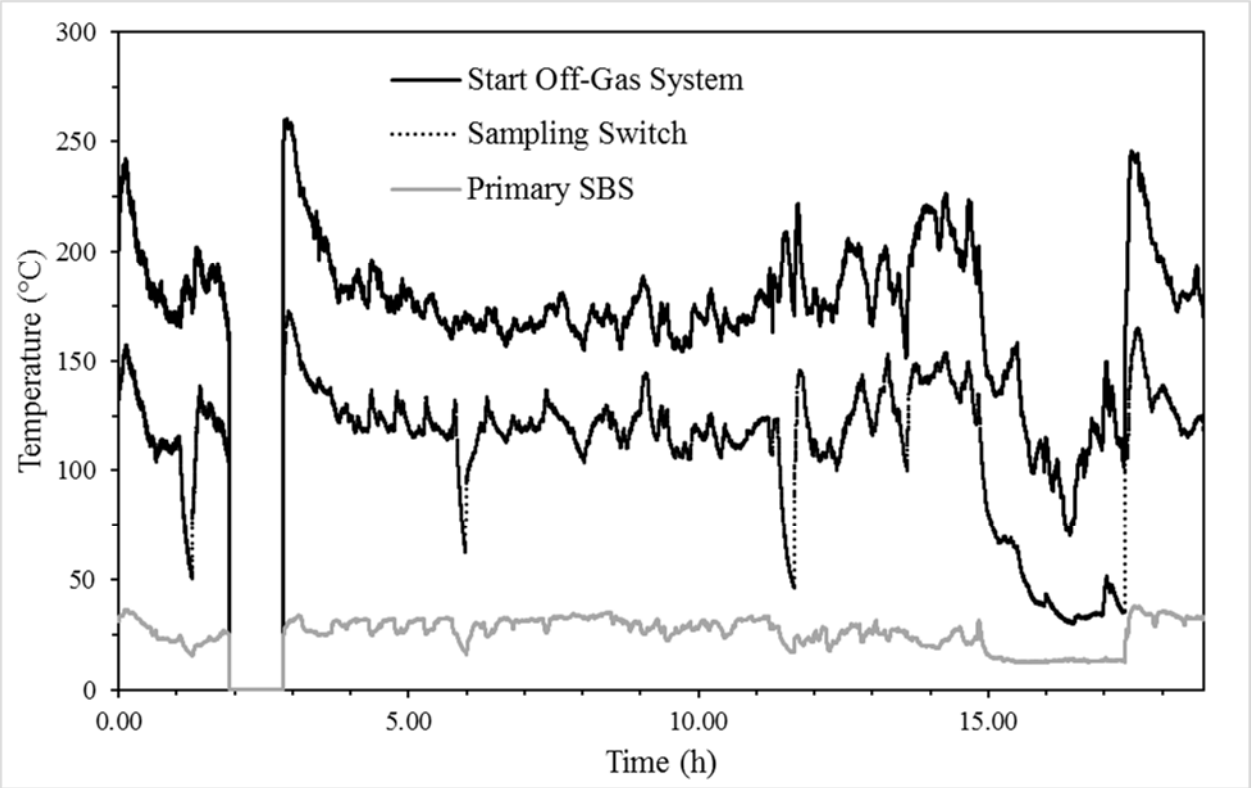

Figure 3.9. Start of off-gas system, sampling switch, and primary SBS temperature from CLSM run with AP-105 waste feed slurry.

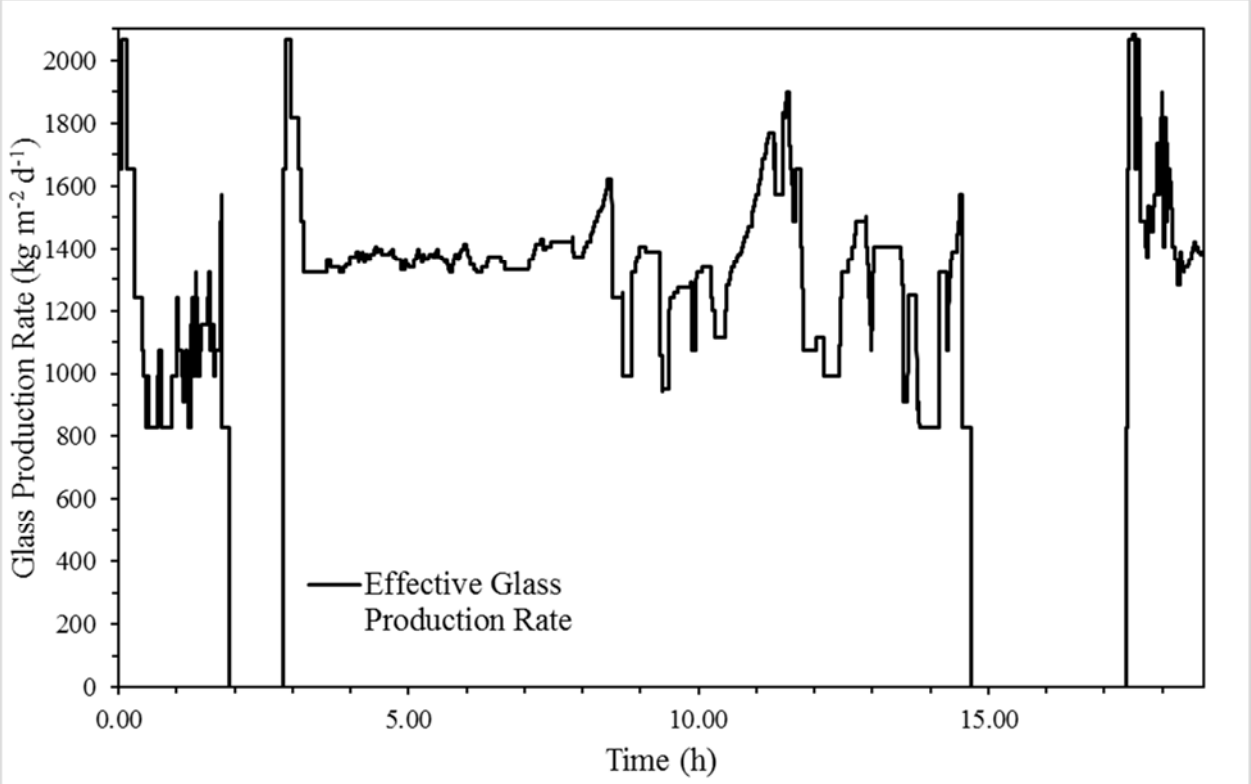

Figure 3.10. Effective glass production rate from CLSM run with AP-105 waste feed slurry.

