Development of a Continuous Liquid-Fed Laboratory-Scale Mini-Melter for Nuclear Waste Glass Development

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February 1987

**Prepared for the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830**

**Pacific Northwest Laboratory
Operated for the U.S. Department of Energy
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PACIFIC NORTHWEST LABORATORY
operated by
BATTELLE MEMORIAL INSTITUTE
for the
UNITED STATES DEPARTMENT OF ENERGY
under Contract DE-AC06-76RLO 1830

Printed in the United States of America
Available from
National Technical Information Service
United States Department of Commerce
5285 Port Royal Road
Springfield, Virginia 22161

NTIS Price Codes
Microfiche A01

Printed Copy

Pages	Price Codes
001-025	A02
026-050	A03
051-075	A04
076-100	A05
101-125	A06
126-150	A07
151-175	A08
176-200	A09
201-225	A010
226-250	A011
251-275	A012
276-300	A013

DEVELOPMENT OF A CONTINUOUS LIQUID-FED
LABORATORY-SCALE MINI-MELTER FOR
NUCLEAR WASTE GLASS DEVELOPMENT

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SUMMARY

Borosilicate glass waste forms are being developed for solidifying existing liquid high-level wastes in the United States. The waste glass will be produced in a liquid-fed, ceramic-lined melter by processing a slurry feed containing wastes and glass-forming chemicals. Physical properties of glasses can be predicted with reasonable accuracy, but in many cases melting behavior cannot. Thus, defining an acceptable glass remains largely a trial and error effort. The testing of waste glass feed slurries in continuous melters that are large enough to provide representative data has typically required a one-tenth scale or larger system, which is expensive and time consuming to operate, especially if real radioactive wastes are used. Significant effort has therefore been devoted to developing laboratory screening tests that can identify unacceptable compositions before further resources are committed.

A small, continuous liquid-fed mini-melter (LFMM) has been developed at Pacific Northwest Laboratory (PNL) to aid in waste glass feed slurry development. The LFMM offers several advantages over testing in large-scale melters. The LFMM requires little lead time in preparing for a test run, and the labor and material requirements are low. Real-time melting behavior of liquid feed slurries can be investigated, and feed rates can be estimated for larger-scale melters. The glass produced is representative of that produced by a larger-scale melter, as well as representative of the processing operation itself. Glass produced by the LFMM may subsequently be analyzed for metallic and other secondary phase formation. In contrast to larger-scale melters, the LFMM may be easily and inexpensively installed in a hot cell because of its relatively small size and simple configuration, enabling testing of radioactive feeds.

A total of six LFMM experimental runs were performed. Feeds were obtained which corresponded to PNL melter runs performed in larger PNL developmental joule-heated research melters. These nonradioactive liquid-fed ceramic melters are known as the High-Bay Ceramic Melter (HBCM), the Experimental Ceramic Melter (ECM), and the Pilot-Scale Ceramic Melter (PSCM). Preliminary results obtained with the limited testing are promising. Feeds processed in the LFMM exhibited behavior closely resembling that observed in the large-scale melters.

Feed rate correlations between the LFMM and ECM, HBCM, and PSCM were developed to estimate a range of expected feed rates for the large-scale melters from LFMM data. More developmental work with LFMM testing needs to be completed. Further testing will eliminate bias and establish the LFMM's true correlation to, and representation of, large-scale melters.

ACKNOWLEDGMENTS

The authors would like to thank Ron Stevens for his technical assistance with the furnace and crucible design. Frank Hara and coworkers performed the ICP analysis, and Jim Coleman performed the SEM/EDX analysis. Roy Bunnell provided a peer review of this report. The editorial support of Andrea Currie and the assistance of the Pacific Northwest Laboratory word processing and graphics staff were much appreciated. This report was prepared under the U.S. Department of Energy's Nuclear Waste Treatment Program at the Pacific Northwest Laboratory. Programmatic guidance was provided by Rick Brouns and Harry Burkholder.

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DEVELOPMENT OF A CONTINUOUS LIQUID-FED LABORATORY-SCALE MINI-MELTER FOR NUCLEAR WASTE GLASS DEVELOPMENT

INTRODUCTION

Several waste forms have been proposed for the permanent containment of radioactive nuclear waste. These include grout, SYNROC (synthetic rock forms), and borosilicate glass (U.S. DOE 1982). Borosilicate glass has been selected as the reference waste form to be used in the containment of high-level radioactive nuclear wastes (Harmon, Lakey, and Leigh 1984). Borosilicate glasses may be produced by a variety of systems, one of which is the continuous liquid-fed joule-heated ceramic melter. Typically, radioactive nuclear waste and glass formers are mixed to produce a liquid slurry. This slurry is continuously fed into the melter where an electric current is passed through molten glass; the liquid in the slurry evaporates, and the resulting solids melt, forming a glass. The glass continuously discharges into specially designed canisters for permanent storage (Westsik et al. 1985).

Because no two nuclear waste sources are identical in composition, different glass compositions must be developed to accommodate each waste source, to ensure safe containment of the waste and efficient processing of the glass. Development of waste glass compositions is both time-consuming and costly. Glass must be processable and must provide good containment of radionuclides. Once a glass composition is developed, a feed slurry must be developed to process it. Each feed slurry composition must be tested at various levels of scale to determine its melting rate and melting behavior. The time and effort necessary to determine the processability of waste glass feeds in full-scale melters have been reduced somewhat by the development of several smaller-scale melters that permit feeds to be tested for processability before they are used in large-scale equipment. Such equipment includes the 8.2-liter Experimental Ceramic Melter (ECM) and the 50-liter High-Bay Ceramic Melter (HBCM) developed at Pacific Northwest Laboratory (PNL); a small-capacity (1-liter) dry-fed joule-heated melter (Plodinec and Chismar 1979) developed at Savannah River

Laboratory (SRL); and a 1-liter-capacity dry-fed joule-heated melter developed by Allied Chemical Corporation, Idaho Falls (Gombert 1979).

Efforts at PNL to provide melting behavior support to large-scale melter operations resulted in the development of a bottom-heated refractory crucible test during 1985 (Goldman 1985; Goldman, Chick, and Blair 1985). This test provides a means for investigating the melting rate and melting behavior of a glass feed slurry. Often during melting of a feed slurry, a thick, impermeable cold cap may develop. The cold cap impedes heat transfer to the feed, slowing the melting process. The cold cap may also trap gas between its bottom and the melt surface, further impairing heat transfer. Melting rates of candidate feed slurries are determined by melting a small amount of feed slurry in a bottom-heated refractory crucible. The crucibles are held in a furnace at temperature and extracted as a function of time. The crucibles are sectioned, and the amount of cold cap is measured and converted to a melting rate. While this method has proven satisfactory for determining melting rates of some glass feeds, accurate results for feeds that produce thin, frail cold caps cannot be obtained. Also, long-term effects of melter operation such as melter surface residue layers and formation of secondary metallic and other phases cannot be evaluated with the bottom-heated crucible test.

The effort to address actual time-dependent melter problems resulted in the design and construction of the liquid-fed mini-melter (LFMM). The LFMM offers several advantages over the bottom-heated crucible tests. The LFMM is a continuously-fed, externally heated melter, with a 300-cm³ volume. Melting behaviors of glass feed slurries can be directly monitored, and feed rates can be directly determined. Information on all types of liquid feed slurries can be obtained regardless of the melting behavior and cold cap generated by the feed slurry. Glass produced by the melter can also be examined for metallic and other secondary phase formation. Thus, the LFMM should more truly represent large-scale melters.

This report documents the development of the LFMM and its use in testing simulated nuclear waste glass feed slurries. This work was conducted at PNL in support of the Nuclear Waste Treatment Program (NWTP). Correlations between the LFMM and the ECM, HBCM, and PSCM are presented. The LFMM has been shown to

be a useful tool for the development of nuclear waste glass slurry feed compositions, and will result in the savings of time and finances associated with waste glass development.

CONCLUSIONS AND RECOMMENDATIONS

The LFMM is proving itself to be a useful tool in liquid slurry melter feed development. Feeds processed by the LFMM exhibited behavior very closely resembling that seen in the engineering-scale melters. Feeds that had processed well in the engineering-scale melters also processed well in the LFMM. Feeds that processed poorly in engineering-scale melters also processed poorly in the LFMM. The cold caps and melt surfaces observed in the LFMM resembled those seen in engineering-scale melters. The glass product produced had similar crystalline content.

Feed rate correlations developed between the LFMM and engineering-scale melters can be used to estimate a range of expected feed rates. Correlations developed using maximum feed rates had better fit than those developed using nominal feed rates.

More development work with LFMM testing needs to be completed. Other feeds previously run in engineering-scale melters should be tested in the LFMM. As foaming incidents were not experienced in the LFMM, feed that has exhibited foaming behavior in other melters should be run in the LFMM. Testing other feeds will add to establishing LFMM correlations. In addition, feeds not previously run in engineering-scale melters should be "screened" by the LFMM. This testing will eliminate bias and establish the LFMM's true correlation to, and representation of, large-scale melters.

MELTER SYSTEM DESIGN

The LFMM was designed as a laboratory-scale melter, to be representative of engineering- and large-scale ceramic melters with respect to melting behavior. The apparatus incorporates basic components of the large-scale melters: a feed agitation tank, slurry pumping and feeding system, melting compartment, heat source, and glass discharge outlet. The large-scale melters are joule-heated while the LFMM is externally heated. Because of the LFMM's small size, joule heating was judged unnecessary. Heat transfer is high enough that the temperature in the melter would be relatively uniform regardless of heating method. External heat applied to the melting chamber walls is rapidly transferred to the melt, thus minimizing effects on the melt cold cap. The system is designed to allow continuous feeding of liquid slurry, with constant processing of molten glass. An overall schematic of the LFMM system is shown in Figure 1. The three major components of the LFMM--the feed system, crucible, and furnace and controller--are described in detail in the following subsections. The design specifications of the LFMM are compared in Table 1 with those of the PNL developmental joule-heated melters.

FEED SYSTEM

The primary component of the LFMM slurry feed system is the Gilson Minipuls 2 peristaltic pump. This unit is currently capable of producing linear flow rates of approximately 1 to 14 mL/min of water. A manifold pump tube rated at 3.90 cc/min connected to 3/32-in.-ID X 5/32-in.-OD Tygon R-3603 tubing comprises the feed line. The diameter of this tubing was found to be sufficient in generating linear velocities high enough at a minimum volumetric flow rate of 2 mL/min to produce enough inline turbulence to prevent inline feed separation. The liquid feed slurry is drawn from a 1000-mL Erlenmeyer flask. The feed is kept homogeneous and in suspension by continuous agitation by a high speed mixer. The slurry is pumped to the top opening of the furnace and dripped into the LFMM crucible. The system is pictured in Figure 2.

FIGURE 1. Experimental Apparatus - Overall Schematic

TABLE 1. Comparison of Liquid-Fed Mini-Melter and PNL Larger-Scale Melter Specifications

	Melter			
	LFMM	ECM	HBCM	PSCM
Surface area, m ²	0.0029	0.054	0.25	0.73
Volume, L	0.31	8.2	50	290
Surface area-to-volume ratio, m ⁻¹	9.4	6.6	5.0	2.5
Glass depth, cm	10.6	15	22	42

LFMM CRUCIBLE

The LFMM crucible is constructed of Inconel and incorporates several unique design features indicated in Figures 3 and 4. First is the curved weir, or partition, which runs vertically 1/2 in. from the bottom of the crucible, extending to the top. This allows the melting chamber to remain isolated from the exit glass compartment. Melted, homogeneous glass travels under the weir from the melting chamber and up toward the exit tube opening in the side of the crucible.

Once the molten glass within the crucible reaches a depth of 4 in., the molten material will exit the crucible through a 1/4-in. hole drilled in the side and will travel down the 3/8-in.-OD exit tube. The exit tube, designed as an overflow device, extends approximately 3/8 in. below the bottom of the LFMM crucible, eliminating the problem of glass clinging to the metal surface while keeping the exit spout well within the heat zone of the furnace. In addition, the exit tube spout is beveled to facilitate the formation of clean glass droplets, which fall and exit the furnace through a 1-in. hole in the bottom. The glass output is collected in a crucible resting upon a Mettler Model 4400 top-loading balance where the amount produced is weighed to determine production rate.

