Vitrification of Hanford Wastes in a Joule-Heated Ceramic Melter and Evaluation of Resultant Canisterized Product

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August 1979

**Prepared for the U.S. Department of Energy
under Contract EY-76-C-06-1830**

**Pacific Northwest Laboratory
Operated for the U.S. Department of Energy
by Battelle Memorial Institute**

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PACIFIC NORTHWEST LABORATORY
operated by
BATTELLE
for the
UNITED STATES DEPARTMENT OF ENERGY
Under Contract EY-76-C-06-1830

Printed in the United States of America
Available from
National Technical Information Service
United States Department of Commerce
5285 Port Royal Road
Springfield, Virginia 22151
Price: Printed Copy \$ ____*; Microfiche \$3.00

*Pages	NTIS Selling Price
001-025	\$4.00
026-050	\$4.50
051-075	\$5.25
076-100	\$6.00
101-125	\$6.50
126-150	\$7.25
151-175	\$8.00
176-200	\$9.00
201-225	\$9.25
226-250	\$9.50
251-275	\$10.75
276-300	\$11.00

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EXECUTIVE SUMMARY

DESCRIPTION OF WORK REPORTED

This report presents the results of a test run that produced glass from simulated radioactive wastes. The simulated wastes approximated wastes that would result from the radionuclide removal process. The wastes were vitrified in an electric melter, and the glass product was cast in metal canisters. These glass castings were subsequently evaluated to determine chemical composition, density, leachability, and surface area both for the "as-cast" condition and after a 7.6-m (25-f) drop onto a massive pad.

The objective of the week-long run was to demonstrate the technical feasibility of vitrifying Hanford wastes in a joule-heated ceramic melter and of producing a large prototypic casting in metal containers.

APPROACH USED

The technical feasibility of incorporating Hanford wastes into borosilicate glass was explored by converting 8180 kg of simulated waste and glass formers into glass in a joule-heated ceramic melter. The waste feed was made by mixing powdered chemicals into a chemical blend or batch^(a) whose composition was typical of that for a Hanford waste depleted of sodium salts. Glass produced was collected in four canisters that had 0.41-, 0.61-, or 0.91-m (16-, 24-, or 36-in.) diameters. The glass in the canisters was subjected to various tests to determine its characteristics.

CONCLUSIONS

Experience gained in the week-long vitrification test and characterization of the glass produced in the run support the following conclusions:

(a) "Batch" is the common word used in the glass industry to describe a chemical blend of oxides, salts and/or minerals. Batch should not be confused with an operational mode--i.e., batch vs. continuous.

- The Hanford waste simulated in this test can be readily vitrified in a joule-heated ceramic melter. Physical properties of the molten glass were entirely compatible with melter operation.
- The average feed rate of 106 kg/h is high enough to make the ceramic melter a feasible piece of equipment for vitrifying Hanford wastes.
- The glass produced in this trial had good chemical durability:

$$6(10)^{-5} \text{ g/cm}^2\text{-d.}$$

- When one of the canisters was purposely dropped onto a steel pad, the damage was limited to deformation of the steel can in the impact area, cracking of a weld, and fracturing of glass in the immediate vicinity of the impact area. No glass was released from the canister as a result of the drop test.
- The results of this vitrification test support the technical feasibility of vitrifying Hanford wastes by means of a joule-heated ceramic melter.
- Surface area for large glass castings is equivalent to the mass median particle diameters between 4.27 cm (1.75 in.) and 8.91 cm (3.51 in.) even when allowed to cool rapidly by standing in ambient air.
- Large canisters (up to 0.91 m in dia) can be cast without large voids while standing in air if the fill rate is over 100 kg/h.

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INTRODUCTION

This report summarizes the results of a week-long vitrification test in which simulated Hanford waste was converted into glass. The Rockwell Hanford Company, which manages wastes stored at Hanford, is evaluating candidate methods for the long-term solidification of wastes generated by the defense program.

One step in the long-term storage of high-level radioactive waste is conversion of waste into a product that exhibits low release potential. Pacific Northwest Laboratory (PNL) tests have shown borosilicate glass to be a desirable waste form because of its low release potential in an aqueous environment and its demonstrated process feasibility for use with other radioactive wastes.

The waste feed used in the run reported here was formulated to produce a borosilicate glass and incorporate chemicals typical of those in waste prepared by a so-called "radionuclide removal process." A dry powder having the desired chemical composition was used as feed in the tests described here.