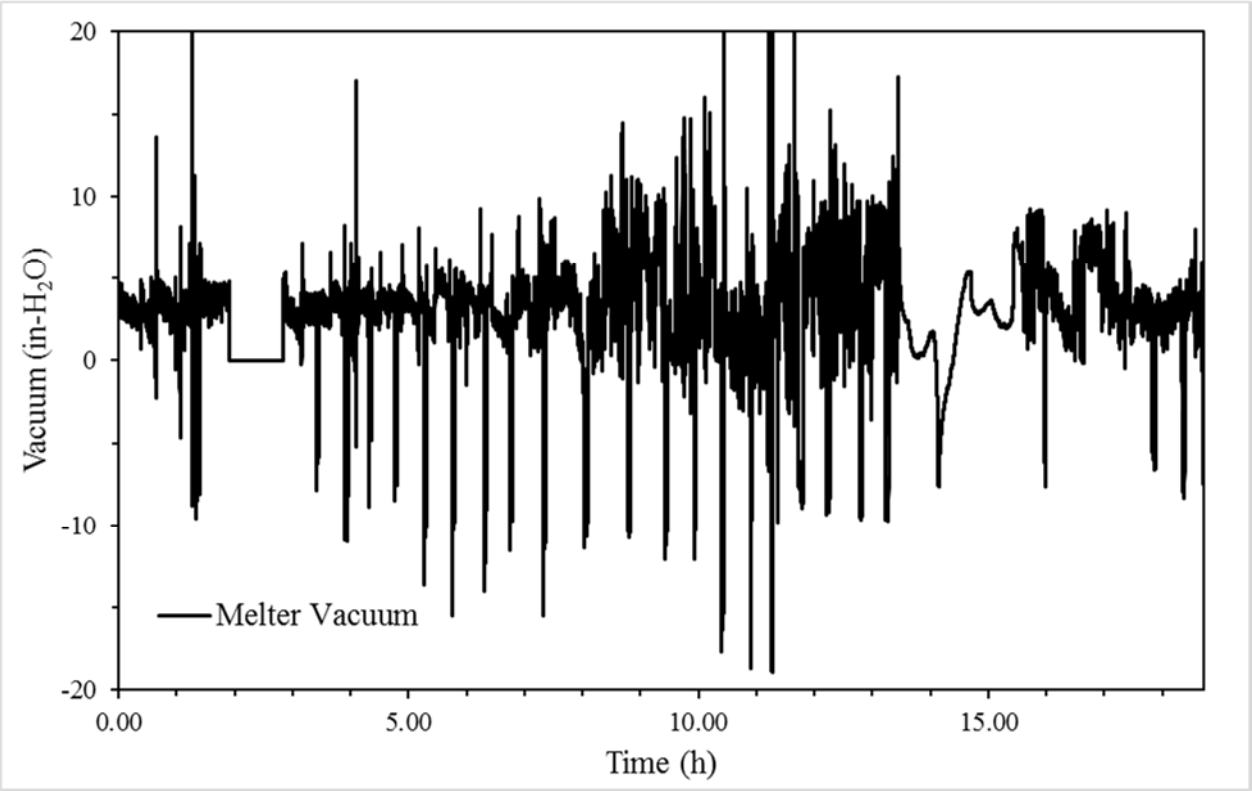

Figure 3.11. Melter vacuum from CLSM run with AP-105 waste feed slurry.

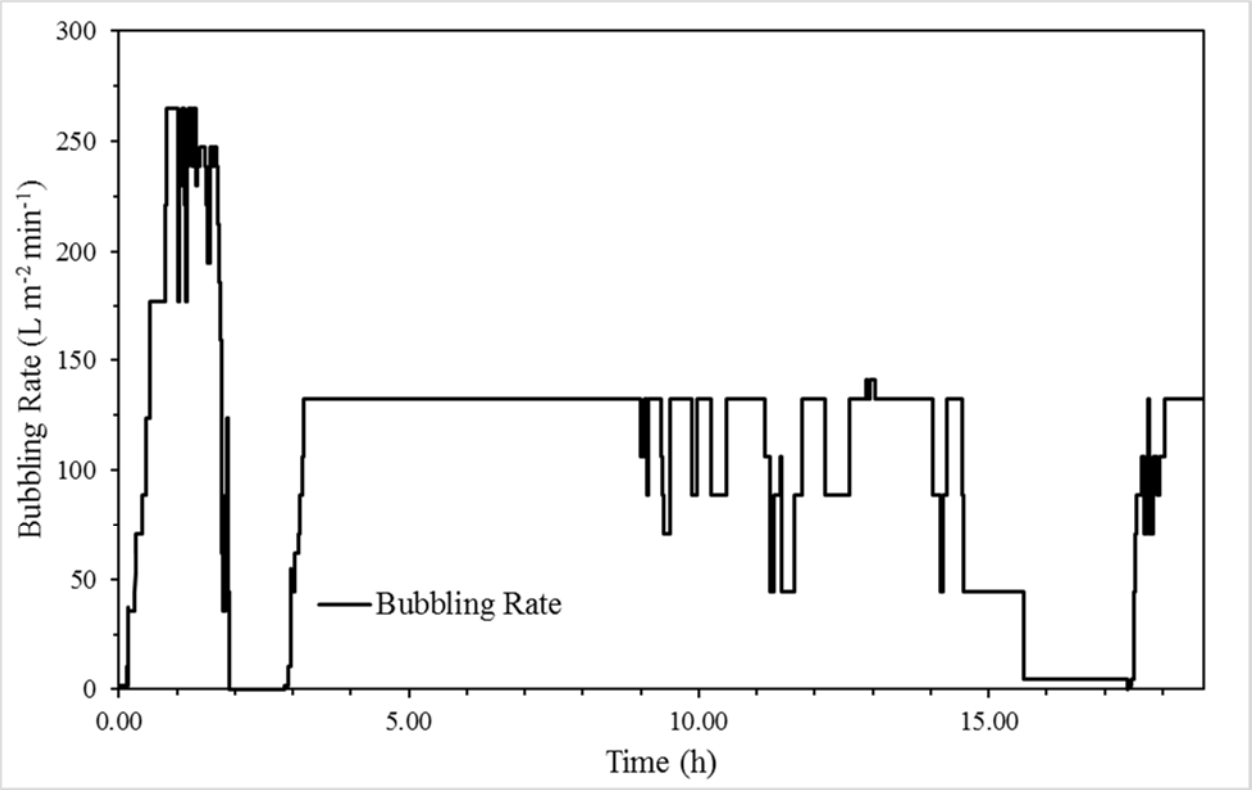

Figure 3.12. Bubbling rate from CLSM run with AP-105 waste feed slurry.

Three off-gas samples were collected during feeding. The timing and duration of each sample are listed in Table 3.8 and their occurrence in the timeline of the plenum temperature is shown in Figure 3.13. During these three times, the temperature at the off-gas sampling switch and primary SBS (Figure 3.9) 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 3.8. Timing of Off-Gas Samples from CLSM Run with AP-105 Waste Feed Slurry

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	4/5/2018	6:27:07 PM	1.07	6:39:08 PM	1.27	12.02
2	4/11/2018	7:48:58 PM	5.82	7:59:11 PM	5.99	10.22
3	4/12/2018	1:22:09 AM	11.37	1:39:02 AM	11.65	16.88

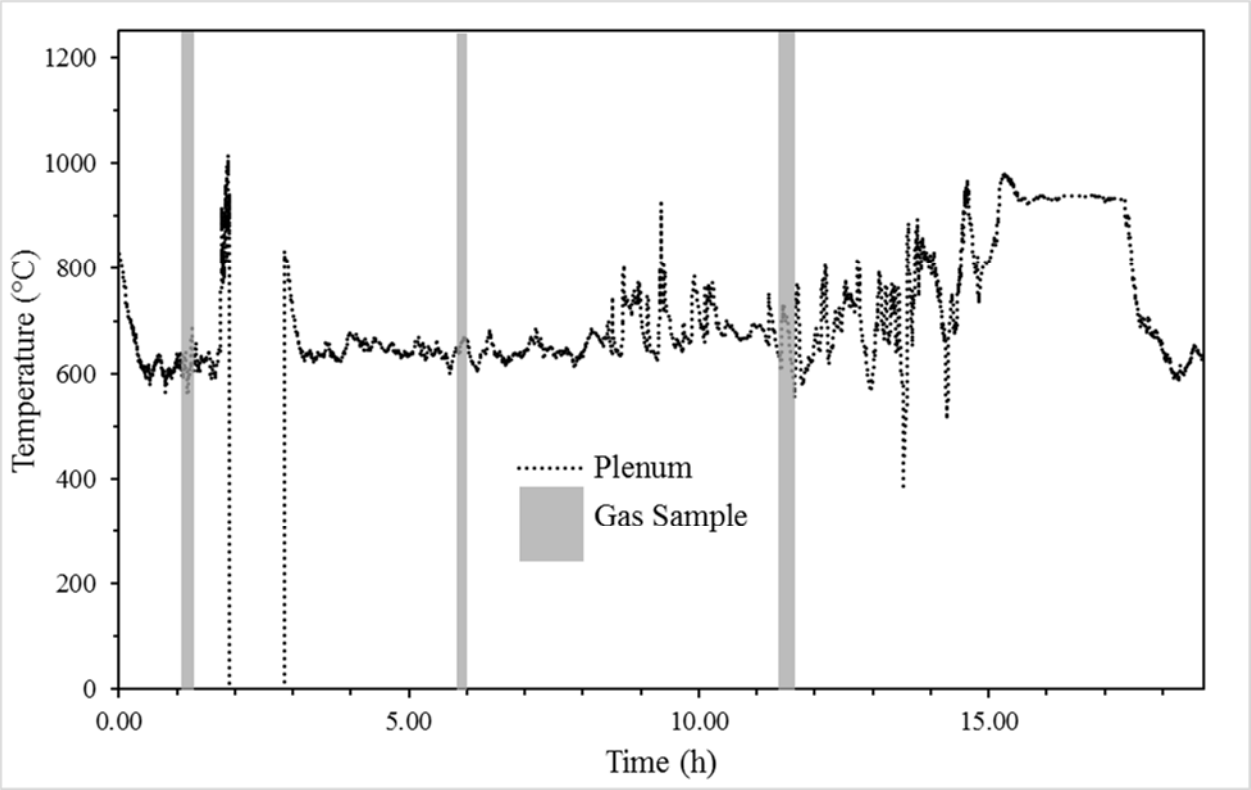

Figure 3.13. Plenum temperature as a function of time for the CLSM run with AP-105 waste feed slurry, showing timing and durations of gas-sampling events.