The operating temperature of the crucible and glass is measured using a Type K thermocouple placed inside an Inconel thermowell. The thermowell is inserted through the top insulating plug of the furnace and into the molten glass, and rests upon the crucible bottom. A vertical temperature profile of

FIGURE 2. LFMM Feed System

the melt may be taken by placing a Type K thermocouple directly into the molten glass and measuring the temperature at specified depths. Temperature measurements taken immediately prior to feeding are typically 1120 to 1135°C at the bottom of the crucible and 1135 to 1150°C at the melt surface; the average

FIGURE 3. LFMM Crucible

temperature differential is 15°C . Temperature measurements taken during slurry feeding are typically 1120 to 1135°C at the bottom of the crucible and 980 to 1010°C at the melt surface, giving an average temperature differential of 110 to 130°C . Larger-scale joule-heated PNL developmental melters typically run with bulk glass temperatures of 1130 to 1150°C , with surface temperatures 50 to 100°C lower.

FURNACE AND CONTROLLER

The LFMM crucible is heated to operating temperature in a Thermcraft Model 35-11-1ZV-CR resistance-heated tube furnace equipped with heavy-gauge Kanthal A1 heating elements capable of generating operating temperatures up to 1300°C . The furnace is controlled by the Thermcraft Temperature Control System, Model 600-208-20-S-D-T, consisting of 1) the RKC Model REX C1000 Type S micro-processor-based temperature controller, 2) an SCR power controller, and 3) a step-down transformer. The REX C1000 controller supplies a linear 4 - to 20 -mV output signal to the power controller. In addition, an Omega Model 6102 Type S

controller is used as an overtemperature controller and operated by a thermocouple separate from the main control thermocouple. This backup system protects against failures of the primary control system. The furnace is pictured in Figure 5. Figure 6 provides the specifications and dimensions of the LFMM furnace.

The LFMM crucible is located inside the furnace such that the midpoint of the molten glass in the crucible is at the midpoint of the furnace heating zone. A 1-in. hole was cut in the top and bottom insulating plugs to allow continuous feeding of glass slurry into, and output of molten glass from, the furnace.

FIGURE 5. LFMM Furnace

FIGURE 6. LFMM Furnace Specifications and Dimensions

FIGURE 8. LPM Furnace Specifications and Dimensions

MELTER TEST PROCEDURE

In this section, the melter test objective is presented. Procedures used to determine feed rates and to examine melt surfaces and glass products are explained.

TEST OBJECTIVE

The overall LFMM test objective is to ultimately provide melting behavior data to be used in liquid slurry melter feed development. During actual large-scale melter operations, the LFMM could be used as a testing system for variations in melter feeds. The LFMM could also be easily installed in a hot cell because it is small, inexpensive, and simply configured. Melting/feed rate and cold cap formation data, including secondary phase formation and melt foaming, are of primary importance. These properties are used to determine whether or not a liquid slurry feed has acceptable processing characteristics for continuous ceramic melter processing. Maximum and nominal feed rates give indications of how the feed will process in larger-scale melters. Foaming and secondary phase formation potentially reduce processing rates by interfering with surface melting. Secondary phase formation, if soluble in an aqueous environment, can also potentially compromise the chemical durability of the glass product. Conductive higher-density secondary phases can also settle and cause a short circuit in joule-heated ceramic melters. Early observation of these melting behaviors can be used to correct the situation before a feed is tested in large-scale processing equipment.

NOMINAL AND MAXIMUM FEED RATE DETERMINATION

A nominal melter feed rate is defined here as a feed rate that produces 75 to 80% cold cap coverage of the melt surface area. Active venting should occur around the cap, and ideally through it. This feed rate may be referred to as the "operating" or "design" feed rate. A maximum melter feed rate produces up to 95% cold cap coverage, with active venting. This is the maximum feed rate that can be achieved without flooding the melter.

During the melter runs, the feed rate was changed by adjusting the pump setting. The volumetric feed rate was determined by measurements taken at the feed inlet (volume collected over 1 min.). A given feed rate was run for 1 to 2 h before it was adjusted up or down. The appearance of the melt surface was used to determine whether further adjustments of the feed rate were necessary to obtain nominal or maximum feeding conditions. The definitions of nominal and maximum feed rates were used as guidelines, not as strict operating requirements. Observation of both the melt surface and cold cap coverage was taken into account.

MELT SURFACE AND GLASS PRODUCT EXAMINATIONS

The melt surface was examined for secondary phase formation, melt foaming, and cold cap shape and formation. As noted earlier, secondary phase formation can affect both processability and product quality. Melt foaming also hinders processability. The cold cap shape and formation is important to observe, as the formation may interfere with processing. Based on work done by B. M. Wise and J. M. Perez of PNL in 1985, cold caps have been observed to not spread out on the melt surface area, thereby melting more slowly than under ideal conditions. Thus, the melt surface was visually observed frequently, and its appearance was noted.

To characterize the glass product produced by the LFMM, samples of the output glass were taken fairly frequently. The samples were submitted for chemical analysis by inductively coupled plasma spectroscopy (ICP). The analyses were used to provide indications of melter mixing characteristics and confirm calculated turnover times. Thin sections made from samples taken of glass produced by the melter after at least one turnover time were examined using optical microscopy and scanning electron microscopy/energy dispersive X-ray (SEM/EDX) analysis for crystal and secondary phase identification.

FEED RATE AND COLD CAP FORMATION RESULTS

The objectives of the first liquid-fed mini-melter runs were to gain operating experience, develop operating procedures, and generate data for developing empirical correlations between the LFMM and PNL larger-scale developmental melters. Of primary importance were melting/feed rate and cold cap formation (including secondary phase formation and melt foaming). These properties are used to determine whether or not a given liquid slurry feed has acceptable processing characteristics for continuous ceramic melter processing.

Maximum and nominal feed rates were determined for each of six runs, designated LFMM-86-1 through LFMM-86-6. Prior to each run, the feed used was analyzed via ICP. Feed and glass samples were taken routinely during each run and submitted for ICP analysis. All feed compositions and feed and glass analyses are listed in the Appendix.

In this section, the experimental conditions and results obtained from each run are described and discussed. Table 2 summarizes the LFMM developmental tests.

TABLE 2. Summary of LFMM Developmental Tests

Variable	Run					
	86-1	86-2	86-3	86-4	86-5	86-6
Feed	SRL-TDSF-131	PSCM-22 (Aged 1 yr)	HBCM-86-2	FRG 8	WV 182	WV 182
Reductant	Sugar	Sugar	Formic Acid	None	None	Sugar
Melt Behavior	Good	Poor	Good	Good	Poor	Poor
Phase Formation	None	Spinel	Spinel	Few Crystals	Ca Phosphate	Ca Phosphate
Cold Cap	Spread Out	"Tower" Formation	Spread Out	Small "Tower"	Thick "Tower"	Thick "Tower"

RUN LFMM-86-1

Experimental run LFMM-86-1 lasted 13 h 17 min. Melter turnover (time for turnover of one melter volume) occurred in approximately 4.3 h, based on mass

of glass output. A total of 6.02 L of SRL-TDSF-131 feed^(a) was processed, producing 2.66 kg of glass. This feed had been used in the HBCM during the HBCM-86-1 run (March 10 through 14, 1986). The same target composition had been run in the PSCM during PSCM-4 (December 7 through 11, 1981). Crushed glass from HBCM-86-1 was used to dry-feed the LFMM prior to slurry feeding. The conditions and results for run LFMM-86-1 are summarized in Table 3.

The SRL-TDSF-131 feed processed very well in the LFMM. The molten glass showed no evidence of significant phase content, and it dripped cleanly from the crucible outlet tube. The cold cap spread out well, was flexible, and vented well. No melting problems were encountered.

Analyses of the feed samples taken during the run show no major deviations from the bulk composition of the feed. This indicates that minimal settling of the slurry components occurred in the feed lines.

Analyses of the glass samples taken show minor variation of the glass composition over time. The redox response of the glass over the run time is illustrated in Figure 7. The $\text{Fe}^{2+}/\text{Fe}^{3+}$ rose substantially after approximately 4 h into the run. This is caused by turnover of the reducing feed. The starting glass composition is reduced only slightly. As more feed is incorporated into the glass volume, the glass becomes more reduced. The jump occurs around the melter turnover time (approximately 5 h).

TABLE 3. LFMM-86-1 Run Conditions and Results

Feed used	SRL-TDSF-131
Total run time	13 h 17 min
Amount of feed processed	6.02 L
Amount of glass produced	2.66 kg
Feed rate range	0.36 to 0.60 L/h
Glass production rate range	0.12 to 0.31 kg/h
Nominal feed rate	0.43 L/h
Maximum feed rate	0.55 L/h
Process temperature	1093 to 1133°C

(a) This feed composition was developed at Savannah River Laboratory (SRL).

FIGURE 7. Redox Response of LFMM-86-1 Glass

Comparison of the melting behavior of this feed in the LFMM to that in the PSCM and HBCM shows no noticeable difference. The feed melted well in all melters with no problems. A thin section of glass was obtained at 10 h into the LFMM run and examined with SEM/EDX, revealing the smooth vitreous product with very few cubic spinel-type crystals as shown in Figure 8. The EDX trace is given in Figure 9. This vitreous characteristic was also observed in glass produced during HBCM-86-1.

When the mini-melter crucible was emptied at the end of the run, approximately 1/4 in. of melter "sludge" was noticed in the bottom. This was examined with SEM/EDX and found to contain many crystals with high amounts of Fe and Ni and crystals with high amounts of Fe and Cr. An SEM micrograph of the sludge appears as Figure 10; the EDX traces are given in Figure 11. These crystals are the product of dissolution of a stainless steel thermowell that was inserted into the melt. This thermowell was replaced with one made of Inconel, which is more resistant to glass attack and high temperatures. In subsequent test runs, sludge formation was not observed.

300x Magnification

FIGURE 8. SEM Micrograph of LFMM-86-1 Glass Sample No. 5

FIGURE 9. EDX Spectra of Feature 1 in Figure 8

300x Magnification

FIGURE 10. SEM Micrograph of LFMM-86-1 Sludge

RUN LFMM-86-2

Experimental run LFMM-86-2 lasted 14 h 40 min. Melter turnover occurred in approximately 5.2 h. A total of 4.94 L of aged (approximately 1-year-old) PSCM-22 feed was processed, producing 1.69 kg of glass. This feed had been run unaged in the PSCM during PSCM-22 (July 17 through August 2, 1985), and in the ECM during ECM-86-1 (May 22 through 23, 1986). The same target composition had been run in the HBCM during HBCM-85-1 (April 17 through May 2, 1985). Crushed PSCM-22 glass was used to dry-feed the melter prior to slurry feeding. The conditions and results for run LFMM-86-2 are summarized in Table 4.

This feed did not process well in the LFMM. The cold cap did not spread out well. A "tower" was built up on the glass surface under the feed inlet, growing with time and occasionally toppling. This peak grew up to 7.6 cm in height. The cold cap was rigid and non-porous. Venting occurred around the edges of the cold cap. This may have been caused by proximity of the hot walls of the crucible. However, this behavior had also been observed during ECM-86-1. During that run, towers were built up, sometimes growing tall enough

FIGURE 11. EDX Spectra of Features in Figure 10

TABLE 4. LFMM-86-2 Run Conditions and Results

Feed used	PSCM-22 feed with sugar, as used in ECM-86-1
Total run time	14 h 40 min
Amount of feed processed	4.94 L
Amount of glass produced	1.69 kg
Feed rate range	0.29 to 0.52 L/h
Glass production rate range	0.10 to 0.21 kg/h
Nominal feed rate	0.29 L/h
Maximum feed rate	0.34 L/h
Process temperature	1107 to 1132°C

to reach the feed nozzle and clog it. The feed processed so poorly during ECM-86-1 that a "fresh" batch of feed (used in HBCM-86-2) was obtained to replace it for the remainder of the run.