OBJECTIVES OF WASTE VITRIFICATION TEST

The overall objective of this test was to demonstrate the technical feasibility of producing a quality borosilicate glass from Hanford waste within a joule-heated ceramic melter. The following aspects of vitrification were important elements to be evaluated:

- production capacity
- use of dry batched feed (a chemical blend)
- size of receiving canisters and ease of filling
- quality of glass product.

DESCRIPTION OF VITRIFICATION EQUIPMENT

JOULE-HEATED CERAMIC MELTER

The primary piece of equipment used in the present test is the liquid-fed ceramic melter (LFCM). Even though this melter is designed for both liquid and calcine feeding, only simulated calcine was fed into the LFCM in this test. The melter is comprised of a ceramic box fitted with metal electrodes. It is pictured schematically in Figure 1.

The refractory in contact with molten glass and unmelted batch is a fused cast-chrome alumina refractory (Monofrax[®] K-3). The refractory is 15 cm thick. It has the required properties of corrosion resistance to molten waste glass and a low electrical conductivity. The primary refractory is backed up with 7.6 cm of Alfrax[®] 66 on the side walls and by 7.6 cm of Zirmul[®] on the floor. These secondary layers are required for thermal insulation because the Monofrax K-3 has relatively high thermal conductivity. The melting chamber is 1.2 m (48 in.) long x 0.86 m (34 in.) wide x 0.71 m (28 in.) deep, with a nominal glass depth of 0.48 m (19 in.).

The refractory melting chamber is supported by a water-cooled 304 stainless-steel box. This box serves as a structural support for the refractory bricks, as a gas-tight seal, and as a heat-transfer surface for controlling refractory temperature.

Two sets of metal electrodes are imbedded into the two ends of the melting chamber as illustrated in Figure 2. The lower electrodes are 0.1 m (4 in.) high by 0.86 m (34 in.) long. The upper electrodes are 0.2 m (8 in.) high by 0.86 m (34 in.) long. Two separate sets of electrodes are essential in controlling the temperature profile in the melter volume. The electrodes are made from a high-temperature nickel-chrome alloy, Inconel[®]-690.

[®]Monofrax is a registered trademark of the Carborundum Company.

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[®]Zirmul is a registered trademark of the Chas. Taylor Sons Co.

[®]Inconel is a registered trademark of the International Nickel Co.

FIGURE 1. LFCM Schematic

FIGURE 2. LFCM Electrodes

Metal sidewalls extend above the refractory to provide a head space for de-entrainment of dust from the off gas. The metal sidewalls extend 0.55 m (21.5 in.) above the brick. They are made from Inconel-601, and can be heated by electrical heaters. The steel cover plate contains a number of flanged nozzles for material input and output, for viewing, and for sensor insertion.

Molten glass drains from the melter by gravity. An overflow-type drain is provided on one side of the melting cavity. Glass rises through a tubular 7.6-cm-dia (3-in.-dia) hole in a refractory block, moves along a horizontal trough, and then flows over a drain spout into the receiving canister. The drain region of the melter is heated with silicon carbide bayonet heaters that maintain the temperature needed for the molten glass to flow along the trough and down the face of the overflow trough block. These are shown in Figure 3. The drain flow path is depicted schematically in Figure 1.

A tilting mechanism is used to control glass drainage. The entire melter is supported within a tilting cradle, as shown in Figure 4. When the screw jack is raised or lowered the melter tilts about a pivot point, allowing control of glass drainage. Holdup capacities of more than 100 kg may be drained with a 2° change in tilt angle.

An auxiliary bottom drain is also incorporated into the floor of the melter. This bottom drain uses a freeze valve to control the flow of glass. It is pictured in Figure 5.

The freeze valve is composed of an annulus 0.48 cm wide. The annulus is provided with cooling jackets on both the internal and external sides. External heaters are provided to initiate draining. When the unit is not in operation there is a frozen glass plug in the annulus that seals the outlet. When glass drainage is desired, the cooling fluid is turned off and heat is applied. This thaws the frozen glass plug and allows the glass to drain. Glass drainage rate can be controlled or stopped by introducing a cooling fluid (water, steam, air) into the cooling jackets. While this bottom drain was not actually operated during the run reported herein, it

FIGURE 3. LFCM Heating Systems

FIGURE 4. LFCM Tilting Assembly

FIGURE 5. Bottom Freeze Valve of the LFCM

is a necessary part of the melter. It would be used to completely remove glass from the melter for shutdown purposes.