The timing and mass of each periodic glass pour are shown in Table 3.9, which correspond with the times that the melter vacuum was set in the range of -5 to -10 in-H_2O in Figure 3.11.

Table 3.9. Timing and Mass of Glass Pours from CLSM Run with AP-105 Waste Feed Slurry

Glass Pour Time	Glass Pour Mass	Glass Pour Time	Glass Pour Mass
hr	g	hr	g
0.00	298.13	9.41	415.00
1.33	493.56	9.92	224.43
1.91	1953.50	10.39	606.50
3.40	242.89	10.89	592.50
3.90	365.28	11.72	274.00
4.31	238.22	12.20	269.70
4.75	240.34	12.78	205.36
5.26	296.21	13.23	368.00
5.75	364.66	14.08	449.00
6.31	359.03	15.97	158.68
6.76	382.79	17.83	287.96
7.32	350.45	18.34	274.18
8.02	562.72	18.70	2429.50
8.77	361.22		

3.2.2 Feed Processing Characteristics

Prior to feeding on April 5, 2018, a transparent silica cylinder that guided the glass as it poured from the bottom of the CLSM vessel broke and was removed from below the system so as to not interfere with the pouring of the glass. However, without the cylinder present, air was allowed to flow into the furnace surrounding the CLSM vessel, resulting in heat loss and reduced temperature in the furnace and glass that were cooler than the target temperature range. In addition, the thermocouple in the glass melt began reporting unrealistic temperature data after ~1.9 hours of feeding. As a result, feeding was terminated at hour 1.91 on April 5, 2018, at 8:18 PM. For all process data (Figure 3.8, Figure 3.9, Figure 3.10, Figure 3.11, Figure 3.12, and Figure 3.13), values were set to 0 after this point and a time gap of 0.93 hour was added to account for the shutdown and downtime of the equipment while delineating the separate testing time periods.

To replace the silica cylinder, the CLSM vessel had to be removed from the system and replaced. The RPL CLSM vessel was replaced with the CLSM vessel that was used for all the prior APEL test runs as described in Section 3.1. The replacement CLSM vessel was loaded with 1.8 kg of previously prepared glass pieces designed after the WDFL1 formulation, the furnace surrounding the CLSM vessel was heated to 1250 °C, and feeding was restarted at hour 2.84 (accounting for the added 0.93-hour time gap in the feeding timeline, shown in Figure 3.10) on April 11, 2018, at 4:51 PM and was operated continuously until system shutdown.

At hour 7.81, the first bucket of AP-105 actual tank waste feed slurry was nearing empty, so the AP-105 actual tank waste feed slurry from the second bucket was transferred into the first bucket. The transfer procedure allowed the CLSM to remain in constant operation without the need to stop feeding. Slightly lower concentrations of Al, Ca, Fe, Si, Ti, Zn, and Zr in the feed slurry analyzed after this transfer (see Table 3.10 in Section 3.2.3) indicated that some solids settled and were not completely transferred from the second bucket to the first.

At hour 13.54, feed particulate buildup on the CLSM vessel walls and lid blocked the melter vacuum pressure sensor, but this buildup did not stop the flow of the off-gas through the vacuum pump. Vacuum control was never lost, but the measured vacuum values, shown in Figure 3.11, are anomalous from hour 13.54 to 17.33. During this time, feed particulate buildup also covered the viewport into the CLSM vessel, preventing visual observation of the cold cap. To eliminate the feed particulate buildup, feeding was stopped at hour 14.69, shown in Figure 3.10, and the cold cap was allowed to burn off. Without the cold cap present, the plenum temperature increased, shown in Figure 3.8 and Figure 3.13, and slowly melted the feed particulate buildup. After the melter vacuum pressure sensor was restored to a normal state and the glass melt surface could again be observed through the viewport, feeding was restarted at hour 17.38. At hour 18.70 (April 12, 2018, at 8:42 AM), the feeding line was flushed with water, the cold cap was allowed to burn off, all of the glass inventory was poured from the melter vessel, and the test was terminated.

3.2.3 Sample Analysis Results

A set of 23 samples that represented the entirety of the CLSM run were sent for chemical analysis. These samples included eight glass samples from pours throughout the run, four samples of the feed slurry, four HEPA filter samples (three sets of sample HEPA filters and one primary HEPA filter), and seven liquid samples (five condensate samples, one sample SBS liquid sample, and one liquid sample from the sample HEPA filter housing). Each sample mass and the resulting concentration of each analyzed cation and anion are given in Table 3.10, where values left blank were below the analysis detection limit. All liquid samples from the test run that were not sent for chemical analysis were combined and sent for grouting and analysis. As a result, the mass of the total condensate was not measured.

Table 3.10. Chemical Analysis of Selected Samples from the CLSM Run with AP-105 Waste Feed Slurry

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 1.33	Glass	493.56	1.44	1.04	32400	15.3	29600	0.931	15000	1210	4.92	7.68	40000
Glass Pour 3.40	Glass	242.89	0.421	2320	25400	1050	29600	89.7	21400	1630	2.93	19.1	46100
Glass Pour 6.31	Glass	359.03	1.79	825	30200	447	31700	36.1	17400	1055	3.68	9.42	42400
Glass Pour 9.41	Glass	415.00	1.83	275	32000	198	30500	15.1	15700	873	3.57	6.36	40200
Glass Pour 11.72	Glass	274.00	2.43	97.1	32800	106	29200	7.35	14900	766	4.21	5.56	39500
Glass Pour 12.78	Glass	205.36	2.78	66.0	32900	85.2	29300	5.59	14700	779	4.24	5.25	39100
Glass Pour 15.97	Glass	158.68	1.63	43.2	33100	63.8	29800	3.77	14700	944	4.12	5.46	39400
Glass Pour 18.70	Glass	2429.50	1.86	28.2	33300	55.8	31800	3.13	14800	899	3.87	5.33	38700
Feed Slurry Bucket 1, 4/5	Feed	n/a	4.66		3010	4.40	9530		1520	220		1.76	3180
Feed Slurry Bucket 2, 4/5	Feed	n/a	4.57		2240	3.26	9080		1150	221			2770
Feed Slurry Bucket 2, 4/11	Feed	n/a	4.84		1370	1.87	8770		484	230			1460
Feed Slurry after Transfer	Feed	n/a	4.87		1340	1.85	8730		533	218		1.37	1340
Sample HEPA Filter 1	HEPA	38.27	11.1	96.7	3120	4080	3375		1225	48.5		0.737	151
Sample HEPA Filter 2	HEPA	38.13	0.931	22.3	3020	3990	2560		1210	49.8		1.48	259
Sample HEPA Filter 3	HEPA	39.10	1.34	8.92	2260	4090	2630		1260			0.403	86.7
Primary HEPA Filter 1	HEPA	38.27	98.7	6070	10200	4160	11500	2.99	4000	200		4.48	762
Condensate after HEPA 1	Liquid	258.50	0.22	0.0427	27.2		90.4		13.2	22.7		0.292	41.2
Condensate after HEPA 2	Liquid	238.50	1.34	71.4	22		121		8.40	6.77			35.6
Condensate after HEPA 3	Liquid	243.47	2.43	39.2	34.4		180		13.1	6.02			198
Sample SBS Liquid	Liquid	621.39	0.266	4.76						0.732			5.17
Condensate Sample 1	Liquid	n/a	2.53	27.4	37.8		221		16.2	12.9			35.1
Condensate Sample 2	Liquid	n/a	2.36	27.3	38.2		223		16.0	12.8			35.2
Wet Sample HEPA Liquid	Liquid	54.55	3.45	41.1	27.5	1.15	288		5.62	26.6		1.08	61.8