The glass output became "stringy" after 6 h into the run. This was caused by the changing composition of the glass output. The feed contained a higher amount of SiO_2 than did the glass that was dry-fed at the beginning of the run. This effectively raised the viscosity of the glass, causing it to string. In addition to being higher in SiO_2 , the feed was lower in B_2O_3 and Fe_2O_3 than was the starting glass composition. The composition of the glass gradually approached that of the feed composition. This is illustrated in Figure 12, where SiO_2 , Fe_2O_3 , and B_2O_3 compositions are plotted against run time. The shape of the SiO_2 curve suggests that the contents of the melt crucible are well mixed.

The redox of the glass increased, then leveled to a $\text{Fe}^{2+}/\text{Fe}^{3+}$ of 0.13 to 0.18 at approximately 7 h into the run, as illustrated in Figure 13. This ferrous-ferric ratio was observed also during PSCM-22, but not observed during HBCM-85-1. The ferrous-ferric ratio was lower during the latter run when identical target feed was run. It is not known why this occurred.

Analyses of the feed samples taken during the run show little deviation from the bulk composition of the feed analyzed prior to the run. This indicates that minimal settling of the slurry components occurred in the feed lines.

FIGURE 12. Change in Oxide Composition of LFMM-86-2 Output Glass

FIGURE 13. Redox Response of LFMM-86-2 Glass

A thin section of a glass sample taken 11 h into the run was examined with optical microscopy and SEM/EDX. Many cubic spinel-type crystals $[(\text{Fe},\text{Cr})(\text{Fe},\text{Ni})_2\text{O}_4]$ high in Cr and Fe with a small amount of Ni were revealed. These crystals were scattered throughout the glass and measured approximately 1 to 2.7 μm across. Figure 14 shows an SEM micrograph of the glass. Figure 15 shows EDX traces of the crystals observed. These crystals were also observed in glasses produced in PSCM-22 and HBCM-85-1.

RUN LFMM-86-3

Experimental run LFMM-86-3 lasted 11 h 50 min. Melter turnover occurred in approximately 8 h, based on mass of glass output. A total of 5.19 L of HBCM-86-2 feed (400 g/L oxide loading) was processed, producing 1.14 kg of glass. This feed was run in the ECM during ECM-86-1 (second feed), the HBCM during HBCM-86-2 (April 26 to May 2, 1986), and its equivalent in the PSCM during a portion of PSCM-22. Crushed glass from PSCM-22 was used to dry-feed the melter prior to slurry feeding. The conditions and results for run LFMM-86-3 are listed in Table 5.

3000x Magnification

FIGURE 14. SEM Micrograph of LFMM-86-2 Glass Sample No. 5

FIGURE 15. EDX Spectra of Feature 1 in Figure 14

TABLE 5. LFMM-86-3 Run Conditions and Results

Feed used	HBCM-86-2 feed, as used in ECM-86-1 (400 g/L oxides)
Total run time	11 h 50 min
Amount of feed processed	5.19 L
Amount of glass produced	1.14 kg
Feed rate range	0.35 to 0.51 L/h
Glass production rate range	0.06 to 0.14 kg/h
Nominal feed rate	0.35 L/h
Maximum feed rate	0.49 L/h
Process temperature	1096 to 1130°C

This feed processed better than that used in LFMM-86-2, but more poorly than that used in LFMM-86-1. The cold cap spread out slowly, forming a small mound approximately 1 in. high. The feed tended to form a small tower, which toppled readily. Good venting was present, both around and through the cold cap.

Although this feed had better processing characteristics than the feed used in LFMM-86-2, the glass production rate was lower. This was caused by the fact that the feed was dilute and contained roughly 300 g/L oxides versus the target of 400 g/L oxides. Analysis of the feed samples taken during the run indicated that negligible settling of components occurred in the feed lines.

After 6 h, the glass output began to form clumps of droplets resembling grape clusters. Many strings were formed as the drops fell from the crucible outlet tube. This was caused by the changing composition of the glass output. As in LFMM-86-2, the feed contained more SiO_2 than did the starting dry-fed glass. The feed was also lower in B_2O_3 than the starting glass. The glass composition eventually approached that of the feed composition. This is illustrated in Figure 16, where SiO_2 and B_2O_3 weight percent are plotted against run time.

FIGURE 16. Change in Oxide Composition of LFMM-86-3 Output Glass

The glass output remained essentially oxidized over the entire run. This behavior was expected, as this feed contained no sugar. Feeds that contained no sugar, and which were processed in PSCM-22, HBCM-85-1, and HBCM-86-2, exhibited similar behavior.

Examination of a thin section of output glass (sampled 10.83 h into the run) with optical microscopy and SEM/EDX revealed the presence of many cubic spinel-type crystals $[(\text{Fe,Cr})(\text{Fe,Ni})_2\text{O}_4]$ containing high amounts of Cr and Fe with a small amount of Ni. Figure 17 shows an SEM micrograph of the glass. Figure 18 shows an EDX trace of the crystals observed. These crystals measured approximately 1.7 to 3.7 microns across. These crystals were also observed in glasses produced in PSCM-22, HBCM-85-1, and LFMM-86-2.

FIGURE 17. SEM Micrograph of LFMM-86-3 Glass Sample No. 5

FIGURE 18. EDX Spectra of Feature 1 in Figure 17

RUN LFMM-86-4

Experimental run LFMM-86-4 lasted 11 h 26 min. Melter turnover occurred in approximately 6 h. A total of 5.17 L of freshly batched FRG 8 composition^(a) feed was processed, producing 1.68 kg of glass. This feed composition was run in the PSCM during PSCM-20 (March 27 through April 11, 1985) and ECM-85-4 and 5 (February 1985). Laboratory-batched glass with the FRG composition was placed in the melter prior to feeding. Conditions and results for run LFMM-86-4 are summarized in Table 6.

The laboratory-batched feed did not contain the sugar that was present in feeds processed in the ECM and PSCM. Zeolite was not added to this feed as it was to the ECM and PSCM feeds. This should not have significantly affected the melt behavior, as sugar reduction and substituting dry chemicals for zeolite substituents were shown to not affect the melt behavior and subsequent processability of the FRG feed during the ECM and PSCM runs.

^(a) Developed for the Federal Republic of Germany (FRG).

TABLE 6. LFMM-86-4 Run Conditions and Results

Feed used	Laboratory-batched FRG 8 feed
Total run time	11 h 26 min
Amount of feed processed	5.17 L
Amount of glass produced	1.68 kg
Feed rate range	0.41 to 0.55 L/h
Glass production rate range	0.05 to 0.23 kg/h
Nominal feed rate	0.46 L/h
Maximum feed rate	Not determined
Process temperature	1079 to 1132°C

The FRG 8 feed processed fairly well in the LFMM. The cold cap tended to spread out, and then form a small tower. Even so, the feed processed quickly. When feeding was stopped, the cold cap burned off in approximately 4 min. The cold cap formed in the ECM and PSCM runs was thin and rigid. No tower formation was seen. The feed did process very quickly in the ECM (3.5 L/h) compared with average ECM feed rates (2.5 L/h). The feed processed more slowly in the PSCM than predicted, based on the ECM run results.

The ICP analyses of the feed samples taken during the run show that negligible settling of the feed occurred in the feed lines. The analyses of the glass present in the crucible prior to feeding and the final output glass show that the glass composition eventually reached that of the bulk feed composition. The glass output remained essentially oxidized over the run. This is expected behavior for feeds containing no sugar or formic acid (sugar and formic acid act as reducing agents).

A thin section of a glass sample taken 10 h into the run was examined with optical microscopy. The glass was very clear, with only a few small crystals seen, and it compared well with glass produced during PSCM-20.

RUN LFMM-86-5

Experimental run LFMM-86-5 lasted 19 h 32 min. Melter turnover occurred in approximately 8 h, based on mass of glass output. A total of 8.35 L of WV 182 feed^(a) (containing no sugar) was processed, producing 2.35 kg of glass. This target feed composition was run in the PSCM during PSCM-19/19E (August 20 through September 1, and September 19 through 21, 1984). Prior to slurry feeding, SRL 165 black frit was used to dry-feed the melter. The feed analysis showed the MnO_2 level to be high relative to the target, and this could affect the melting behavior. Because this was a minor constituent (less than 3 wt%), no effect was anticipated for the LFMM-85-5 run. In Table 7, the conditions and results of this run are summarized.

The WV 182 feed processed fairly well in the LFMM. The cold cap spread out well, forming a small dome with a pool of liquid slurry in the center. A rather porous cold cap was formed, with a somewhat viscous material surrounding it. This type of cold cap was formed in PSCM-19E. When the crucible was overfed, the cold cap kept the same appearance and the entire surface rose very slowly toward the top of the crucible. Hence, nominal and maximum feed rates were difficult to determine.

The ICP analyses of the feed samples taken during the run show that negligible settling of feed occurred in the lines. Analyses of the glass output

TABLE 7. LFMM-86-5 Run Conditions and Results

Feed used	WV 182, no sugar
Total run time	19 h 32 min
Amount of feed processed	8.35 L
Amount of glass produced	2.35 kg
Feed rate range	0.36 to 0.48 L/h
Glass production rate range	0.06 to 0.17 kg/h
Nominal feed rate	0.41 L/h
Maximum feed rate	0.43 L/h
Process temperature	1132 to 1150°C

(a) Composition developed at PNL for West Valley (WV) Nuclear Services.

showed that it remained essentially oxidized during the run, as the feed did not contain sugar or formic acid. The glass composition changed with time during the run due to compositional differences between the glass present prior to feeding and the feed itself. This is illustrated in Figure 19, in which B_2O_3 and SiO_2 weight percents are plotted against the run time. The shapes of these curves suggest the occurrence of true ideal mixing of crucible contents. Figure 20 gives the actual time distribution curve of B_2O_3 as compared to distribution curves representing "ideal mixing" with mean particle residence times of 3 and 4 h. This comparison shows that the crucible contents are mixed well and that its mean particle residence time is between 3 and 4 h. Despite this residence time, it takes approximately twice that long to achieve complete turnover of crucible contents.

A sample of the cold cap was sectioned, polished, and examined with SEM/EDX. The interface area between the glassy matrix and the cold cap consisted of mainly a calcium phosphate phase containing a small amount of rare earth (cerium). This is shown in Figure 21. Figure 22 gives EDX traces of the phases seen. This phase was also observed in the PSCM-19 surface material (Goldman 1985; Goldman, Chick, and Blair 1985). In the cold cap, phases containing relatively high amounts of Si are seen. A micrograph of this area is given in Figure 23. Figure 24 is an EDX trace of the phase mentioned.

Examination of a thin section of output glass (sampled 12.5 h into the run) with optical microscopy and SEM/EDX revealed the presence of a few small crystals containing Ru. Very few of these crystals were observed. Figure 25 shows an SEM micrograph of the glass. Figure 26 shows an EDX trace of the crystals observed. A small amount of crystals was observed in output glass produced by PSCM-19/19E.

RUN LFMM-86-6

Experimental run LFMM-86-6 lasted 17 h 55 min. Melter turnover occurred in approximately 6.5 h, based on mass of glass output. A total of 8.28 L of WV 182 feed (containing 75 g/L sugar) was processed, producing 2.23 kg of glass.

FIGURE 19. Change in Oxide Composition of LFMM-86-5 Output Glass

FIGURE 20. Time Distribution of B_2O_3 in LFMM-86-5: Comparison with Theoretical Distribution

FIGURE 21. SEM Micrograph of LFMM-86-5 Cold Cap Interface

FIGURE 22. EDX Spectra of Features in Figure 21

Feature 1

300x Magnification

FIGURE 23. SEM Micrograph of LFMM-86-5 Cold Cap

FIGURE 24. EDX Spectra of Feature 1 in Figure 23

1000x Magnification

FIGURE 25. SEM Micrograph of LFMM-86-5 Output Glass (12.5 h into Run)

FIGURE 26. EDX Spectra of Feature 1 in Figure 25

This feed composition was run in the ECM during ECM-84-4 and ECM-84-5A through 5C, and the PSCM during PSCM-19/19E. The crucible was not dry-fed; the glass remaining from LFMM-86-5 was used as the starting base glass. It should be noted that the feed was slightly off target (CeO_2 and Re_2O_3), but this should not have affected the melting behavior. The conditions and results for LFMM-88-6 are listed in Table 8.