Power to the electrodes is controlled by means of silicon-controlled rectifiers. The phase angle at which firing occurs, and therefore the power delivered to the electrodes, is controllable by several alternate means. The option is open to control by a load temperature, load current, load power, or by manual voltage settings. A block diagram of the electrode control system is shown in Figure 6. In the present run the current control option was employed.

The melter is installed in room EDL-102 of the 324 Building. An overall view of the system is pictured in Figure 7.

WASTE FEED SYSTEM

In the present run the feed was a dry powder batch. Fifty-five open-top barrels of dry batch were emptied into a bucket elevator, which carried the batch to a feed hopper located above the melter. Feed was metered continuously into the melter by means of a volumetric feeder. Inside the melter the batch was distributed over the melt surface using a rotating shaft. The general feed system arrangement is shown schematically in Figure 8, and the batch distributor is defined in Figures 9 and 10.

OFF-GAS SYSTEM

Nitrates and carbonates decomposed when the simulated waste feed was vitrified. These gases, mixed with any air that leaked into the system, were passed into an off-gas cleanup system that is designed to trap particles and nitrogen oxides. A particulate filter was installed near the melter to prevent solids deposition in the pipe that connected the melter to the off-gas system. Components of the gas-cleaning scheme are shown in Figure 11.

FIGURE 6. LFCM Electrode Power System

FIGURE 7. LFCM and System in Development Laboratory

FIGURE 8. LFCM Powder Feed System

FIGURE 9. Function and Location of Batch Distributor

FIGURE 10. Picture of Batch Distributor Assembly

FIGURE 11. Off-Gas System for the LFCM

PRODUCT RECEIVERS

The glass-product receivers were cylindrical steel tanks. They were made from schedule-10 carbon-steel pipe. The four canisters are described in Table 1, and are shown schematically in Figure 12.

TABLE 1. Description of Glass Canisters

Canister No.	Outside Diameter, m (in.)	Wall Thickness, cm	Nominal Height, m	Internal Volume, m ³
1	0.41 (16)	0.64	2.4	0.343
2	0.61 (24)	0.64	2.4	0.767
3	0.91 (36)	0.95	2.4	1.72
4	0.61 (24)	0.64	2.4	0.767

These canisters are of prototypic size for a waste vitrification plant. During the filling the canisters were cooled by natural convection in ambient air. No attempt was made to anneal the glass, so a degree of thermal glass fracturing was anticipated. In a commercial vitrification plant it is possible that the canister would be maintained in a heated or insulated jacket with cooling capabilities to control the cooling rate--thereby reducing the degree of fracturing. However, the filling of bare canisters resulted in a modest increase in total surface area, and glass breakup was not extreme.

FIGURE 12. Canisters Used in This Test

WASTE FEED PREPARATION

CHEMICAL COMPOSITION

Feed for the present run was formulated to simulate average Hanford defense wastes that have been modified by the radionuclide removal process, which removes most of the soluble salts. The feed contained the glass-formers SiO_2 and B_2O_3 , as well as the chemicals used to simulate the waste. The chemical formulation is give in Table 2.

TABLE 2. Composition of Waste Feed and Final Glass

<u>Component</u>	<u>Waste Oxides, wt%</u>	<u>Glass Formers, wt%</u>	<u>Glass as Oxides, wt%</u>	<u>Added As: (a)</u>	<u>Gas Evolved on Heating</u>
SiO_2		46.7	46.7		
B_2O_3		13.63	13.63		
Li_2O		3.05	3.05	Li_2CO_3	CO_2
Ti_2O	0.2	3.06	3.26		
Ce_2O	1.00		1.00		
P_2O_5	1.72		1.72	$\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$	
Bi_2O_5	0.91		0.91		
MnO	0.71		0.71		
Al_2O_3	7.02		7.02	$\text{Al}(\text{OH})_3$	H_2O
Fe_2O_3	4.27		4.27	$\text{Fe}(\text{OH})_3$	H_2O
NiO	0.62		0.62		
Cr_2O_3	0.41		0.41		
SrO	1.89		1.89	SrCO_3	CO_2
CaO	0.64		0.64	CaCO_3	CO_2
Na_2O	13.43		13.43	Na_2CO_3	CO_2
ZrO_2	1.03		1.03	ZrSiO_4	
	33.85	66.44	100.29		

(a) If not noted the substance was added as oxide.

PHYSICAL FORMULATION

The chemicals listed in Table 2 were nominally 200-mesh powders and were mixed together as a dry batch by the supplier. This material was reasonably free-flowing, and no notable problems were experienced in feeding it to the melter.