Table 3.10. Chemical Analysis of Selected Samples from the CLSM Run with AP-105 Waste Feed Slurry (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 1.33	5.88	21.7	53.7	8120	110	29.4	683	770	3820	193000	157000	14.5	1520
Glass Pour 3.40	7850	68.7	3350	6940	150	2850	929	1200	3330	208000	126000	756	1340
Glass Pour 6.31	3200	34.6	1365	8140	99.4	1185	534	856	3825	205000	144500	320	1385
Glass Pour 9.41	1260	19.4	548	7920	76.4	514	340	716	4110	207000	156000	139	1450
Glass Pour 11.72	559	16.4	242	8350	67.9	257	262	692	4110	204000	157000	73.9	1480
Glass Pour 12.78	396	14.5	172	8360	65.6	204	238	613	4090	209000	155000	59.2	1460
Glass Pour 15.97	235	14.1	106	8520	69.2	147	339	673	4070	204000	156000	44.2	1410
Glass Pour 18.70	168	13.2	81.1	7840	64.5	122	280	648	4180	204000	163000	38.5	1440
Feed Slurry Bucket 1, 4/5		1.63		110	2.94	39.3	21.7	152	2410	30600	87500	3.06	895
Feed Slurry Bucket 2, 4/5				48.3	2.17	39.9	21.4	131	2440	27900	87600	1.81	831
Feed Slurry Bucket 2, 4/11				16.2	1.02	40.6	21.2	121	2540	28000	88800	0.997	879
Feed Slurry after Transfer				21.8	1.02	39.1	20.7	110	2390	25100	86000	1.24	938
Sample HEPA Filter 1		0.849	2.42	179	19.0	11.2	9.93	14.5	2515	106000	11000	46.2	207
Sample HEPA Filter 2		1.02		179	20.7	8.76	11.3		2130	111000	6170	45.0	102
Sample HEPA Filter 3		0.63		348	15.6	5.66	5.72		2290	104000	6290	46.4	67.0
Primary HEPA Filter 1		3.63	77.5	556	129	168	33.8	43.8	10100	336000	56300	138	560
Condensate after HEPA 1					0.547		0.334		21.9	34.1	396		19.1
Condensate after HEPA 2			1.08			2.27	0.254	6.24	38.6	33.2	583		39.2
Condensate after HEPA 3						1.50	0.558		73.0	74.6	1090		40.0
Sample SBS Liquid					1.4		0.588		8.93		116		6.13
Condensate Sample 1						1.03	0.453		73.5	51.2	1170		42.0
Condensate Sample 2						0.977	0.482		72.1	51.5	1130		42.8
Wet Sample HEPA Liquid					29.0	1.38	11		116	17.3	1630		54.8

Table 3.10. Chemical Analysis of Selected Samples from the CLSM Run with AP-105 Waste Feed Slurry (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 1.33	12.6	7680	240	58.1	43.1	27000	20800	2.68	4.40				
Glass Pour 3.40	12.6	9970	241	47.0	37.5	30100	18900	2.27	2.27				
Glass Pour 6.31	14.7	8725	302	52.6	39.4	29300	20650	2.87	2.89				
Glass Pour 9.41	15.3	7790	312	51.7	38.4	29300	20300	3.25	3.23				
Glass Pour 11.72	14.7	7730	330	51.9	39.0	29100	20800	3.09	2.75				
Glass Pour 12.78	15.7	7700	325	50.9	39.1	28500	21000	2.99	2.32				
Glass Pour 15.97	16.5	7630	331	51.6	39.3	28900	21100	2.44	2.07				
Glass Pour 18.70	15.3	7110	344	48.3	36.7	29800	19400	7.10	3.59				
Feed Slurry Bucket 1, 4/5		162	96.5	0.983		4110	58.4	2805	2170	535	34.0	19200	13650
Feed Slurry Bucket 2, 4/5		127	95.1			3670	54.7	2850	2210	549	34.6	19700	14100
Feed Slurry Bucket 2, 4/11		59.7	86.9			2130	27.8	2960	2280	563	35.6	20300	14500
Feed Slurry after Transfer		63.6	83.8			1980	28.2	2890	2230	541	35.0	19700	14100
Sample HEPA Filter 1		8.82	29.0		0.722	3035	30.0	4770	425	37.5	55.9	840	567
Sample HEPA Filter 2	1.23	6.16	25.8	0.435	0.717	3000	28.4	300	103		2.95	665	
Sample HEPA Filter 3	1.29	7.43	24.5		0.632	3010	27.4	586	24.3		4.41	285	117
Primary HEPA Filter 1	2.19	45.3	111	1.06	2.29	10400	121	4880	9660	19.8	34.6	21200	
Condensate after HEPA 1		1.16				33.7	1.25	286	145		14.4	1435	
Condensate after HEPA 2		1.74				31.6	1.81	601	228		13.0	3620	
Condensate after HEPA 3		3.72	1.85			51.5	3.60	1080	236		13.9	4570	
Sample SBS Liquid						5.38		111	75.8			940	
Condensate Sample 1		2.07				59.3	2.17	1030	248		17.8	4460	
Condensate Sample 2		2.14	1.48			59.1	2.13	1040	244		19.1	4480	
Wet Sample HEPA Liquid						40.1		1540	188	10.7	21.1	1030	24.4

Table 3.10. Chemical Analysis of Selected Samples from the CLSM Run with AP-105 Waste Feed Slurry (cont.)

Sample Name	P (Phosphate)	Np-237	Cm-244	Am-241	Pu-239/240
	mg kg^{-1}	pCi g^{-1}	pCi g^{-1}	pCi g^{-1}	pCi g^{-1}
Glass Pour 1.33		2.81E+00			
Glass Pour 3.40		1.69E+00			
Glass Pour 6.31		5.87E+00			
Glass Pour 9.41		8.58E+00			
Glass Pour 11.72		9.36E+00			
Glass Pour 12.78		1.05E+01			
Glass Pour 15.97		1.02E+01			
Glass Pour 18.70		9.22E+00			
Feed Slurry Bucket 1, 4/5		2.90E+00			
Feed Slurry Bucket 2, 4/5		2.90E+00			
Feed Slurry Bucket 2, 4/11		1.71E+00			
Feed Slurry after Transfer		1.59E+00			
Sample HEPA Filter 1					
Sample HEPA Filter 2					
Sample HEPA Filter 3					
Primary HEPA Filter 1					
Condensate after HEPA 1					
Condensate after HEPA 2				6.47E-01	
Condensate after HEPA 3				7.99E-01	7.27E-01
Sample SBS Liquid					
Condensate Sample 1					
Condensate Sample 2					
Wet Sample HEPA Liquid			3.91E-01	8.07E-01	

4.0 Discussion

4.1 Rhenium in AP-105 Simulant Glass Pours

The effective glass production rate and plenum temperature for the AP-105 simulant feed slurry test run in APEL (Figure 3.3 and Figure 3.1, respectively) have been plotted together with the concentration of Re in each analyzed sample of glass product (Table 3.5) from the glass pours with respect to time of the glass pour during the test run and are shown in Figure 4.1. The Re concentration in the glass remained relatively constant over the duration of the test run despite the production interruptions when the cold cap was completely (hour 2.48 to 2.95) or partially (hour 5.84 to 6.06) burned off. Prior to the final glass pour at hour 6.51, the cold cap was burned off and combined into the glass melt. The slight increase in Re concentration of the final glass pour may be due to the Re that had been trapped by the cold cap or on the CLSM vessel walls becoming incorporated in the glass and then being immediately poured from the melter and cooled before the Re had an extended time to volatilize.