This feed processed more slowly than the "no sugar" feed run in LFMM-86-5. The cold cap appearance was also different. The cold cap was darker and tended to form taller towers. This tower formation was observed during ECM-84-4 and 5 and PSCM-19/19E. A thick, viscous phase surrounded the cold cap and appeared to be more viscid (less venting activity seen) than the material seen in LFMM-86-5. A viscous, insoluble phase had formed in the ECM and PSCM runs; this phase grew worse with increased sugar levels, as was observed in LFMM-86-6.

The "sugared" feed did process more slowly in the LFMM, but a very large difference between the two feeds was not seen. As noted for the LFMM-86-5 run, the nominal and maximum feed rates of the sugared WV 182 feed were also difficult to determine due to the cold cap appearance.

Analyses of the feed samples taken during the run show that minimal feed settling occurred in the feed lines. Analyses of the glass show the ferrous/ferric ratio to increase over time to approximately 1.7. This ratio dropped to

TABLE 8. LFMM-86-6 Run Conditions and Results

Feed used	WV 182, 75 g/L sugar added
Total run time	17 h 55 min
Amount of feed processed	8.28 L
Amount of glass produced	2.23 kg
Feed rate range	0.32 to 0.54 L/h
Glass production rate range	0.09 to 0.19 kg/h
Nominal feed rate	0.33 L/h
Maximum feed rate	0.42 L/h
Process temperature	1125 to 1141°C

approximately 0.6 after 18 h. It is not known why this occurred. Regardless, these ferrous/ferric ratios are much higher than the maximum of about 0.20 seen in PSCM-19 output glass. Figure 27 illustrates the redox response.

A sample of the surface material of the melt was sectioned, polished, and examined with SEM/EDX. No significant amounts of calcium phosphate phases, as seen in PSCM-19, were observed. This was most probably caused by the over-reduced state of the LFMM glass as compared to the PSCM-19 glass. At the glass/cold cap interface, many spinel-type crystals and phases containing Ru and Si were observed. Figure 28 is a micrograph of this interface. Figure 29 gives EDX traces of the phases observed. The cold cap contained a high concentration of Si, with many partially dissolved silica particles. The high Si concentration in the cold cap would definitely inhibit melt rates, and serves to explain the poor melting behavior observed. Figures 30 and 31, respectively, show a micrograph of this region and an EDX trace of the partially dissolved Si particles observed.

FIGURE 27. Redox Response of LFMM-86-6 Glass

FIGURE 28. SEM Micrograph of LFMM-86-6 Cold Cap Interface

Examination of a thin section of output glass (sampled 10 h into the run) with optical microscopy and SEM/EDX revealed the presence of many crystals high in Cr, Fe, and Ni. These cubic spinel-type crystals measured approximately 2.5 to 26 microns across. A few Ru-containing crystals were also present. Figure 32 shows an SEM micrograph of the glass. The amount of crystal formation in the output glass was shown to increase with more reducing conditions during PSCM-19/19E. This behavior is seen again here. Figure 33 shows an EDX trace of the spinel-type crystal observed.

FIGURE 29. EDX Spectra of Features in Figure 28

1000x Magnification

FIGURE 30. SEM Micrograph of LFMM-86-6 Cold Cap

FIGURE 31. EDX Spectra of Feature 1 in Figure 30

FIGURE 32. SEM Micrograph of LFMM-86-6 Output Glass (10 h into Run)

FIGURE 33. EDX Spectra of Feature 1 in Figure 32

FEED RATE CORRELATIONS

Attempts were made to correlate the feed rates achieved in the LFMM to those of the PNL developmental joule-heated melters. Nominal and maximum feed rates as determined in the LFMM were compared to feed rates obtained in the engineering- and pilot-scale melters.

MAXIMUM FEED RATE CORRELATIONS

Data were available from HBCM and PSCM runs for three of the feeds run in the LFMM. These data are compared in Table 9 with the LFMM runs with the corresponding feeds.

Figure 34 compares the maximum feed rates achieved in the large-scale melters (specifically the HBCM and PSCM) with the maximum feed rates achieved in the LFMM. A positive correlation is seen for both melters. The lines drawn represent the "best fit" as determined by linear least-squares regression. Table 10 gives the regression parameters and equations with best fit. The slope of the PSCM correlation is greater than that of the HBCM correlation. This is expected, as the PSCM has a greater surface area and volume available for melting than does the HBCM. The R^2 values indicate that the PSCM

TABLE 9. Maximum Feed Rates Achieved by PNL Melters with Feeds Run in the LFMM

Feed	Maximum Feed Rate, L/h					
	LFMM	Run	HBCM	Run	PSCM	Run
SRL-TDSF-131	0.55	86-1	15.50	86-1	62.0	PSCM-4
Aged PSCM-22 (w/sugar)	0.34	86-2	13.70	85-1	39.1	PSCM-22
HWVP-RF, (a) as run in HBCM-86-2, 40D g/L oxides, no sugar	0.49	86-3	18.90	86-2	50.9	PSCM-22

(a) Hanford Waste Vitrification Program reference feed (HWVP-RF).

FIGURE 34. Maximum Feed Rate Correlations Between the PSCM/HBCM and the LFMM

TABLE 10. Maximum Feed Rate Correlations With LFMM

Melter	Equation	R ²
HBCM	$F_H = 13.59 * F_L + 9.76$	0.31
PSCM	$F_P = 103.20 * F_L + 2.99$	0.95

F_H = HBCM maximum feed rate, F_P = PSCM maximum feed rate, F_L = LFMM maximum feed rate.

correlation is better than the HBCM correlation. The equation derived for the HBCM-LFMM correlation could not be used with confidence for predicting maximum feed rates in the HBCM from LFMM data. The PSCM-LFMM correlation could be used with reasonable confidence. It should be noted here that the regressions are based on three data points only, and that more data must be collected to establish true confidence in predicting maximum feed rates from LFMM feed rates.

Figure 35 compares the normalized (with respect to melt surface area) maximum feed rates achieved by the melters as a function of feed. The normalized feed rates achieved in the PSCM and HBCM are much lower than those achieved in the LFMM because of the large differences in melt surface area. The relative magnitudes of the feed rates, as related to specific melters, compare reasonably well as a function of feed. The ratio of normalized maximum

FIGURE 35. Normalized Maximum Feed Rates Achieved by Melters

feed rates achieved in the LFMM to those obtained in the HBCM and PSCM are 2.47 ± 0.51 and 2.27 ± 0.12 , respectively. The LFMM correlates better with the PSCM than the HBCM in this analysis (smaller standard deviation), as was also noted for the linear feed rate correlations described earlier.

NOMINAL FEED RATE CORRELATIONS

The data used for the nominal feed rate correlations are listed in Table 11. The runs and feeds are identified.

Figure 36 plots the nominal feed rates achieved in the engineering-scale melters against the nominal rates achieved in the LFMM. Positive correlations are seen for all melters (ECM, HBCM, PSCM). The equations derived from linear least-squares regression are given in Table 12.

As evidenced by the R^2 values in Table 12, these correlations are rather poor; the best correlation was obtained for the ECM. This is caused by the large amount of scatter seen in the data. Some scatter was expected, as the data are based on observation of melt surfaces and will be influenced by the

TABLE 11. Nominal Feed Rates Achieved by Melters with Feeds Run in the LFMM

Feed	LFMM		ECM		HBCM		PSCM	
	Melter Run	Feed Rate, L/h	Melter Run	Feed Rate, L/h	Melter Run	Feed Rate, L/h	Melter Run	Feed Rate, L/h
SRL-TDSF-131	86-1	0.43	--	--	86-1	14.0	4	57.3
Aged PSCM-22, with sugar	86-2	0.29	86-1	2.25	85-1	11.0	22	35.9
HWVP-RF as run in HBCM 86-2, 400 g/L oxides	86-3	0.35	86-1	2.9	86-2	17.3	22	39.1
FRG 8	86-4	0.46	85-5	3.5	--	--	20	32.0
WV 182, no sugar	86-5	0.41	--	--	--	--	19	34.0
WV 182, 75 g/L sugar	86-6	0.33	84-5	1.8	--	--	19	20.0

FIGURE 36. Nominal Feed Rate Correlations Between the ECM, HBCM, PSCM and the LFMM

TABLE 12. Nominal Feed Rate Correlations With LFMM

Melter	Equation	R^2
ECM	$F_E = 8.92 * F_L - 0.57$	0.70
HBCM	$F_H = 16.44 * F_L + 8.22$	0.13
PSCM	$F_P = 71.82 * F_L + 9.21$	0.14

F_E = ECM nominal feed rate, F_H = HBCM nominal feed rate, F_P = PSCM nominal feed rate, F_L = LFMM nominal feed rate.

objectivity of the individual taking the data. These correlations cannot be used with reasonable confidence; however, they can be used to pinpoint a range of expected feed rates.

Note that the slopes of the correlations consistently increase with increase in melter size. This trend was noted earlier when discussing Figure 34. Further examination indicates that the slopes are related to specific melter surface area or volume. Table 13 lists the melter surface areas and volumes. Also shown are the ratios of these to those of the LFMM.

Comparing the ratios given in Table 13 with the slopes of the correlations shows that the slopes do not increase with the same level of magnitude as do the ratios. Regardless, the slopes reflect the increase in surface area and volume. The discrepancies are most probably due to inherent scale-up factors and the differences in geometric configuration of the melters. Further information can be gathered with further testing of the LFMM.

TABLE 13. Melter Surface Areas and Volumes

Melter	Surface Area, m^2	Surface Area as Ratioed to the LFMM	Volume, L	Volume as Ratioed to the LFMM
LFMM	0.0029	1.00	0.31	1.00
ECM	0.054	18.62	8.2	26.71
HBCM	0.25	86.21	50.0	162.87
PSCM	0.73	251.72	290.0	944.62

Figure 37 compares the nominal feed rates achieved by the melters, normalized with respect to melt surface area, as a function of feed. The relative magnitudes of the feed rates, as related to specific melters, compare reasonably well as a function of feed. The ratio of normalized nominal feed rates achieved in the LFMM to those obtained in the ECM, HBCM, and PSCM are 2.62 ± 0.56 , 2.22 ± 0.45 , and 2.83 ± 0.94 , respectively. The ratio of LFMM:HBCM is smaller than the ratio of LFMM:PSCM. This situation is the reverse of that observed when comparing the normalized maximum feed rates. One would expect them to be relatively similar. More data need to be collected and analyzed to resolve this discrepancy.