As noted in Table 2, hydrates and carbonates would decompose on heating. The weight loss on ignition was computed to be 21.5%.

PROCESSING EXPERIENCE

FEED PROCESSING RATE

An important design aspect of the ceramic melter is its throughput capacity. In the present run, the feed rate was gradually increased to determine the capacity of the melter.

The integrated mass of feed delivered to the melter is shown in Figure 13 as a function of time. As noted on the figure, four canisters were filled, and feed was terminated while the empty canister was being placed under the melter. The solid lines drawn through the data of Figure 13 connect the initial and final points, and therefore the slope of the four lines represents the average feed rates for the individual canisters.

FIGURE 13. Accumulated Weight of Material Feed to the LFCM

During the filling of canister No. 3, the batch distributor failed and had to be repaired. The feed was terminated for a time, and this shows a leveling of the data at about 44 hours. The average glass production rate for canister No. 3 was thus appreciably lower than it would have been had the repair job on the batch distributor not been needed.

As indicated by the progressively higher slopes of the lines on Figure 13, the production rate was increased as more operating experience was gained. Average feed rates for each of the four canister-filling periods are listed in Table 3.

The highest feed rate, 132 kg/h (290 lb/h), is 1.6 times higher than the value for the first canister-filling period. An appreciably higher feed rate of 173 kg/h (380 lb/h) was attained during the first 10-h period while filling canister No. 3. Even though this peak rate was not sustained (due to the failure of the feed distributor), observations made by the melter operators suggest that the 173-kg/h rate is below the capacity of the melter. Since 21.5% of the feed weight was lost upon ignition, the glass production capacity of the melter is higher than 136 kg/h (300 lb/h).

GLASS PRODUCTION AND DRAINING

Molten glass was drained from the overflow spout system pictured in Figure 1. Draining was done on a nearly continuous basis, with the flow rate being determined mainly by the glass melting rate. Glass flow was

TABLE 3. Feed and Production Rates for Canisters Filled with Glass

<u>Canister No.</u>	<u>Average Feed Rate, kg/h</u>	<u>Average Glass Production Rate, kg/h</u>
1	83.2	65.4
2	95.9	75.0
3	114.5	90.0
4	131.8	103.2

terminated between canisters by tilting the melter $\sim 2^\circ$ from horizontal using the tilt mechanism shown in Figure 2. When flow was initiated into a new canister, the glass flow rate was higher than the melting rate during the period when the melter was tilted back to the operating level.

The glass stream that was discharged from the drain spout could be viewed through an observation port. A picture of the luminous glass stream typically observed during this run is shown in Figure 14. The stream shown

FIGURE 14. Picture of the LFCM Draining Glass

is about 0.48 cm in diameter. Experience with the melter has allowed operators to judge glass production rate by the size of the drain stream. Also, small variations in pressure in the melter's gas space are reflected in changes in the size of the drain stream and its lateral position. The character of the drain stream has thus been found to be a meaningful qualitative indicator of melter operating condition.

Four canisters were filled with glass during the 85-h run. Weights and volumes of glass in the filled canisters are summarized in Table 4, and a photograph of the filled canisters is presented in Figure 15.

As indicated in Table 4, only canister No. 1 was filled to 2.74 m-- the nominal height of all four canisters. Also, the bulk glass density decreased from 2.7 g/cm³ to 2.4 g/cm³ during the run. As discussed later in this report, the reduction in density occurred because the glass produced from the defense waste feed had a lower density than did the glass that was present in the melter at the beginning of the run.

Information on the mixing within the molten glass in the melter was obtained from zinc analyses on samples taken from the drain stream. Because the feed used in the run contained no zinc, zinc content in produced glass originated from glass in the melter at the beginning of the run. Measured zinc content is shown in Figure 16 as a function of the volume of glass drained. Also shown are predicted zinc concentrations based upon a plug flow model and a perfectly mixed fluid model.

TABLE 4. Mass and Volume of Glass in Filled Canisters

Canister No.	Outside Diameter, m (in.)	Filled Height, m	Glass Volume, m ³	Net Glass Weight, kg	Bulk Density, g/cm ³
1	0.41 (16)	2.74	0.334	913	2.73
2	0.61 (24)	2.24	0.627	1630	2.60
3	0.91 (36)	1.70	1.08	2660	2.46
4	0.61 (24)	2.25	0.627	1520	2.43

Initially, the zinc concentration followed the plug flow prediction. Thus, the heavier glass initially present was displaced by freshly formed lighter glass, with little mixing. After 10 h, however, when about 50% of the original glass was displaced, the zinc concentration fell to the value predicted by the model based on perfect mixing. Hence, intermixing of glass occurred at about 10 h and the tank contents remained quite well mixed for the remainder of the run.