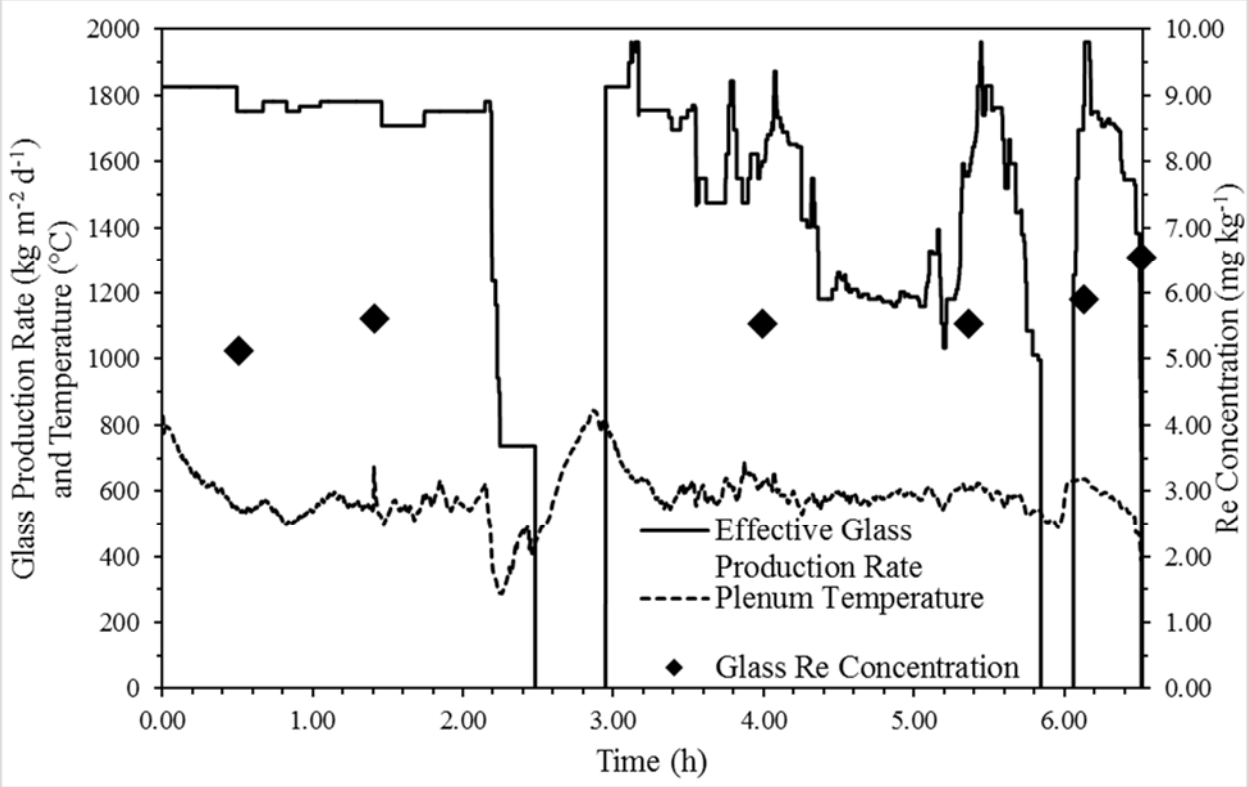

Figure 4.1. Effective glass production rate, plenum temperature, and Re concentration in analyzed glass pour samples the CLSM run with AP-105 simulant feed slurry.

4.2 Rhenium Retention and Recovery for AP-105 Simulant

The retention of the Re in the in the AP-105 glass product (R_{Re}) is given in Eq. (4.1):

$$R_{Re} = \frac{\dot{m}_{Re,glass}}{\dot{m}_{Re,slurry}} \tag{4.1}$$

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

$$R_{Re} = \frac{m_{Re,glass}}{m_{Re,slurry}} \tag{4.2}$$

The mass of Re input into the system during collection of off-gas samples 1 and 2 was calculated from the feeding rate of AP-105 simulant feed slurry (Table 3.2), the time duration of the off-gas sampling periods (Table 3.3), and the concentration of Re in the slurry (Table 3.5). The results of this calculation are shown in Table 4.1. The mass of Re output from the system during these periods was calculated from the effective glass production rate of AP-105 glass product (Figure 3.3) and the concentration of Re in the glass product (Table 3.5) that was poured following each off-gas sampling period. Glass pours 1.41 and 5.36 were subsequent to off-gas samples 1 and 2, respectively, and the concentrations of Re in each resultant glass product is given in Table 3.5. The retention of Re during each off-gas sample period was then calculated and the results are listed in Table 4.1. These retention values are comparable to single-pass retention of Re in a LAW glass of 0.43 measured in previous studies (Matlack et al. 2010).

Table 4.1. Rhenium Retention and Recovery during Off-Gas Sampling Periods

Off-gas Sample Number	Re Mass Input Feed Slurry	Re Mass Output Glass	Re Mass Output Sample HEPA	Re Mass Output Sample SBS and Off-Gas Wash	R_{Re}	Re Recovery
	mg	mg	mg	mg		%
1	2.996	1.172	0.846	1.21	0.39	~ 108
2	2.028	0.780	0.737	0.82	0.38	~ 115

The percent recovery of Re in the CLSM system during collection of off-gas samples 1 and 2 (Re Recovery) is given in Eq. (4.3):

$$\text{Re Recovery} = \frac{m_{Re,glass} + m_{Re,off-gas}}{m_{Re,slurry}} \times 100 \tag{4.3}$$

where $m_{Re,\,off\text{-}gas}$ is the total mass of Re captured in the off-gas system. Throughout each sampling period, Re was collected in three different locations: the sample HEPA filters, the sample SBS, and the walls of the off-gas system leading up to the sample HEPA filters. The mass of Re on each set of sample HEPA filters was calculated from the mass of each filter (Table 3.5) and concentration of Re on each filter (Table 3.5) and is given in Table 4.1. Sample SBS liquid collection and washing of the off-gas sampling line were performed once the CLSM system was shutdown, thus, the Re collected from these liquids was deposited during the totality of all three sampling events. As a result, the total mass of Re in the liquids was partitioned to each off-gas sample based on the duration of each sampling time (Table 3.3) and

feeding rate during the sampling time (Figure 3.3). The resulting masses of Re captured by the sample SBS and walls of the off-gas system during off-gas samples 1 and 2 (calculated from the mass of each solution and concentration of Re in each solution, both shown in Table 3.5) are given in Table 4.1 as well as the calculated Re Recovery for each sample. Both Re Recovery values were slightly greater than 100%, likely due to small amounts of Re deposited in the off-gas sampling line during the non-sampling portions of the test run.

4.3 Technetium-99 in AP-105 Actual Waste Glass Pours

The effective glass production rate and plenum temperature for the AP-105 waste feed slurry test run in the RPL (Figure 3.10 and Figure 3.8, respectively) have been plotted together in Figure 4.2 with the concentration of ^{99}Tc in each analyzed sample of glass product (Table 3.10) from the glass pours with respect to time of the glass pour during the test run. Glass pour 3.40 occurred 0.54 hour after the start of feeding into the replacement CLSM vessel, which had been loaded with new glass that did not contain ^{99}Tc. This is likely the reason that the sample of glass product from glass pour 3.40 had the lowest measured ^{99}Tc concentration. The increase in ^{99}Tc concentration in the glass product of the glass pours from 9.41 to 12.78 indicates that the incorporation of ^{99}Tc into the glass had not yet reached steady state or that the cold cap coverage/thickness was unsteady due to variable feeding rates. The drop in ^{99}Tc concentration measured in the sample of glass product from glass pour 15.97 compared to the ^{99}Tc concentration measured in the sample of glass product from glass pour 12.78 may have been caused by the cold cap burn-off that occurred beginning at hour 14.69 and resulted in the glass pool in the CLSM vessel idling for over an hour before glass pour 15.97. Technetium has been shown to volatize from an idling glass melt (Pegg 2015).