FIGURE 37. Normalized Nominal Feed Rates Achieved by Melters

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APPENDIX

LFMM FEED COMPOSITIONS AND FEED AND GLASS ANALYSES

TABLE A.1. SRL-TDSF-131 Feed Composition (LFMM-86-1 Feed)

Compounds	Concentration, g/L	Oxide	TDSF Waste	(1) Frit 131	(2) Zeolite	Total	Final Glass Composition, wt%
Frit 131	341.30						
Zeolite	9.90						
Fe(OH) ₃	81.20	Fe ₂ O ₃	60.71			60.71	12.70
Al(OH) ₃	34.20	Al ₂ O ₃	22.34		1.91	24.25	5.10
MnCO ₃	22.3 ^(a)	MnO	13.78			13.78	2.90
(Mn(CHO ₂) ₂)	(28.2)						
Ni(CO ₂ H) ₂	8.30	NiO	4.07			4.07	0.80
Ca(CHO ₂) ₂	16.70	CaO	7.22		1.05	8.27	1.70
SiO ₂	15.60	SiO ₂	15.60	197.61	4.74	217.95	45.60
NaCHO ₂	8.42	Na ₂ O	3.83	60.43	0.24	64.50	13.60
NaNO ₃	2.01		0.72				
Na ₂ SO ₄	0.72	Na ₂ SO ₄	0.72			0.72	0.10
HCHO ₂	pH 5.5						
		B ₂ O ₃		50.19		50.20	10.50
		Li ₂ O		19.42		19.40	4.10
		MgO		6.84		6.80	1.40
		TiO ₂		3.40		3.40	0.70
		La ₂ O ₃		1.72		1.70	0.40
		ZrO ₂		1.72		1.70	0.40
TOTAL	546.60		128.99	341.33	7.94	477.45	100.00

(a) Value based on reaction of MnCO₃ and MCHO₂.(1) Frit-131 Composition (-200 mesh) (2) Zeolite Composition
(Linde Ionsiv IE-95)

Oxide	wt%	Component	wt%
SiO ₂	57.9	CaAl ₂ Si ₄ O ₁₂ •6H ₂ O	80.0
B ₂ O ₃	14.7	NaCa ₁ •5A ₁₃ Si ₈ O ₂₄ •8H ₂ O	20.0
Na ₂ O	17.7		
Li ₂ O	5.7	Assumed Oxide Form	
MgO	2.0		
TiO ₂	1.0	CaO	10.6
La ₂ O ₃	0.5	Al ₂ O ₃	19.2
ZrO ₂	0.5	SiO ₂	47.7
		Na ₂ O	2.5
Total	100.0	H ₂ O	20.0
		Total	100.0

TABLE A.2. ICP Analyses of Feed Samples Taken During LFMM-86-1

Hours Into Run Sample Number	1.03		2.00		5.70		8.70		Bulk (a)		Target	
	1		2		3		4					
	g/L Oxides	wt%	g/L Oxides	wt%	g/L Oxides	wt%	g/L Oxides	wt%	g/L Oxides	wt%	g/L Oxides	wt%
Al ₂ O ₃	26.30	5.30	27.00	5.31	26.00	5.32	26.30	5.33	25.80	5.16	24.25	5.10
B ₂ O ₃	50.80	10.24	51.80	10.18	49.80	10.18	50.30	10.20	51.10	10.22	50.20	10.50
CaO	9.35	1.88	9.85	1.94	9.32	1.91	9.35	1.90	9.53	1.91	8.27	1.70
Fe ₂ O ₃	67.20	13.55	68.80	13.52	66.00	13.49	66.60	13.50	67.20	13.44	12.70	13.44
La ₂ O ₃	1.53	0.31	1.57	0.31	1.51	0.31	1.54	0.31	1.35	0.27	1.70	0.40
Li ₂ O	19.80	3.99	20.00	3.93	19.20	3.93	19.50	3.95	18.35	3.67	19.40	4.10
MgO	6.75	1.36	6.94	1.36	6.64	1.36	6.81	1.38	5.58	1.12	6.80	1.40
MnO	17.10	3.45	17.50	3.44	16.70	3.41	16.80	3.41	13.97	2.79	13.78	2.90
Na ₂ O	70.40	14.19	72.20	14.19	69.20	14.15	69.20	14.03	70.65	14.13	64.50	13.60
NiO	3.41	0.69	3.46	0.68	3.36	0.69	3.48	0.71	3.16	0.63	4.07	0.80
SiO ₂	218.00	43.94	224.00	44.03	216.00	44.16	218.00	44.20	228.00	45.60	217.95	45.60
TiO ₂	3.66	0.74	3.74	0.74	3.58	0.73	3.60	0.73	3.62	0.72	3.40	0.70
ZrO ₂	1.78	0.36	1.90	0.37	1.79	0.37	1.76	0.36	1.73	0.34	1.70	0.40
TOTAL	496.08	100.00	508.76	100.00	489.10	100.00	493.24	100.00	500.02	100.00	477.45	100.00

(a) Analysis of feed as received.

TABLE A.3. Normalized Compositions of Glass Samples Taken During LFMM-86-1
(Analysis performed by ICP)

		Normalized Weight Percent							
Hours Into Run		1.00	2.67	3.83	7.33	10.33	13.33		
Oxide	Starting Glass	#1	#2	#3	#4	#5	#6	Bulk Feed	Target
Al ₂ O ₃	5.16	5.23	5.21	5.19	5.16	5.16	5.18	5.16	5.10
B ₂ O ₃	9.89	9.84	9.92	9.89	9.99	9.93	9.92	10.22	10.50
CaO	1.93	1.92	1.87	1.88	1.86	1.84	1.86	1.91	1.70
Fe ₂ O ₃	13.67	13.24	13.33	13.12	13.13	13.20	13.23	13.44	12.70
La ₂ O ₃	0.26	0.29	0.30	0.30	0.29	0.29	0.30	0.27	0.40
Li ₂ O	3.43	3.85	3.81	3.86	3.89	3.87	3.84	3.67	4.10
MgO	1.10	1.28	1.28	1.23	1.26	1.24	1.28	1.12	1.40
MnO	2.82	2.78	2.80	2.72	2.71	2.72	2.73	2.79	2.90
Na ₂ O	13.57	13.54	13.33	13.94	13.73	13.90	13.73	14.13	13.60
NiO	0.69	0.99	0.94	0.85	0.77	0.77	0.74	0.63	0.80
SiO ₂	46.46	45.98	46.09	45.99	46.15	46.01	46.14	45.60	45.60
TiO ₂	0.70	0.70	0.71	0.70	0.72	0.72	0.72	0.72	0.70
ZrO ₂	0.32	0.35	0.33	0.34	0.34	0.34	0.34	0.34	0.40
TOTAL	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	99.90
Fe ²⁺ /Fe ³⁺	0.05	0.23	0.33	0.32	0.62	0.62	0.66		

TABLE A.4. Composition of Simulated Melter Feed Slurry^(a) Used in LFMM-86-2

Compound ^(b)	Concentration, g/L	Assumed Oxide	Concentration, g/L	Glass wt%
Fe(OH) ₃	59.5	Fe ₂ O ₃	44.4	11.10
Al(OH) ₃	26.3	Al ₂ O ₃	17.2	4.30
NaNO ₃	13.4	Na ₂ O	10.7	2.68
Cr(OH) ₃	7.21	Cr ₂ O ₃	5.3	1.33
NaOH	3.58	ZrO ₂	2.4	0.60
Na ₂ C ₂ O ₄ ^(c)	3.37	SiO ₂	3.0	0.75
Zr(OH) ₄	3.11	NiO	2.4	0.50
SiO ₂	3.00	SO ₃	1.5	0.38
Ni(OH) ₂	3.00	La ₂ O ₃	2.2	0.55
Na ₂ SO ₄	2.32	MoO ₃	1.2	0.30
LaF ₃	2.16	Nd ₂ O ₃	2.1	0.53
Na ₂ MoO ₄ ·2H ₂ O	2.00	Cs ₂ O	1.0	0.25
Nd(OH) ₃	1.84	CeO ₂	0.7	0.18
CsOH	1.05	CuO	0.6	0.15
Ce(OH) ₃	0.79	MnO ₂	0.7	0.18
Cu(OH) ₂	0.74	BaO	0.4	0.10
Mn(OH) ₂	0.74	F ⁻	1.2	0.30
BaSO ₄	0.53	SrO	0.4	0.10
NdF ₃	0.58	Pr ₆ O ₁₁	0.4	0.10
Sr(OH) ₂	0.47	MgO	0.3	0.08
Pr(OH) ₃	0.47	CaO	0.3	0.08
La(OH) ₃	0.47	Y ₂ O ₃	0.2	0.05
NaF	0.47	Sm ₂ O ₃	0.2	0.05
Mg(OH) ₂	0.42	TOC	0.6	0.15
CaF ₂	0.42	I ⁻	0.04	0.01
Y(OH) ₃	0.26			
Sm(OH) ₃	0.21			
NaI	0.05			
SUBTOTALS	138.56 g/L		99.4 g/L	24.9%
HR39 Frit ^(d)	300.0	SiO ₂	201.8	50.44
		B ₂ O ₃	38.2	9.56
		Na ₂ O	30.8	7.69
		Li ₂ O	15.0	3.75
		CaO	11.2	2.81
		MgO	3.0	0.75
SUBTOTALS	300.0 g/L		300.0 g/L	75.0 wt%
TOTALS:	438.5 g/L		400.0 g/L	100.0 wt%
Reductants: 12.2 g/L formic acid, 3.5 g/L sugar				

(a) Basis: 400 g oxide/L slurry, 25% waste oxide loading.

(b) Prior to Reaction with Formic Acid.

(c) TOC substitute.

(d) HK39 frit size distribution as follows: 1/3 to be -80 + 200 mesh
2/3 to be -200 mesh.

TABLE A.5. Feed Sample Compositions Taken During LFMM-86-2

Oxide	Hours Into Run Sample Number		1.00		2.12		7.00		10.00		11.00		14.00		Bulk		Target	
	1		2		3		4		5		6							
	g/L Oxides	wt%	g/L Oxides	wt%	g/L Oxides	wt%	g/L Oxides	wt%	g/L Oxides	wt%	g/L Oxides	wt%	g/L Oxides	wt%	g/L Oxides	wt%	g/L Oxides	wt%
Al ₂ O ₃	17.80	4.24	17.70	4.22	17.40	4.17	17.80	4.20	17.80	4.19	16.60	3.89	16.60	4.01	17.2	4.30		
B ₂ O ₃	36.70	8.74	37.00	8.82	36.20	8.67	37.20	8.78	37.10	8.74	37.10	8.69	36.10	8.72	38.2	9.56		
BaO	1.14	0.27	1.13	0.27	1.15	0.28	1.16	0.27	1.15	0.27	1.15	0.27	1.07	0.26	0.40	0.10		
CaO	10.80	2.57	10.90	2.60	10.80	2.59	10.80	2.55	11.00	2.59	11.30	2.65	11.30	2.73	11.5	2.89		
CeO ₂	0.56	0.13	0.65	0.15	0.59	0.14	0.61	0.14	0.59	0.14	0.39	0.09	0.70	0.17	0.7	0.18		
Cr ₂ O ₃	5.07	1.21	4.91	1.17	5.11	1.22	5.04	1.19	5.15	1.21	5.13	1.20	4.90	1.18	5.3	1.33		
Cs ₂ O	1.30	0.31	1.30	0.31	1.40	0.34	1.40	0.33	1.70	0.40	1.40	0.33	1.32	0.32	1.0	0.25		
CuO	0.65	0.15	0.63	0.15	0.64	0.15	0.64	0.15	0.64	0.15	0.64	0.15	0.44	0.11	0.6	0.15		
F(a)	1.20	0.29	1.20	0.29	1.20	0.29	1.20	0.28	1.20	0.28	1.20	0.28	1.20	0.29	1.2	0.30		
Fe ₂ O ₃	44.50	10.59	43.50	10.37	44.60	10.68	44.60	10.53	44.50	10.49	44.80	10.49	44.50	10.75	44.4	11.10		
La ₂ O ₃	2.09	0.50	2.16	0.51	2.03	0.49	2.12	0.50	2.12	0.50	2.13	0.50	1.90	0.46	2.2	0.55		
Li ₂ O	14.30	3.40	14.40	3.43	14.10	3.38	14.50	3.42	14.60	3.44	14.50	3.40	13.30	3.21	15.0	3.75		
MgO	3.39	0.81	3.51	0.84	3.42	0.82	3.41	0.81	3.45	0.81	3.48	0.81	2.04	0.49	3.3	0.83		
MnO ₂	0.80	0.19	0.79	0.19	0.80	0.19	0.81	0.19	0.79	0.19	0.81	0.19	0.76	0.18	0.7	0.18		
MoO ₃	1.42	0.34	1.40	0.33	1.41	0.34	1.40	0.33	1.43	0.34	1.42	0.33	0.75	0.18	1.2	0.30		
Na ₂ O	43.00	10.24	43.30	10.32	43.40	10.39	43.40	10.25	43.80	10.32	44.60	10.44	49.10	11.86	41.5	10.37		
Nd ₂ O ₃	1.92	0.46	1.91	0.46	1.96	0.47	1.94	0.46	1.96	0.46	1.93	0.45	0.93	0.22	2.1	0.53		
NiO	2.26	0.54	2.18	0.52	2.23	0.53	2.25	0.53	2.25	0.53	2.16	0.51	1.91	0.46	2.4	0.60		
SiO ₂	225.00	53.57	225.00	53.62	223.00	53.40	227.00	53.60	227.00	53.49	230.00	53.86	219.00	52.92	204.8	51.19		
SO ₃ (a)	1.50	0.36	1.50	0.36	1.50	0.36	1.50	0.35	1.50	0.35	1.50	0.35	1.50	0.36	1.5	0.38		
SrO	0.39	0.09	0.37	0.09	0.39	0.09	0.38	0.09	0.38	0.09	0.38	0.09	0.36	0.09	0.4	0.10		
TiO ₂	1.17	0.28	1.15	0.27	1.17	0.28	1.17	0.28	1.17	0.28	1.18	0.28	1.13	0.27	0.0	0.00		
TOC(a)	0.60	0.14	0.60	0.14	0.60	0.14	0.60	0.14	0.60	0.14	0.60	0.14	0.60	0.14	0.6	0.15		
ZrO ₂	2.47	0.59	2.42	0.58	2.53	0.61	2.55	0.60	2.49	0.59	2.65	0.62	2.45	0.59	2.4	0.60		
TOTAL	420.03	100.00	419.61	100.00	417.62	100.00	423.48	100.00	424.37	100.00	427.05	100.00	413.86	100.00	398.6	99.69		
pH	10.50		10.60		10.50		10.50		10.60		10.60		10.60					

(a) Not analyzed for, assumed Target Value.