FIGURE 15. Canisters Filled With Simulated Waste Glass

FIGURE 16. Relative Zinc Concentration in Outflow Glass as a Function of Glass Volume Produced

OFF-GAS FILTER LOADING

As shown in Figure 11, off gas from the melter first passed through a high-efficiency particulate filter. This trapping scheme is not thought to be prototypic of a plant installation, but was useful in the present run to help determine the mass of solids entrained in off gas. Each filter was 30 cm x 30 cm x 15 cm deep and was rated for 3.5 m³/min. A bypass valving arrangement allowed the HEPA filter to be replaced during the run.

Four separate filters were used during the 85-h run. Based upon weight gained by the filters, approximately 1 kg of materials, including liquid water, was held by each filter at the time it was replaced. Thus, only 4 kg out of 9000 kg of feed were entrained in the off gas.

ELECTRICAL POWER CONSUMPTION

The constant-current setting was adjusted manually to maintain the highest glass temperature without exceeding an internal electrode temperature of 1050°C. As shown in Figure 2, the upper set of electrodes has

twice the exposed surface area, and is considered to be the primary set. The lower electrode set typically carried only about 20% of the total power.

Total power dissipated in the glass melt is shown as a function of time in Figure 17. As indicated, power was typically increased with time after an empty canister was installed under the melter drain. The large dip in power that occurred at about 43 h corresponds to the stoppage of feeding caused by batch-spreader failure.

Power level is expected to be closely related to the glass melting rate. This expectation is borne out if one compares the waste feed rate from Figure 13 to the power consumption data of Figure 17. When feed was terminated, power level had to be reduced to about 35 kW to maintain a steady temperature. The peak power duration experienced in filling the

FIGURE 17. Total Power Dissipated During Test

fourth canister (74 h to 84 h at 130 kW) led to a feed processing rate of 130 kg/h. Thus, as a first estimate, the electric melting process involves the dissipation of 1 kWh/kg of waste feed. This energy usage per unit mass would not be expected to vary greatly with melter size, and can be used to estimate the electrical requirements of larger melters.

The upper limit to allowable power dissipation is determined by current density limits on the electrodes and by the capability of the power supply system. While we have not yet established an upper power limit for the present melter, it may be conjectured to be as much as a factor of 1.5 or more higher than the peak value (140 kW) reached in the present run.

MELTER TEMPERATURES

Temperature is an important parameter in glass melter operation. Temperatures in the glass were recorded during this run. Thermocouples were placed at numerous locations in the refractory, on the side walls of the steel container, on cooling-water lines, within electrodes, and in the vapor space of the melter. Temperature within the glass melt was measured periodically by lowering an Inconel thermowell into the glass. Glass melt temperatures are inferred from the electrode temperatures measured during the major part of the run.

Temperature of the upper electrodes is plotted as a function of time in Figure 18. It may be seen that electrode temperature varied between 1000°C and 1100°C as feed rate and electric power levels changed. These electrode temperatures are within the desired range, and typify those that would be experienced in a full-scale melter.

Air space temperature, as shown in Figure 18, averaged approximately 300°C during the period when the glass surface was covered with feed. At the beginning and end of the run, when molten glass was exposed, temperature in the vapor space of the melter was about 800°C. Several maxima in temperature occurred during the run. These apparently correspond to periods during which feed was reduced, allowing the feed cap to become thin and preventing it from covering the entire surface of the molten glass.

FIGURE 18. Temperature of Air Space in Melting Chamber

After 20 h of continuous feeding a thermowell was inserted into the molten glass to permit the temperature profile to be measured. Results are shown in Figure 19. The peak glass temperature, 1290°C, was some 270°C higher than the electrode temperature of 1020°C.

The temperature profile measurements show the glass interface to be 38.1 cm above the melter floor. This is some 10.2 cm below the nominal glass level. Thus the unmelted batch decreases the glass depth in the central portion of the melter. The decreased glass depth in turn decreases the cross-sectional area available for electric current flow, and thereby increases the power dissipation per unit volume of glass. A high-temperature region consequently develops in the central portion of the melter. This is desirable because it causes higher reaction rates and more thorough reactions without exposing the sidewalls and electrodes to the highest temperatures.