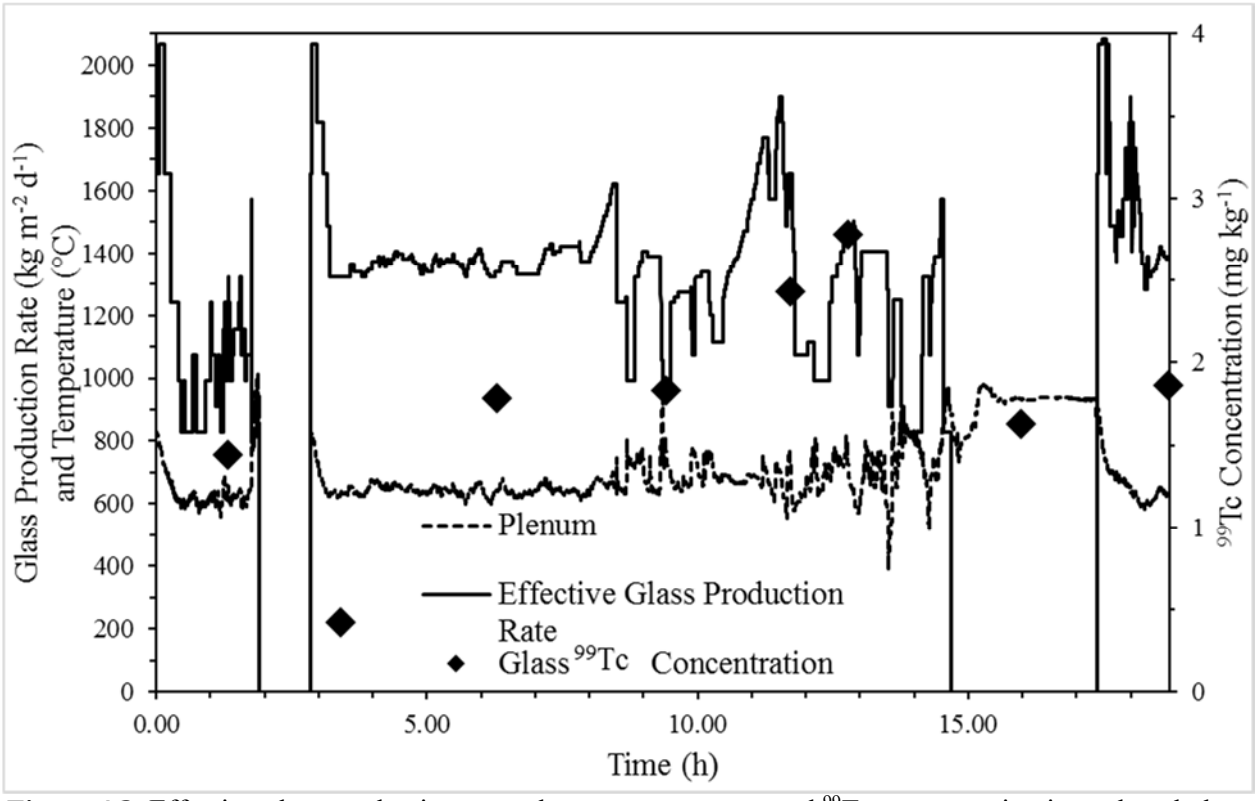

Figure 4.2. Effective glass production rate, plenum temperature, and ^{99}Tc concentration in analyzed glass pour samples from the CLSM run with AP-105 waste feed slurry.

4.4 Technetium-99 Retention and Recovery in AP-105 Actual Waste

The retention of ^{99}Tc in the AP-105 actual waste glass product ($R_{99_{Tc}}$) during the three off-gas sampling periods was derived in the same way as R_{Re}, Eq. (4.2), in Section 4.2 and is shown in Eq. (4.4):

$$R_{99_{Tc}} = \frac{m_{99_{Tc},glass}}{m_{99_{Tc},slurry}} \tag{4.4}$$

where $m_{99_{Tc},glass}$ is the mass of ^{99}Tc out of the melter via the glass and $m_{99_{Tc},slurry}$ is the mass of ^{99}Tc into the melter via the feed slurry.

Since the feeding rate of AP-105 actual waste feed slurry could not be measured for the test run, the feeding rate during each off-gas sample period was determined from the effective glass production rate (Figure 3.10) and the value for glass yield per kilogram of feed slurry (426 g-glass kg-feed slurry^{-1}) as calculated by Matlack et al. (2017). The feed slurry buckets used during collection of gas samples 1, 2, and 3 were bucket 2 (4/5), bucket 2 (4/11) and the bucket after transfer, respectively, and the concentration of ^{99}Tc in each feed slurry sample is given in Table 3.10. The $m_{99_{Tc},slurry}$ was then calculated during each off-gas sample period (Table 3.8) and is shown in Table 4.2. The $m_{99_{Tc},glass}$ during each off-gas sample period was calculated from the effective glass production rate of AP-105 actual waste glass product (Figure 3.10) and the concentration of ^{99}Tc in the sample of glass product that was poured following each off-gas sampling period (Table 3.10). Glass pours 1.33, 6.31, and 11.72 were

subsequent to off-gas samples 1, 2, and 3, respectively, and the concentration of ^{99}Tc in each resultant glass product is given in Table 4.2. The retention of ^{99}Tc during each off-gas sample period was then calculated and the results are listed in Table 4.2. The increasing $R_{99_{Tc}}$ values as the test run progressed may indicate the reduction of ^{99}Tc volatility due to the presence of the cold cap or that ^{99}Tc had not yet reached the point of steady-state retention.

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

Off-gas Sample Number	^{99}Tc Mass Input Feed Slurry	^{99}Tc Mass Output Glass	^{99}Tc Mass Output Sample HEPA	^{99}Tc Mass Output Sample SBS	^{99}Tc Mass Output Sample HEPA Liquid	$R_{99\,Tc}$	^{99}Tc Recovery
	mg	mg	mg	mg	mg		%
1	0.983	0.132	0.425	0.05	--	0.13	~ 62
2	1.267	0.199	0.035	0.04	0.07	0.16	~ 28
3	2.583	0.628	0.052	0.07	0.12	0.24	~ 34

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. (4.5):

$$^{99}\text{Tc Recovery} = \frac{m_{99_{Tc},glass} + m_{99_{Tc},off-gas}}{m_{99_{Tc},slurry}} \times 100 \quad (4.5)$$

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 in the off-gas system was collected in three potential locations: the sample HEPA filters, the sample SBS, and the walls of the off-gas system leading up to the sample 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. However, during disassembly of the off-gas sampling system, liquid was discovered in the sample HEPA filter housing for samples 2 and 3. This HEPA filter housing liquid was another potential source of ^{99}Tc in the off-gas system for those two samples. Consequently, the total mass of ^{99}Tc in the HEPA filter housing liquid was partitioned to off-gas samples 2 and 3 based on the duration of each sampling time (Table 3.8) and the feeding rate during that time, while the total mass of ^{99}Tc in the SBS liquid (calculated from the mass of solution and concentration of ^{99}Tc in the solution, both shown in Table 3.10) was partitioned similarly between all three off-gas samples based on the sampling duration (Table 3.8). The resulting mass of ^{99}Tc from each potential source in the off-gas system as well as the final ^{99}Tc recovery are reported in Table 4.2. During collection of sample 1, the ^{99}Tc recovery was less than 100%, likely because the ^{99}Tc from the wall of the off-gas sampling system could not be included in the final calculation. During collection of samples 2 and 3, the amount of ^{99}Tc recovered specifically from the sample HEPA filters was less than expected based on the measured ^{99}Tc on the sample HEPA filter from sample 1. This was possibly connected to the presence of the liquid in the HEPA filter housing. The liquid in the HEPA

filter housing was unexpected and the cause, whether it was condensation of the off-gas, liquid from the sample SBS, or from an alternative source, is being investigated.

4.5 Comparison between AP-105 Simulant and Actual Waste CLSM Systems

Table 4.3 compares the average compositions (determined by converting the metals concentration in the glass product, Table 3.5 and Table 3.10, to their associated oxides) of the samples of glass product from the AP-105 simulant feed slurry test run in APEL and the AP-105 actual waste feed slurry test run in RPL to the target glass composition from Table 2.2. For these comparisons, minor components (target values <1.0 wt%) were removed and the glass composition renormalized. The percent difference between the AP-105 simulant glass and AP-105 actual waste glass compositions in

Table **4.3** shows that the weight percents of the major glass forming oxides are within 10% of each other, except for ZnO. In addition, the order of magnitude of the concentration of sulfur in the glass product from all of the glass pours in the AP-105 simulant feed slurry (Table 3.5) and the AP-105 actual waste feed slurry (Table 3.10) remained the same throughout each test run, indicating that the sulfur had reached a steady-state concentration in the glass.

Table 4.3. Comparison of AP-105 Simulant and AP-105 Waste Glass Products with Target Glass Composition

Metal Oxide Component	Average AP-105 Simulant Glass wt%	Average AP-105 Waste Glass wt%	Target AP-105 (WDFL1) Glass wt%	% Difference Between Simulant and Waste Glass %
Al_2O_3	6.4	6.1	6.2	4.5
B_2O_3	9.8	10.0	10.1	2.6
CaO	2.2	2.3	2.1	4.7
Fe_2O_3	5.8	6.0	5.6	2.9
MgO	1.4	1.4	1.5	1.6
Na_2O	20.9	21.1	21.3	1.2
SiO_2	46.4	45.1	45.2	2.9
TiO_2	1.3	1.4	1.4	2.6
ZnO	3.2	3.7	3.5	15.1
ZrO_2	2.6	2.8	3.0	10.3

The concentrations of metals found in the samples of the total collected condensate from the AP-105 simulant feed slurry test run in APEL (Table 3.5) and AP-105 actual waste feed slurry test run in RPL (Table 3.10) are shown in Table 4.4. The concentration of most metals' were found at the same order of

magnitude in both the simulant and waste condensate samples. Cs, Mo, W, and ^{99}Tc were not added to the AP-105 simulant feed slurry and thus were not measured in the simulant condensate samples.