TABLE A.6. Normalized Compositions of Glass Samples Taken
During LFMM-86-2 (Analysis Performed by ICP)

Hours Into Run	Normalized Weight Percent								Bulk Feed	Target
	Starting Glass	1.00	4.08	7.00	10.00	11.00	14.00	14.83		
Oxide		#1	#2	#3	#4	#5	#6	#7		
Al ₂ O ₃	4.03	4.09	4.11	4.10	4.13	4.16	4.13	4.13	4.01	4.30
B ₂ O ₃	10.51	10.61	10.28	9.05	8.58	8.74	8.59	8.68	8.72	9.56
BaO	0.12	0.11	0.15	0.23	0.26	0.26	0.26	0.26	0.26	0.10
CaO	1.28	1.33	1.61	2.33	2.59	2.57	2.56	2.54	2.73	2.89
CeO ₂	0.18	0.00	0.05	0.10	0.12	0.13	0.13	0.13	0.17	0.18
Cr ₂ O ₃	0.11	0.15	0.33	0.97	1.13	1.16	1.14	1.11	1.18	1.33
Cs ₂ O	0.06	0.06	0.12	0.26	0.30	0.31	0.31	0.30	0.32	0.25
CuO	0.44	0.02	0.04	0.11	0.16	0.14	0.15	0.14	0.11	0.15
F(a)	0.30	0.30	0.30	0.30	0.30	0.30	0.30	0.30	0.29	0.30
Fe ₂ O ₃	12.13	12.43	11.97	10.95	11.02	10.51	10.66	10.65	10.75	11.10
La ₂ O ₃	0.11	0.13	0.21	0.40	0.48	0.48	0.48	0.48	0.46	0.55
Li ₂ O	3.68	3.77	3.64	3.46	3.38	3.44	3.41	3.40	3.21	3.75
MgO	1.43	1.49	1.34	0.93	0.82	0.81	0.82	0.82	0.49	0.83
MnO ₂	3.18	3.25	2.61	0.90	0.32	0.28	0.32	0.32	0.18	0.18
MoO ₃	0.76	0.00	0.18	0.31	0.35	0.35	0.36	0.36	0.18	0.30
Na ₂ O	12.63	12.93	12.07	10.85	10.53	10.21	10.36	10.15	11.86	10.37
Nd ₂ O ₃	0.54	0.04	0.11	0.33	0.42	0.42	0.42	0.42	0.22	0.53
NiO	1.21	1.27	1.20	0.73	0.58	0.60	0.63	0.62	0.46	0.60
SiO ₂	44.88	45.68	47.60	52.07	53.03	53.64	53.48	53.67	52.92	51.19
SO ₃ (a)	0.38	0.38	0.38	0.38	0.38	0.38	0.38	0.38	0.36	0.38
SrO	0.02	0.02	0.03	0.07	0.09	0.09	0.09	0.09	0.09	0.10
TiO ₂	0.74	0.73	0.63	0.37	0.30	0.29	0.29	0.29	0.27	0.00
TOC(a)	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.14	0.15
ZrO ₂	1.11	1.03	0.89	0.67	0.60	0.57	0.62	0.58	0.59	0.60
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	99.69
Fe ²⁺ /Fe ³⁺	<.005	<.005	0.06	0.13	0.15	0.12	0.18	0.18		

(a) Not analyzed for assumed, target level.

TABLE A.7. Composition of HWVP Simulated Melter Feed Used in LFMM-B6-3

	Total Oxide (g/L)		400	
	Waste Loading (%)		25	
	Waste Oxides (g/L)		100	
Compound(a)	Compound Concentration, g/L	Oxide	Oxide Concentration, g/L	Glass wt%
Al(OH) ₃	26.62	Al ₂ O ₃	17.40	4.35
BaSO ₄	0.61	BaO	0.40	0.10
CaF ₂	0.45	CaO	0.32	0.08
Ce(OH) ₃	0.80	CeO ₂	0.72	0.18
Cr(OH) ₃	7.32	Cr ₂ O ₃	5.40	1.35
CsOH·3H ₂ O	1.45	Cs ₂ O	1.00	0.25
Cu(OH) ₂	0.74	CuO	0.60	0.15
Fe(OH) ₃	60.06	Fe ₂ O ₃	44.88	11.22
Mg(OH) ₂	0.46	MgO	0.32	0.08
Mn(OH) ₂	0.74	MnO ₂	0.72	0.18
Na ₂ MoO ₄ ·2H ₂ O	2.02	MoO ₃	1.20	0.30
Ni(OH) ₂	2.98	NiO	2.40	0.60
Pr(OH) ₃	0.45	Pr ₆ O ₁₁	0.40	0.10
SiO ₂	3.08	SiO ₂	3.08	0.77
Sm(OH) ₃	0.23	Sm ₂ O ₃	0.20	0.05
Sr(OH) ₂ ·8H ₂ O	1.02	SrO	0.40	0.10
Y(OH) ₃	0.25	Y ₂ O ₃	0.20	0.05
Zr(OH) ₄ ·5H ₂ O	4.94	ZrO ₂	2.44	0.61
		La ₂ O ₃	2.20	0.55
La(OH) ₃	0.47			
LaF ₃	2.16			
		Na ₂ O	10.88	2.72
NaOH	3.26			
NaNO ₃	13.60			
Na ₂ C ₂ O ₄	3.42			
		Nd ₂ O ₃	2.12	0.53
Nd(OH) ₃	1.84			
NdF ₃	0.64			
		F-	1.20	0.30
NaF	0.22			
		SO ₃	1.52	0.38
Na ₂ SO ₄	2.33			
Subtotals	142.16		100.00	25.00
	HW-39 Frit	SiO ₂	201.76	50.44
		B ₂ O ₃	38.24	9.56
		Na ₂ O	30.76	7.69
		Li ₂ O	15.00	3.75
		CaO	11.24	2.81
		MgO	3.00	0.75
	Subtotals		300.00 g/L	75.0 wt%
	Totals		400.00 g/L	100.00 wt%

(a) Prior to reaction with formic acid.

TABLE A.8. Feed Sample Compositions Taken During LFMM-86-3

Hours Into Run	1.00		5.00		7.00		10.00		12.50					
Sample Number	1		2		3		4		5		Bulk		Target	
Oxide	g/L	wt%	g/L	wt%	g/L	wt%	g/L	wt%	g/L	wt%	g/L	wt%	g/L	wt%
Al ₂ O ₃	13.80	4.45	14.00	4.40	14.40	6.14	14.60	5.58	14.00	4.60	13.10	5.20	17.40	4.35
B ₂ O ₃	29.80	9.60	30.80	9.69	20.20	8.62	23.60	9.02	29.20	9.59	23.30	9.25	38.24	9.56
BaO	0.31	0.10	0.31	0.10	0.33	0.14	0.33	0.13	0.31	0.10	0.28	0.11	0.40	0.10
CaO	4.99	1.61	5.09	1.60	3.50	1.49	4.04	1.54	4.74	1.56	3.88	1.54	11.56	2.89
CeO ₂	0.45	0.14	0.56	0.18	0.63	0.27	0.51	0.20	0.50	0.16	0.70	0.28	0.72	0.18
Cr ₂ O ₃	5.08	1.64	5.04	1.59	5.22	2.23	5.32	2.03	5.05	1.66	5.04	2.00	5.40	1.35
Cs ₂ O	1.10	0.35	1.10	0.35	1.10	0.47	1.10	0.42	1.10	0.36	0.97	0.38	1.00	0.25
CuO	0.48	0.15	0.49	0.15	0.51	0.22	0.54	0.21	0.49	0.16	0.44	0.17	0.60	0.15
F(a)	1.20	0.39	1.20	0.38	1.20	0.51	1.20	0.46	1.20	0.39	1.20	0.48	1.20	0.30
Fe ₂ O ₃	34.00	10.96	34.20	10.76	35.30	15.06	35.70	13.65	34.60	11.36	35.40	14.05	44.88	11.22
La ₂ O ₃	1.55	0.50	1.64	0.52	1.62	0.69	1.63	0.62	1.65	0.54	1.39	0.55	2.20	0.55
Li ₂ O	10.30	3.32	10.50	3.30	6.33	2.70	7.89	3.02	9.84	3.23	6.95	2.76	15.00	3.75
MgO	4.10	1.32	4.22	1.33	2.92	1.25	3.55	1.36	3.98	1.31	2.10	0.83	3.32	0.83
MnO ₂	0.59	0.19	0.60	0.19	0.65	0.28	0.63	0.24	0.60	0.20	0.60	0.24	0.72	0.18
MoO ₃	1.25	0.40	1.24	0.39	1.31	0.56	1.21	0.46	1.29	0.42	0.60	0.24	1.20	0.30
Na ₂ O	39.70	12.79	39.20	12.33	30.30	12.93	32.80	12.54	38.30	12.58	31.40	12.46	41.64	10.41
Nd ₂ O ₃	1.40	0.45	1.47	0.46	1.46	0.62	1.49	0.57	1.45	0.48	0.53	0.21	2.12	0.53
NiO	1.83	0.59	1.81	0.57	1.91	0.81	1.89	0.72	1.84	0.60	1.66	0.66	2.40	0.60
SiO ₂	154.00	49.62	160.00	50.32	101.00	43.10	119.00	45.51	150.00	49.25	118.00	46.83	204.84	51.21
SO ₃ ^(a)	1.50	0.48	1.50	0.47	1.50	0.64	1.50	0.57	1.50	0.49	1.50	0.60	1.52	0.38
SrO	0.34	0.11	0.34	0.11	0.35	0.15	0.35	0.13	0.34	0.11	0.33	0.13	0.40	0.10
TiO ₂	0.07	0.02	0.06	0.02	0.06	0.03	0.08	0.03	0.06	0.02	0.20	0.08	0.00	0.00
ZrO ₂	1.91	0.62	1.99	0.63	1.96	0.84	1.94	0.74	1.93	0.63	1.79	0.71	2.44	0.61
Total	310.35	100.00	317.96	100.00	234.36	100.00	261.50	100.00	304.57	100.00	251.96	100.00	399.20	99.80
pH	8.60		8.60		8.60		8.60		8.70		8.60			

(a) Not analyzed for, assumed target value.