FIGURE 19. Temperatures in the Molten Glass After 20 h of Operation

OBSERVATIONS ON COLD-CAP BEHAVIOR

Several observations that are important to melter operation were made regarding the behavior of the batch layer floating on top of the molten glass (the cold cap).

The cold cap forms a layer that floats on top of molten glass. The fact that the cold cap was free-floating was shown when glass outflow responded very rapidly to feed termination. If a solid bridge had existed, glass level would not have responded immediately to feed input. The free-floating cold cap is considered highly beneficial because it eliminates potential surges in production that could occur if a solid crust periodically fell into the melt. Also, if a solid bridge formed it would allow an insulating gas gap to form between molten glass and feed, thereby reducing the capacity of the melter.

Under normal operating conditions the cold cap contained vent holes that were effective in discharging the gases released by the decomposition of more refractory carbonates in the feed. When the natural vents were sealed by covering them with feed, the steady out-flow of gases was

terminated. The released gases were contained for a time, but eventually formed new vent paths. When the gases escaped, the cold cap and molten glass interface were disturbed, causing momentary surges in the off-gas flow rate. In the run completed, sealing of the gas vents could be prevented by simply raising the batch-distributor paddle.

The vast majority of gases created by decomposition of the solid batch were released before a viscous glassy phase was formed. This is important, because foaming problems have been observed for other feed compositions. Therefore, while the feed used in the present run was highly compatible with the melter operation, the physical formulation of chemically similar feeds can be important in terms of foam formation. Future tests should be done to explore this question.

WASTE GLASS CHARACTERISTICS

PHYSICAL APPEARANCE OF GLASS IN SECTIONED CANISTERS

The pouring of molten glass into unheated steel canisters, as shown in Figure 20, can be expected to produce a glass mass with numerous cracks, particularly near the glass-steel boundary. Three of the canisters were sectioned to permit visual observation of the cooled glass.

The glass was found to have formed the reasonably compact, massive form shown in Figure 21. The canister in Figure 21 had a 24-in. outside diameter and was 9.5 ft long. One-half of the cylindrical surface was removed by slitting the pipe wall with a rotary cutter. While the glass is obviously not a monolith, the volume of the canister is well filled by glass and the glass mass is solid enough to be resistant to dispersion. The top of the casting shown in Figure 22 is a location that is subject to severe thermal shock, yet fracturing is clearly not dramatic.

The outside surface of the glass had an oxidized appearance. This is most apparent on the surface of glass in the 16-in.-dia canister, which is shown in Figure 23. The outer greyish color is apparently iron oxide, formed when the canister was heated by hot glass. The glass was not tightly bonded to the steel wall in any of the three canisters that were split.

The small glass stream filled the canisters in a helical pattern. This is shown in the surface patterns evident in the sectioned 36-in.-dia canister in Figure 24. The fact that the layers are separated by a glass-glass interface is evidence that molten glass accumulated on top of previously solidified glass surfaces. This behavior is as expected for filling unheated canisters. As will be shown later, the helically shaped wedges are larger in size than are individual glass pieces removed by core drilling; therefore, the wedge-type structure has little impact on the average glass particle size in the filled canister.

FIGURE 20. Uninsulated Canister Ready for Filling Beneath the LFCM

FIGURE 21. Canister Number 2 (0.61 m in dia) With Sidewall Removed

FIGURE 22. Top of 0.61-m-dia Casting, Canister Number 2

FIGURE 23. Canister Number 1 (0.41 m in dia) With Sidewall Removed

FIGURE 24. Canister Number 3 (0.91 m in dia) With Sidewall Removed

PHYSICAL PROPERTIES OF GLASS

Particle Size From Canisters

The extent to which glass was cracked by thermal stresses was evaluated by removing glass chunks from the sectioned canisters pictured in Figures 21, 23 and 24. Glass was removed by means of a screw driver, with attention being paid to avoiding mechanical fracturing of the glass. Samples were taken from the outer layer (outer surface to 25.4 mm deep) and from a deeper layer (from 25.4 mm to 50.8 mm deep). The glass pieces recovered in this way were then hand-sorted to remove the large particles. Screenable particles were separated using a standard screen set with a one-hour shaking time.

Mass median particle diameters based upon the screening tests are listed in Table 5. Also shown are the mass percentages of glass which are sub-sieve--that is, the fraction which passes through a 325-mesh screen.

As shown by the data in Table 5, the mass median particle diameters vary from 42.7 mm (1.75 in.) to 89.1 mm (3.51 in.) and are thus sizeable chunks. Even though they are much smaller than the canister itself, the large particle sizes still act to reduce dispersion.