Table 4.4. Comparison of AP-105 Simulant and AP-105 Actual Waste Condensate Samples

Metal Component	Average AP-105 Simulant Condensate Samples mg kg^{-1}	Average AP-105 Waste Condensate Samples mg kg^{-1}
Rhenium	2.40	--
Technetium-99	--	2.44
Cesium	--	27.3
Aluminum	17.8	38.0
Boron	143	222
Calcium	10.9	16.1
Chromium	6.47	6.79
Iron	27.1	35.1
Molybdenum	--	1.00
Nickel	--	0.468
Potassium	38.3	72.8
Silicon	29.7	51.4
Sodium	694	1150
Titanium	0.896	2.11
Tungsten	--	1.48
Zinc	33.2	59.2
Zirconium	0.999	2.15
Chloride	553	1035
Sulfate	161	246
Fluoride	10.4	18.4
N (Nitrate)	1440	4470

The average concentrations of Re and ^{99}Tc measured in samples of the major input and output streams to and from the CLSM system collected during the AP-105 simulant feed slurry test run in APEL (Table 3.5) and AP-105 actual waste feed slurry test run in RPL (Table 3.10), respectively, were converted to molality and are given in

Table **4.5**. For each comparative stream between the simulant and waste test runs, the Re and ^{99}Tc molality order of magnitude was the same, with the exception of the off-gas sampling loop consisting of the sample HEPA filters and SBS liquid. The difference in Re and ^{99}Tc molality in the off-gas sampling loop may be related to the anomalous behavior that caused liquid to deposit in the sample HEPA filter housing during the AP-105 actual waste feed slurry test run in RPL or it may indicate that Re isn't a

perfect surrogate for ^{99}Tc retention in glass and volatility behavior. Further investigation is need to elucidate the difference between the behavior of Re and ^{99}Tc incorporation into glass.

Table 4.5. Comparison between Re and ^{99}Tc Molality at Specific Locations in the CLSM System

CLSM System Stream	Re molality in AP-105 Simulant Samples mole kg^{-1}	^{99}Tc molality in AP-105 Waste Samples mole kg^{-1}
Feed Slurry	3.08E-05	4.78E-05
Glass	3.07E-05	1.79E-05
Sample HEPA	3.56E-04	4.50E-05
Sample SBS	2.04E-05	2.69E-06
Condensate	1.29E-05	2.47E-05
Primary HEPA	4.57E-04	9.97E-04

The concentrations of Cs, Ba, Pb, and Cd in each analyzed sample of glass product from the glass pours of the AP-105 actual waste feed slurry test in RPL (Table 3.10) were plotted with respect to time of the glass pour during the test run and are shown in Figure 4.3. The CLSM vessel replacement between glass pour 1.33 and glass pour 3.40 may have resulted in Cs, Ba, Pb, and Cd contamination from the replacement vessel and is postulated to have caused the spike in the Cs, Ba, Pb, and Cd concentrations between those two glass pours. The decrease in Cs, Ba, Pb, and Cd concentrations measured in all subsequent glass pours after 3.40 indicated that the metals contamination in the replacement melter was slowly incorporated into the glass melt and exited the melter. Figure 4.3 indicates that, after a spike of metals into the CLSM system, the composition of glass product may return to expected level after ~10 h of continuous slurry feeding.

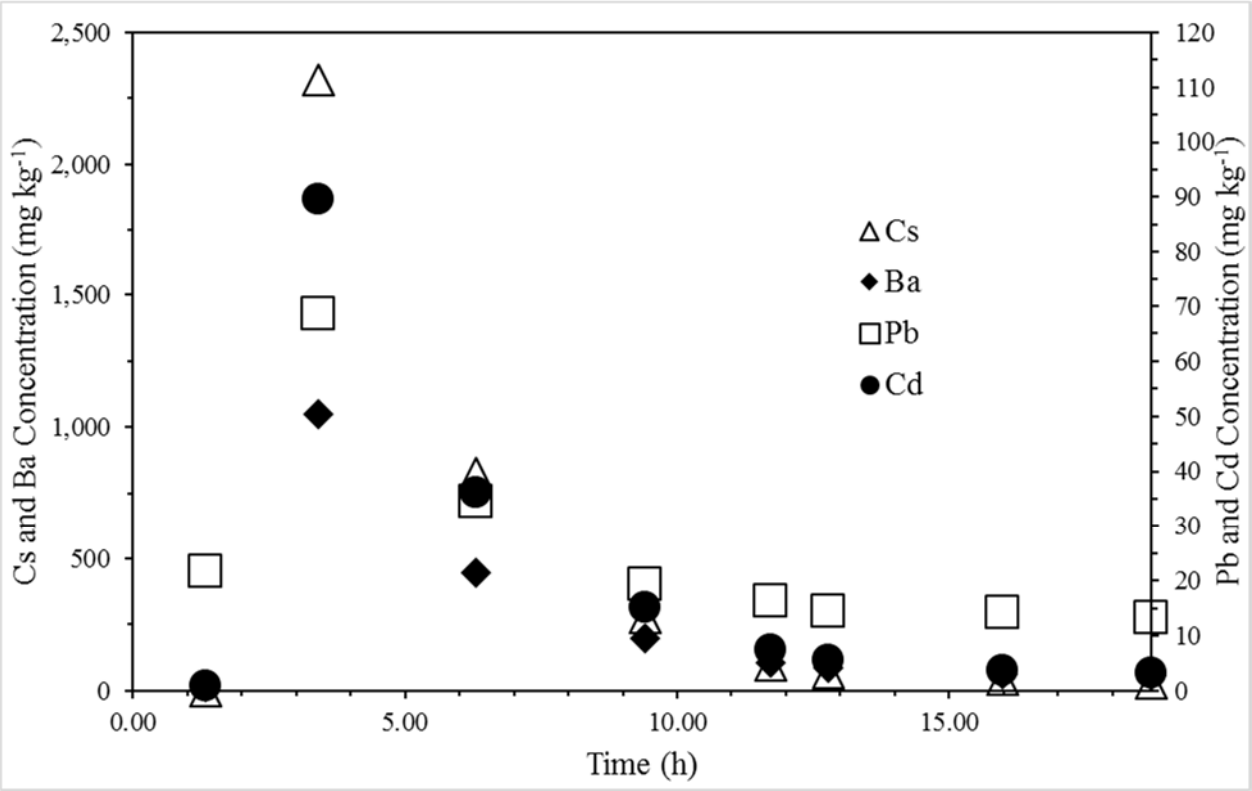

Figure 4.3. Cs, Ba, Pb, and Cd concentrations in selected glass samples from the CLSM run with AP-105 waste feed slurry.

The recoveries of Cr, Pb, Ni, and Ba (Resource Conservation and Recovery Act metals of interest designated by Tilanus et al. 2017) during the off-gas sampling periods of the AP-105 simulant feed slurry test were calculated as described in Section 4.2 from the data in Table 3.5 and are given in Table 4.6 along with the location of the mass of each of the metals in the APEL CLSM system. The recovery of both Cr and Ni was greater than 100%, likely due to the incorporation of Cr and Ni from the walls of the Inconel-690 CLSM vessel into the AP-105 glass product due to corrosion of the vessel. Ba was one of the primary metal components of the HEPA filters, as seen in the chemical analysis of a blank HEPA filter (Table 3.5); thus, the concentration of Ba in the sample HEPA filter samples was a result of the filter itself and not deposits from the AP-105 simulant off-gas. The concentration of Pb in the composition of the blank HEPA filter was also similar to the Pb concentration measured in the sample HEPA filters. However, the mass of Pb in the glass product was multiple orders of magnitude greater than in the HEPA filter and was not a factor in the Pb recovery calculation. The recovery of Ba and Pb in CLSM system was ~100%.

Table 4.6. Recovery of Selected Metals during the Off-Gas Sampling Periods of the AP-105 Simulant Feed Test Run

AP-105 Simulant Test Metal	Off-Gas Sample	Metal Mass in Feed	Metal Mass in Glass	Metal Mass on Sample HEPA filter	Metal Mass in Other Off-Gas Sources	Metal Recovery
		mg	mg	mg	mg	%
Chromium	1	118	362	0.76	2.55	310 [(a)]
	2	79.6	168	0.95	2.55	215 [(a)]
Lead	1	5.80	5.71	0.03	0.00	99
	2	3.93	3.92	0.03	0.00	101
Nickel	1	16.1	521	0.08	0.21	3238 [(a)]
	2	10.9	240	0.08	0.21	2204 [(a)]
Barium	1	1.56	1.76	0[(b)]	0.00	113
	2	1.05	1.08	0[(b)]	0.00	102

[(a)]Cr and Ni were primary components of the CLSM vessel and corrosion by the glass melt contributed to the mass Cr and Ni detected in the glass product

[(b)]Ba present in blank HEPA filter in equal quantity to the sample HEPA filters resulting in no additional Ba after subtracting the Ba concentration of the blank filter

The recoveries of Cr, Pb, Ni, Ba, and Cd during the off-gas sampling periods of the AP-105 waste feed slurry test were calculated as described in Section 4.4 from the data in Table 3.10 and are given in Table 4.7 along with the location of the mass of each metals in the RPL CLSM system. Cr and Ni recoveries were >100%, similar to the recovery measured for each metal in the AP-105 simulant system due to corrosion of the CLSM vessel. Pb and Cd were measured in analyzable quantities in the samples of AP-105 waste glass product, but were below the reporting limit for the AP-105 waste feed slurry. As a result, the recovery for those metals could not be calculated. The recovery of barium was much greater than 100% due to the spike of metals in the glass product from the CLSM vessel replacement.