TABLE A.9. Normalized Composition of Glass Samples Taken During LFMM-86-3

Hours Into Run		1.00	4.00	7.00	10.00	10.83	11.42		
Oxide	Starting Glass	Normalized Weight Percent						Bulk Feed	Target
		#1	#2	#3	#4	#5	#6		
Al ₂ O ₃	4.03	4.12	4.21	4.59	4.90	5.01	5.35	5.20	4.35
B ₂ O ₃	10.51	10.63	10.40	9.60	9.28	9.16	8.96	9.25	9.56
BaO	0.12	0.12	0.12	0.11	0.12	0.12	0.12	0.11	0.10
CaO	1.28	1.46	1.45	1.47	1.49	1.48	1.50	1.54	2.89
CeO ₂	0.18	0.00	0.08	0.13	0.17	0.17	0.17	0.28	0.18
Cr ₂ O ₃	0.11	0.11	0.38	1.08	1.32	1.40	1.40	2.00	1.33
Cs ₂ O	0.06	0.05	0.11	0.23	0.29	0.33	0.33	0.38	0.25
CuO	0.44	0.02	0.04	0.12	0.14	0.15	0.15	0.17	0.15
F(a)	0.30	0.30	0.31	0.30	0.30	0.31	0.30	0.48	0.30
Fe ₂ O ₃	12.13	11.95	11.93	12.11	12.50	12.74	12.59	14.02	11.22
La ₂ O ₃	0.11	0.15	0.22	0.41	0.48	0.50	0.50	0.55	0.55
Li ₂ O	3.68	3.86	3.67	3.30	3.20	3.15	3.05	2.76	3.75
MgO	1.43	1.49	1.46	1.35	1.35	1.33	1.28	0.83	0.83
MnO ₂	3.18	3.15	2.64	1.33	1.03	0.94	0.80	0.24	0.18
MoO ₃	0.76	0.14	0.20	0.33	0.39	0.41	0.40	0.24	0.30
Na ₂ O	12.63	13.06	12.95	12.72	12.70	12.54	12.49	12.46	10.41
Nd ₂ O ₃	0.54	0.06	0.13	0.34	0.42	0.44	0.44	0.21	0.53
NiO	1.21	1.02	1.00	0.85	0.86	0.91	0.84	0.66	0.60
SiO ₂	44.88	46.07	46.61	47.94	47.44	47.31	47.74	46.83	51.21
SO ₃ (a)	0.38	0.38	0.39	0.38	0.39	0.39	0.38	0.60	0.38
SrO	0.02	0.02	0.04	0.08	0.10	0.10	0.11	0.13	0.10
TiO ₂	0.74	0.70	0.58	0.27	0.21	0.18	0.17	0.08	0.00
ZrO ₂	1.11	0.97	0.91	0.78	0.76	0.80	0.77	0.71	0.61
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	99.80
Fe ²⁺ /Fe ³⁺	<.005	<.005	<.005	<.005	<.005	<.005	<.005		

(a) Not analyzed for, assumed target value.

TABLE A.10. FRG 8 Feed Composition Used in LFMM-86-4

Compound	Concentration, g/L	Assumed Oxide	Concentration, g/L	Glass wt%
$\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	100.84	Al_2O_3	8.40	2.40
$\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$	143.74	B_2O_3	52.50	15.00
$\text{Ba}(\text{NO}_3)_2$	7.76	BaO	4.55	1.30
$\text{Ca}(\text{NO}_3)_2$	9.10	CaO	2.84	0.81
$\text{Ce}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	2.88	CeO_2	1.02	0.29
$\text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	12.90	Cr_2O_3	2.45	0.70
CsNO_3	27.98	Cs_2O	20.23	5.78
$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	178.65	Fe_2O_3	35.32	10.09
$\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$	11.08	La_2O_3	4.20	1.20
$\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	11.80	MgO	1.86	0.53
$\text{Mn}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$	12.64	MnO_2	2.70	0.77
NaNO_3	51.10	Na_2O	42.00	12.00
Nd_2O_3	3.85	Nd_2O_3	3.85	1.10
$\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	4.09	NiO	1.05	0.30
PbO	2.42	PbO	2.42	0.69
SiO_2	157.50	SiO_2	157.50	45.00
$\text{Sr}(\text{NO}_3)_2$	13.72	SrO	6.72	1.92
ZnO	0.18	ZnO	0.18	0.05
$\text{ZrO}(\text{NO}_3)_2 \cdot 2\text{H}_2\text{O}$	0.53	ZrO_2	0.24	0.07
Total	752.76		350.03	100.00

TABLE A.11. ICP Analyses of Feed Samples Taken During LFMM-86-4

Hours Into Run	3.00		8.40					
Sample Number	1		2		Bulk Feed		Target	
Oxide	g/L	wt%	g/L	wt%	g/L	wt%	g/L	wt%
Al ₂ O ₃	9.09	2.09	9.38	2.11	8.76	2.26	8.40	2.40
B ₂ O ₃	71.80	16.53	76.50	17.23	69.90	18.04	52.50	15.00
BaO	4.68	1.08	4.87	1.10	4.55	1.17	4.55	1.30
CaO	2.83	0.65	2.89	0.65	2.73	0.70	2.84	0.81
CeO ₂	1.28	0.29	1.29	0.29	1.22	0.31	1.02	0.29
Cr ₂ O ₃	2.62	0.60	2.70	0.61	2.54	0.66	2.45	0.70
Cs ₂ O	23.50	5.41	24.20	5.45	23.00	5.94	20.23	5.78
Fe ₂ O ₃	36.70	8.45	37.90	8.54	35.60	9.19	35.32	10.09
La ₂ O ₃	4.32	0.99	4.49	1.01	4.19	1.08	4.20	1.20
MgO	2.21	0.51	2.25	0.51	2.13	0.55	1.86	0.53
MnO ₂	3.32	0.76	3.39	0.76	3.18	0.82	2.70	0.77
Na ₂ O	53.40	12.29	55.00	12.39	53.50	13.81	42.00	12.00
Na ₂ O ₃	4.12	0.95	4.25	0.96	3.97	1.02	3.85	1.10
NiO	1.12	0.26	1.16	0.26	1.05	0.27	1.05	0.30
PbO	0.00	0.00	0.00	0.00	0.00	0.00	2.42	0.69
SiO ₂	206.00	47.43	206.00	46.40	164.00	42.33	157.50	45.00
SrO	6.79	1.56	7.05	1.59	6.58	1.70	6.72	1.92
ZnO	0.20	0.05	0.21	0.05	0.19	0.05	0.18	0.05
ZrO ₂	0.36	0.08	0.39	0.09	0.31	0.08	0.24	0.07
Total	434.34	100.00	443.92	100.00	387.40	100.00	350.03	100.00

TABLE A.12. ICP Analyses of Glass Samples Taken During LFMM-86-4

Hours Into Run	0.00	11.50		
<u>Oxide</u>	<u>Starting Glass</u>	<u>Final Glass</u>	<u>Bulk Feed</u>	<u>Target</u>
Al ₂ O ₃	4.06	2.35	2.26	2.40
B ₂ O ₃	11.17	17.29	18.04	15.00
BaO	0.91	1.18	1.17	1.30
CaO	1.34	0.74	0.70	0.81
CeO ₂	0.13	0.30	0.31	0.29
Cr ₂ O ₃	1.11	0.63	0.66	0.70
Cs ₂ O	4.87	5.50	5.94	5.78
Fe ₂ O ₃	10.36	9.42	9.19	10.09
La ₂ O ₃	1.01	1.09	1.08	1.20
MgO	0.86	0.55	0.55	0.53
MnO ₂	0.86	0.84	0.82	0.77
Na ₂ O	14.72	13.09	13.81	12.00
Nd ₂ O ₃	0.61	1.01	1.02	1.10
NiO	1.51	0.44	0.27	0.30
PbO	0.23	0.00	0.00	0.69
SiO ₂	44.37	43.78	42.33	45.00
SrO	1.45	1.71	1.70	1.92
ZnO	0.00	0.00	0.05	0.05
ZrO ₂	0.41	0.09	0.08	0.07
Total	100.00	100.00	99.98	100.00

TABLE A.13. Simulated West Valley Melter Feed Composition (WV182)
Used in LFMM-86-5 and LFMM-86-6^(a)

Substituted Waste Compound	g/L	Assumed Oxide Compound	g/L	Final Glass Oxide	g/L	wt%	Normalized ICP Analysis of Feed wt%	
							LFMM-86-5	LFMM-86-6
Fe(NO ₃) ₃ ·9H ₂ O	158.0	Fe ₂ O ₃	41.51	SiO ₂	156.4	44.7	45.00	44.37
NaNO ₃	8.9	Na ₂ O	3.64	Na ₂ O	52.8	15.1	14.86	14.57
Al(NO ₃) ₃ ·9H ₂ O	14.7	Al ₂ O ₃	2.00	B ₂ O ₃	44.4	12.7	13.09	13.18
Cr(NO ₃) ₃ ·6H ₂ O	12.5	Cr ₂ O ₃	2.38	Fe ₂ O ₃	43.0	12.3	11.79	11.48
Ni(NO ₃) ₂ ·6H ₂ O	6.1	NiO	1.58	RE ₂ O ₃	10.2	2.9	0.06	0.07
Ce(NO ₃) ₃ ·9H ₂ O	14.6	RE ₂ O ₃	10.26	CeO ₂	0.6	0.17	1.75	1.72
Rare Earth	1.7	CeO ₂	0.60	Al ₂ O ₃	8.4	2.4	2.58	2.49
KNO ₃	0.60	K ₂ O	0.28	CaO	10.5	3.0	2.48	2.44
CsNO ₃	0.48	Cs ₂ O	0.35	P ₂ O ₅	9.1	2.6	2.51	2.58
Mn(NO ₃) ₂ ·4H ₂ O	4.9	MnO ₂	1.68	Cr ₂ O ₃	2.38	0.68	0.68	0.66
ZrO(NO ₃) ₂ ·2H ₂ O	14.7	ZrO ₂	5.88	ZrO ₂	5.88	1.68	1.06	2.33
NaCl	0.34	--	--	MnO ₂	1.72	0.49	2.80	2.73
Ba(NO ₃) ₂	0.36	BaO	0.21	NiO	1.58	0.45	0.45	0.43
FePO ₄ ·2H ₂ O	24.1	P ₂ O ₅	9.14	Cs ₂ O	0.35	0.10	0.08	0.07
Na ₂ SO ₄	0.87	SO ₃	0.49	K ₂ O	0.60	0.17	0.17 ^(b)	0.19 ^(b)
RuO ₂	0.28	RuO ₂	0.28	BaO	0.35	0.10	0.08	0.07
Y ₂ O ₃	0.07	Y ₂ O ₃	0.07	SO ₃	0.70	0.20	0.20 ^(b)	0.22 ^(b)
Sr(NO ₃) ₂	0.22	SrO	0.11	RuO ₂	0.28	0.08	0.08 ^(b)	0.09 ^(b)
Subtotals	263.4		80.5	Y ₂ O ₃	0.07	0.02	0.02 ^(b)	0.02
				SrO	0.10	0.03	0.06	0.06
				TiO ₂	0.10	0.03	0.04	0.04
				MgO	0.35	0.10	0.17	0.19
					349.9	100.0	100.00	100.00

(a) LFMM-86-5 feed contains no sugar, LFMM-86-6 feed contains 75 g/L sugar.

(b) Not analyzed for, assumed target levels prior to normalization.

TABLE A.13. (contd)

Substituted Waste Compound	g/L	Assumed Oxide Compound	g/L
<u>Zeolite</u>			
SiO ₂	24.1	SiO ₂	24.1
Al ₂ O ₃	6.5	Al ₂ O ₃	6.5
Na ₂ O	1.3	Na ₂ O	1.3
Fe ₂ O ₃	1.4	Fe ₂ O ₃	1.4
CaO	1.5	CaO	1.5
SO ₃	0.2	SO ₃	0.2
K ₂ O	0.3	K ₂ O	0.3
BaO	0.1	BaO	0.1
MgO	0.4	MgO	0.4
TiO ₂	0.1	TiO ₂	0.1
H ₂ O	4.5		35.9
Subtotals	40.4		

Glass Formers and Additive

SiO ₂	132.3	SiO ₂	132.3
Na ₂ B ₄ O ₇ ·10H ₂ O	128.2(c)	B ₂ O ₃	44.6(c)
Ca(NO ₃) ₂	26.3	CaO	9.0
Na ₂ C ₂ O ₂	56.0	Na ₂ O	47.7
Sugar	(d)	--	--
Subtotals	342.8		233.6
Totals	646.6		350.0

(c) Assumes a 5% loss of B due to volatility.

(d) Sugar concentration of 0 g/L used for LFMM-86-5 feed, 75 g/L used for LFMM-86-6 feed.