The sub-sieve fraction varies from 2.65×10^{-4} to less than 1×10^{-5} . While these are quite small fractions, the observed fraction that passed through the 325-mesh sieves is probably much higher than that which existed in the intact canister. The sieving process itself is thought to have produced a significant fraction of the fines through attrition. Thus, the indicated particle sizes are somewhat smaller than the in situ sizes.

The particle-size data were used to compute the geometric surface area. Based on an assumed spherical particle shape, the specific surface area was found to be $0.63 \text{ cm}^2/\text{g}$ for canister No. 1 and $0.91 \text{ cm}^2/\text{g}$ for canister No. 3. These specific surface areas are equivalent to spheres having diameters of about 3.2 cm (1.26 in.). Thus, thermal stresses have caused cracking of the glass into 3-cm particles. This size is smaller than could be achieved using an annealing process, but is large enough to assure low dispersion potential.

TABLE 5. Size of Glass Particles Removed From Sectioned Canisters

Canister No.	Sample Description		Mass Median Screen Size, mm	Percent Subsieve, 325-mesh
	Zone	Distance From Bottom		
1	(a)	0-0.3 m	42.7	0.0127
1	(b)	0-0.3 m	63.1	0.0073
1	(a)	1.22 m-1.52 m	50.1	0.00108
1	(b)	1.22 m-1.52 m	75.9	0.00362
1	(a)	at top	56.2	0.0265
1	(b)	at top	89.1	0.00197
2	(a)	at bottom	50.1	0.0104
2	(a)	at bottom	57.5	0.0214
2	(a)	at top	57.5	0.0134
3	(a)	at bottom	60.3	0.00386
3	(b)	at bottom	49.0	0.0063
3	(a)	0.89 m	63.1	0.00412
3	(b)	0.89 m	85.1	0.00178
3	(a)	at top	66.1	0.00115
3	(b)	at top	89.1	0.000811
4	(c)	(c)	3.72	1.32

-
- (a) From outer surface to 25.4 mm deep.
 (b) From 25.4 mm deep towards center.
 (c) Impacted area from drop test.

Canister No. 4 was subjected to a drop test to evaluate the impact resistance of both the steel can and the glass. This round-bottomed, 0.61-m (24-in.)-dia canister was dropped onto a steel slab from a height of 7.6 m, as shown in Figure 25. The canister was deformed, and glass in the immediate area of the impact was fractured.

Particle size in the most severely impacted region of the canister was found to be 3.7 mm in mass median diameter, as listed in Table 5. Thus, the surface area per gram was increased by more than an order of magnitude

FIGURE 25. Picture of Canister Just Before Impact from 7.6-m Drop

(to 21.3 cm²/g) by the impact. The fraction of the material that was subsieve increased about two orders of magnitude, to 1.3%. But even though the impact fractured a small volume of glass, the resulting particles are too large to be easily dispersed. Also, since the canister was not ruptured, the glass was totally retained within the canister.

The impact area on the canister is pictured in Figure 26. Cracks were formed in both the weldment and in the base metal, but the steel envelope did not open up.

Internal Pore Structure

All solid waste forms contain minute gaseous inclusions, which are termed pores. If the pores were voluminous they would degrade the waste properties by presenting additional surface area.

A number of glass samples from canisters No. 1 and No. 3 were subjected to pore analysis. Pore volumes, sizes, and number densities are summarized in Table 6 for canister No. 1 and in Table 7 for canister No. 3.

Pore volumes amount to 1% of glass volume at most. The average porosity for the 21 individual specimens is only 0.34%. The average surface-to-volume ratio for the 21 samples is 0.25/cm, which is comparable to the surface area of cracked glass. This internal surface area is very small, showing that the glass product is a low-porosity, desirable waste form.

Uniformity of Glass

Glass uniformity is important because it is an indicator of the degree to which waste components have been dissolved in the glass and of the subsequent separation of crystalline materials. A good-quality glass would be free from solid phases caused either by undissolved feed components or crystal formation during cooling.

Samples of glass from canisters No. 1, No. 2, and No. 3 were removed by core-drilling and analyzed in several ways to determine glass uniformity. A typical core sample is shown in Figure 27.