Table 4.7. Recovery of Selected Metals during the Off-Gas Sampling Periods of the AP-105 Actual Waste Feed Test Run

AP-105 Waste Test Metal	Off-Gas Sample	Metal Mass in Feed	Metal Mass in Glass	Metal Mass on Sample HEPA filter	Metal Mass in Other Off-Gas Sources	Metal Recovery
		mg	mg	mg	mg	%
Chromium	1	47.5	111	1.86	0.140	237 [(a)]
	2	60.2	118	1.90	0.666	200 [(a)]
	3	116	176	0.00	1.10	153 [(a)]
Lead	1	0.00	1.99	0.032	0.00	--
	2	0.00	3.85	0.039	0.00	--
	3	0.00	3.28	0.025	0.00	--
Nickel	1	4.60	62.6	0.380	0.112	1370 [(a)]
	2	5.55	59.5	0.431	0.322	1087 [(a)]
	3	11.0	53.8	0.224	0.532	497 [(a)]
Barium	1	0.70	1.40	0[(b)]	0.00	200
	2	0.490	49.8	0[(b)]	0.024	10176
	3	0.981	19.2	0[(b)]	0.039	1966
Cadmium	1	0.00	0.09	0.00	0.00	--
	2	0.00	4.02	0.00	0.00	--
	3	0.00	1.26	0.00	0.00	--

[(a)]Cr and Ni were primary components of the CLSM vessel and corrosion by the glass melt contributed to the mass Cr and Ni detected in the glass product

[(b)]Ba present in blank HEPA filter in equal quantity to the sample HEPA filters resulting in no additional Ba after subtracting the Ba concentration of the blank filter

5.0 Conclusions

The CLSM system was designed to convert waste from Hanford tank 241-AP-105 to glass while collecting process data and product samples for analysis. Before radioactive waste vitrification was performed, a CLSM system was assembled in a non-radioactive environment for testing with AP-105 simulant. Test runs were executed in the non-radioactive environment to determine the optimum design of the CLSM feeding system, melter vessel, and off-gas system. Once the optimum design had been determined, a test run in the non-radioactive CLSM system was used to determine the appropriate feeding and bubbling rates necessary to achieve the desired glass production rate of AP-105 waste glass. The average glass production rate reached during the AP-105 simulant feed slurry test run was1574 kg m^{-2} d^{-1}, demonstrating that the CLSM could be operated within the desired production range of 1500 to 2000 kg m^{-2} d^{-1}. Samples of glass product, off-gas condensate, HEPA filters, off-gas SBS solutions, and off-gas system wash solutions from the final non-radioactive CLSM test run were collected and sent for chemical analysis.

A second CLSM system was assembled in a radioactive fume hood and used to vitrify 12.4 L of actual AP-105 waste. GFCs were added to this waste volume and then successfully converted into 9.5 kg of radioactive glass product over 15.09 hours of feeding. Given the CLSM vessel glass inventory of ~2 kg, the mass of glass produced from the AP-105 real waste test run accomplished the test goal of turning over the glass inventory 3 times during a test run and established the capability for the system to operate for extended feeding times. Comparable samples to those collected from the AP-105 simulant test run were gathered from the AP-105 real waste test run and sent for chemical analysis.

Results of chemical analysis for samples of glass product from glass pours of both the simulant and waste systems indicated that the samples had similar compositions and were comparable with target values. Likewise, the compositions of off-gas condensate produced from both the simulant and waste systems were shown from chemical analysis to be similar. The retention of Re (used as a ^{99}Tc surrogate in the AP-105 waste simulant) in the glass product and recovery of Re during off-gas sampling were comparable to expected values in similar melter systems. The retention of ^{99}Tc (in the AP-105 actual waste) was lower than Re and further investigation is required to determine if the difference was due to the CLSM system or the chemical behavior of Re and ^{99}Tc in LAW glass. Analysis of other metals (including Cr, Pb, Ni, and Ba) in the glass and off-gas product streams indicated that appropriate recovery within the CLSM system was achievable.

The observed dynamic behavior from the AP-105 real waste test run and comparison with the AP-105 waste simulant test run and other scaled simulant melter systems confirm the viability of the CLSM test platform to produce data that are representative of full scale WTP melter operations. The test objectives for the CLSM system were successfully achieved and condensate collected from the test run with the actual AP-105 waste was routed for further testing.

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, WA.

Dixon DR. 2018. *Continuous Laboratory-Scale Melter Testing of AP-105 Waste Simulant*. Report No. TI-DFTP-033, Pacific Northwest National Laboratory, Richland, WA.

Dixon DR. 2018a. *Continuous Laboratory-Scale Melter Testing of AP-105 Waste Simulant, Part 2*. Report No. TI-DFTP-043, Pacific Northwest National Laboratory, Richland, WA.

Dixon DR. 2018b. *Continuous Laboratory-Scale Melter Testing of AP-105 Waste Simulant, Part 3*. Report No. TI-DFTP-044, Pacific Northwest National Laboratory, Richland, WA.

Dixon DR. 2018c. *Continuous Laboratory-Scale Melter Testing of AP-105 Waste Simulant, Part 4*. Report No. TI-DFTP-045, Pacific Northwest National Laboratory, Richland, WA.

Dixon DR. 2018d. *Continuous Laboratory-Scale Melter Testing of AP-105 Waste Simulant, Part 5*. Report No. TI-DFTP-046, Pacific Northwest National Laboratory, Richland, WA.

Fiskum, SF, 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.

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, IS Muller, RA Callow, N D'Angelo, T Bardakci, I Joseph, and IL Pegg. 2011. *Improving Technetium Retention in Hanford LAW Glass – Phase 1,* VSL-11R2260-1, 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. A. Vitreous State Laboratory, The Catholic University of America, Washington, DC and Atkins Energy Federal EPC, Inc., Calverton, Maryland.

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

Peterson RA, WC Eaton, SK Fiskum, 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.

PNNL. 2017. *DFLAW Test Platform Cesium Ion Exchange Testing with AP-105 Tank Waste with Spherical Resorcinol-Formaldehyde Resin*. Report No. TP-DFTP-001, Rev.0.2, Pacific Northwest National Laboratory, Richland WA.

PNNL. 2018. *Continuous Laboratory-Scale Melter Testing of AP-105 Waste Simulant*. Report No. TP-DFTP-025, Rev. 0.0, Pacific Northwest National Laboratory, Richland WA.

PNNL. 2018a. *DFLAW Test Platform Vitrification of AP-105 LAW Waste Simulant*. 2018. Report No. TP-DFTP-026, Rev. 0.0, Pacific Northwest National Laboratory, Richland WA.

PNNL. 2018b. *DFLAW Test Platform Vitrification of AP-105 LAW Waste Simulant*. Report No. TP-DFTP-027 Rev. 0.0, Pacific Northwest National Laboratory, Richland WA.

PNNL. 2018c. *DFLAW Test Platform Vitrification of AP-105 LAW Waste*. 2018. Report No. TI-DFTP-041, Pacific Northwest National Laboratory, Richland, WA.

Tilanus SN, AN Praga, LM Bergmann, MN Wells, RO Lokken, KW Burnett, AJ Schubick, CS Smalley, EB West, JK Bernards, RT Jasper, SD Reaksecker, SL Orcutt, TL Waldo, TM Holh. 2017. *River Protection Project System Plan*. ORP-11242, Revision 8, Office of River Protection, U.S. Department of Energy, Richland, WA.

Venarsky JJ. 2018. *Continuous Laboratory-Scale Melter Testing of AP-105 Waste.* Report No. TI-DFTP-046, Pacific Northwest National Laboratory, Richland, WA.

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