TABLE A.14. ICP Analyses of Feed Samples Taken During LFMM-86-5

Sample Number	1.00 (unadjusted)(b) #1		1.00 (adjusted)(c) #1A		9.30 #2A		Bulk Feed		Target	
	g/L		g/L		g/L		g/L		g/L	
	Oxides	wt%	Oxides	wt%	Oxides	wt%	Oxides	wt%	Oxides	wt%
Al ₂ O ₃	10.30	2.62	9.55	2.60	10.10	2.61	8.89	2.58	8.40	2.40
B ₂ O ₃	50.00	12.74	48.80	13.27	53.20	13.74	45.10	13.09	44.40	12.70
BaO	0.30	0.08	0.28	0.08	0.29	0.07	0.26	0.08	0.35	0.10
CaO	10.40	2.65	9.39	2.55	9.81	2.53	8.54	2.48	10.50	3.00
CeO ₂	7.04	1.79	6.60	1.79	6.76	1.75	6.03	1.75	0.60	0.17
Cr ₂ O ₃	2.74	0.70	2.51	0.68	2.65	0.68	2.33	0.68	2.38	0.68
Cs ₂ O	0.27	0.07	0.26	0.07	0.29	0.07	0.27	0.08	0.35	0.10
Fe ₂ O ₃	47.00	11.97	43.60	11.85	45.70	11.81	40.60	11.79	43.00	12.30
K ₂ O ^(a)	0.60	0.15	0.60	0.16	0.60	0.16	0.60	0.17	0.60	0.17
MgO	0.79	0.20	0.89	0.24	0.65	0.17	0.59	0.17	0.35	0.10
MnO ₂	11.30	2.88	10.40	2.83	10.80	2.79	9.64	2.80	1.72	0.49
Na ₂ O	61.70	15.72	54.80	14.90	57.30	14.80	51.20	14.86	52.80	15.10
NiO	1.77	0.45	1.62	0.44	1.75	0.45	1.54	0.45	1.58	0.45
P ₂ O ₅	10.40	2.65	9.78	2.66	10.00	2.58	8.63	2.51	9.10	2.60
RE ₂ O ₃	0.32	0.08	0.45	0.12	0.35	0.09	0.21	0.06	10.20	2.90
RuO ₂	0.40	0.10	0.44	0.12	0.00	0.00	0.28	0.08	0.28	0.08
SiO ₂	176.00	44.83	161.00	43.77	169.00	43.66	155.00	45.00	156.40	44.70
SO ₃ ^(a)	0.70	0.18	0.70	0.19	0.70	0.18	0.70	0.20	0.70	0.20
SrO	0.24	0.06	0.22	0.06	0.23	0.06	0.21	0.06	0.10	0.03
TiO ₂	0.15	0.04	0.16	0.04	0.16	0.04	0.13	0.04	0.10	0.03
Y ₂ O ₃ ^(a)	0.07	0.02	0.07	0.02	0.07	0.02	0.07	0.02	0.07	0.02
ZrO ₂	0.10	0.03	5.68	1.54	6.66	1.72	3.66	1.06	5.88	1.68
Total	392.59	100.00	367.80	100.00	386.07	100.00	344.48	100.00	349.86	100.00

(a) Not analyzed for, assumed target value.

(b) Feed low in ZrO₂.(c) Feed adjusted in ZrO₂.

TABLE A.15. Normalized Compositions of Glass Samples Taken During LFMM-86-5

Hours Into Run	Unadjusted ^(b)			Normalized Weight Percent							Bulk Feed	Target
Oxide	Starting Glass	#1	1.00 #1A	4.00 #2A	7.00 #3A	9.00 #4A	12.00 #5A	15.00 #6A	18.00 #7A			
Al ₂ O ₃	4.43	4.34	3.91	3.25	2.94	2.79	2.58	2.65	2.61	2.58	2.40	
B ₂ O ₃	7.01	7.17	8.52	10.16	11.27	12.22	12.59	12.39	12.15	13.09	12.70	
BaO	0.08	0.11	0.10	0.10	0.09	0.09	0.08	0.08	0.08	0.08	0.10	
CaO	1.55	1.46	1.68	2.06	2.26	2.33	2.36	2.39	2.42	2.48	3.00	
CeO ₂	0.00	0.00	0.36	1.02	1.31	1.46	1.57	1.67	1.66	1.75	0.17	
Cr ₂ O ₃	0.00	0.04	0.20	0.43	0.52	0.59	0.64	0.63	0.65	0.68	0.68	
Cs ₂ O	0.00	0.08	0.11	0.10	0.10	0.10	0.10	0.09	0.08	0.08	0.10	
CuO	0.00	0.00	0.00	0.04	0.00	0.00	0.00	0.00	0.00	0.00	0.00	
Fe ₂ O ₃	10.87	10.76	11.02	11.38	11.57	11.51	11.57	11.58	11.63	11.79	12.30	
K ₂ O ^(a)	0.00	0.17	0.17	0.17	0.17	0.17	0.17	0.17	0.18	0.17	0.17	
Li ₂ O	4.92	4.50	3.51	1.85	1.09	0.77	0.39	0.36	0.13	0.00	0.00	
MgO	0.85	0.70	0.59	0.37	0.34	0.23	0.18	0.31	0.18	0.17	0.10	
MnO ₂	2.79	2.68	2.68	2.71	2.72	2.76	2.74	2.72	2.73	2.80	0.49	
Na ₂ O	10.87	11.16	11.73	13.51	14.29	14.14	14.74	14.73	14.75	14.86	15.10	
NiO	0.86	0.95	1.01	0.76	0.66	0.62	0.59	0.59	0.53	0.45	0.45	
P ₂ O ₅	0.00	0.00	0.54	1.50	1.88	2.18	2.25	2.41	2.43	2.51	2.60	
RE ₂ O ₃	0.00	0.00	0.03	0.03	0.05	0.04	0.04	0.07	0.07	0.06	2.90	
RuO ₂	0.00	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08	
SiO ₂	55.05	54.81	52.87	49.38	47.19	46.35	45.45	45.19	45.59	45.00	44.70	
SO ₃ ^(a)	0.00	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.21	0.20	0.20	
SrO	0.00	0.05	0.06	0.08	0.08	0.08	0.07	0.07	0.06	0.06	0.03	
TiO ₂	0.06	0.05	0.05	0.05	0.05	0.04	0.04	0.05	0.04	0.04	0.03	
Y ₂ O ₃ ^(a)	0.00	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	
ZnO	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	
ZrO ₂	0.67	0.66	0.56	0.74	1.11	1.22	1.50	1.55	1.71	1.06	1.68	
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	
Fe ²⁺ /Fe ³⁺	0.03	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00			

(a) Not analyzed for, assumed target value.

(b) Feed low in ZrO₂, subsequent feed adjusted in ZrO₂.

TABLE A.16. ICP Analyses of Feed Samples Taken During LFMM-86-6

Hours Into Run	1.00		9.00					
Sample Number	1		2		Bulk Feed		Target	
Oxide	g/L Oxides	wt%	g/L Oxides	wt%	g/L Oxides	wt%	g/L Oxides	wt%
Al ₂ O ₃	8.25	2.58	9.67	2.85	7.91	2.49	8.40	2.40
B ₂ O ₃	42.50	13.31	35.40	10.45	41.90	13.18	44.40	12.69
BaO	0.24	0.08	0.27	0.08	0.23	0.07	0.35	0.10
CaO	8.40	2.63	9.02	2.66	7.74	2.44	10.50	3.00
CeO ₂	5.65	1.77	6.09	1.80	5.47	1.72	0.60	0.17
Cr ₂ O ₃	2.21	0.69	2.41	0.71	2.10	0.66	2.38	0.68
Cs ₂ O	0.24	0.08	0.29	0.09	0.23	0.07	0.35	0.10
Fe ₂ O ₃	38.40	12.03	41.40	12.22	36.50	11.48	43.00	12.29
K ₂ O(a)	0.60	0.19	0.60	0.18	0.60	0.19	0.60	0.17
MgO	0.59	0.18	0.89	0.26	0.59	0.19	0.35	0.10
MnO ₂	9.12	2.86	9.90	2.92	8.69	2.73	1.72	0.49
Na ₂ O	49.50	15.50	51.90	15.32	46.30	14.57	52.80	15.09
NiO	1.41	0.44	1.60	0.47	1.37	0.43	1.58	0.45
P ₂ O ₅	8.46	2.65	9.17	2.71	8.19	2.58	9.10	2.60
RE ₂ O ₃	0.24	0.08	0.56	0.17	0.22	0.07	10.20	2.92
RuO ₂	0.00	0.00	0.47	0.14	0.28	0.09	0.28	0.08
SiO ₂ (a)	142.00	44.48	150.00	44.27	141.00	44.37	156.40	44.70
SO ₃	0.70	0.22	0.70	0.21	0.70	0.22	0.70	0.20
SrO	0.19	0.06	0.22	0.06	0.19	0.06	0.10	0.03
TiO ₂	0.14	0.04	0.17	0.05	0.13	0.04	0.10	0.03
Y ₂ O ₃ (a)	0.07	0.02	0.07	0.02	0.07	0.02	0.07	0.02
ZrO ₂	0.37	0.12	8.05	2.38	7.40	2.33	5.88	1.68
Total	319.28	100.00	338.85	100.00	317.81	100.00	349.86	100.00

(a) Not analyzed for, assumed target value.

TABLE A.17. Normalized Compositions of Glass Samples Taken During LFMM-86-6

Oxide	Hours Into Run	1.00	4.00	7.00	9.00	12.00	15.00	18.00	Bulk Feed	Target
	Starting Glass	#1	#2	#3	#4	#5	#6	#7		
Al ₂ O ₃	2.61	2.64	2.55	2.54	2.62	2.62	2.55	2.61	2.49	2.40
B ₂ O ₃	12.15	12.46	12.52	12.41	12.87	12.82	12.76	12.88	13.18	12.70
BaO	0.08	0.08	0.07	0.08	0.07	0.07	0.07	0.07	0.07	0.10
BaO	2.42	2.49	2.42	2.54	2.48	2.42	2.41	2.44	2.44	3.00
CeO ₂	1.66	1.69	1.69	1.69	1.74	1.76	1.75	1.78	1.72	0.17
Cr ₂ O ₃	0.65	0.65	0.65	0.69	0.72	0.67	0.65	0.64	0.66	0.68
Cs ₂ O	0.08	0.08	0.08	0.08	0.08	0.07	0.07	0.07	0.07	0.10
CuO	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Fe ₂ O ₃	11.63	11.53	11.71	11.51	11.25	11.39	11.50	11.47	11.48	12.30
K ₂ O ^(a)	0.18	0.18	0.17	0.17	0.17	0.17	0.18	0.17	0.19	0.17
Li ₂ O	0.13	0.08	0.07	0.06	0.07	0.06	0.04	0.05	0.00	0.00
MgO	0.18	0.20	0.15	0.18	0.26	0.26	0.24	0.26	0.19	0.10
MnO ₂	2.73	2.70	2.76	2.77	2.74	2.77	2.74	2.77	2.73	0.49
Na ₂ O	14.75	14.75	14.35	14.72	14.39	14.34	14.24	14.09	14.57	15.10
NiO	0.53	0.56	0.56	0.57	0.60	0.62	0.57	0.52	0.43	0.45
P ₂ O ₅	2.43	2.47	2.49	2.55	2.61	2.61	2.54	2.67	2.58	2.60
RE ₂ O ₃	0.07	0.07	0.04	0.05	0.14	0.11	0.11	0.21	0.07	2.90
RuO ₂	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.09	0.08
SiO ₂	45.59	45.17	45.50	44.95	44.79	44.76	45.04	44.77	44.37	44.70
SO ₃ ^(a)	0.21	0.21	0.20	0.20	0.20	0.20	0.21	0.20	0.22	0.20
SrO	0.06	0.06	0.06	0.06	0.06	0.06	0.06	0.06	0.06	0.03
TiO ₂	0.04	0.05	0.04	0.05	0.05	0.05	0.05	0.04	0.04	0.03
Y ₂ O ₃ ^(a)	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02
ZnO	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
ZrO ₂	1.71	1.77	1.79	2.00	1.97	2.03	2.09	2.12	2.33	1.68
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Fe ²⁺ /Fe ³⁺	0.00	0.00	0.07	1.15	1.33	1.72	1.69	0.57		

(a) Not analyzed for, assumed target value.

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