FIGURE 26. Impact Damage Area on Canister Dropped 7.6 m

TABLE 6. Summary of Porosity Characteristics of Simulated Waste Glass Specimens,
Test Series: RHO-2H-1(a)

Specimen No.	Total Pore Volume, %			Median Pore Diameter, μm				Pore Density, pores/cc		
	All Pores	Pores >20- μm dia	Pores >200- μm dia	All Pores	Pores >20- μm dia	Pores >200- μm dia	Pores >200- μm dia	All Pores	Pores >20- μm dia	Pores >200- μm dia
1	0.003	0.003	0.1	24.6	24.6	--	24.6	1.2×10^3	4.2×10^3	0
2	0.278	0.278	0.256	465	465	465	118	1.6×10^3	1.6×10^3	8.9×10^1
3	1.14	1.14	1.02	875	875	875	162	3.9×10^3	3.9×10^3	1.5×10^2
4	0.606	0.226	0.128	18.1	291	359	14.0	1.7×10^7	7.4×10^4	6.1×10^1
5	0.753	0.753	0.688	972	972	972	132	1.4×10^3	1.4×10^3	9.7×10^1
6	1.14	1.14	1.07	788	788	788	132	2.2×10^3	2.2×10^3	2.1×10^2
7	0.293	0.293	0.246	465	465	517	146	1.0×10^3	2.0×10^3	7.8×10^1
8	1.00	1.00	0.972	638	638	638	146	6.8×10^2	6.8×10^2	1.8×10^2
9	0.067	0.067	0.053	340	340	340	146	4.8×10^2	4.8×10^2	1.9×10^1
10	0.223	0.223	0.175	709	709	709	20.8	4.4×10^2	4.4×10^2	5.7×10^0

(a) Symbol for Canister 1 in major test run for Rockwell Hanford Operations.

**TABLE 7. Summary of Porosity Characteristics of Simulated Waste Glass Specimens,
Test Series: RHO-2H-3(a)**

Specimen No.	Total Pore Volume, %			Median Pore Diameter, μm				Pore Density, pores/cc		
	All Pores	Pores	Pores	All Pores	Pores	Pores	Pores	All Pores	Pores	Pores
		>20- μm dia	>200- μm dia		>20- μm dia	>200- μm dia	>200- μm dia		>20- μm dia	>200- μm dia
1	0.003	0.003	0	51.0	51.0	--	51.0	1.5×10^3	1.5×10^3	0
2	0.013	0.013	0.009	201	201	201	41.3	2.8×10^3	2.8×10^3	1.8×10^1
3	0.811	0.811	0.638	1334	1334	1334	20.8	4.7×10^2	4.7×10^2	3.1×10^0
4	0.0	--	--	--	--	--	--	--	--	--
5	0.0	--	--	--	--	--	--	--	--	--
6	0.222	0.222	0.194	377	377	419	162	5.2×10^2	5.2×10^2	1.1×10^2
7	0.030	0.030	0.016	248	248	248	180	1.2×10^3	1.2×10^3	1.8×10^1
8	0.0	--	--	--	--	--	--	--	--	--
9	0.006	0.006	0	77.7	77.7	--	77.7	5.9×10^2	5.9×10^2	0
9B	0.142	0.142	0.121	517	517	517	132	1.2×10^3	1.2×10^3	3.8×10^1
9C	0.016	0.016	0.007	201	201	201	95.8	1.9×10^3	1.9×10^3	1.5×10^1
9D	0.291	0.291	0.287	639	639	639	86.3	6.7×10^2	6.7×10^2	6.8×10^1
10	0.003	0.003	0	56.7	56.7	--	56.7	8.4×10^2	8.4×10^2	0
11	0.0	--	--	--	--	--	--	--	--	--

(a) Symbol for Canister 3 in major test run for Rockwell Hanford Operations.

FIGURE 27. Typical Core Sample from the Waste Canister

First, sample specimens were ground to a powder and subjected to an x-ray diffraction test. Results obtained for canister No. 3 are typical: of eleven different samples withdrawn at various canister locations, none contained detectable crystalline materials. Therefore, the glass is substantially free of crystals, showing that the glass formulation yields a crystal-free glass.

Second, the samples were analyzed with an x-ray fluorescence system. This analyzer identifies the amount of specific elements present. Results obtained for samples withdrawn from canister No. 3 are listed in Table 8.

Peak heights were normalized with respect to strontium at 68 units. The data show the glass to be quite uniform in chemical composition at the 12 different sampling locations. One exception is the zinc content. This element was not a component of the simulated waste feed for the present run, and originated from a zinc-containing glass present in the melter at the beginning of the run. Its concentration decreases with the height of

TABLE 8. Relative Peak Heights from X-Ray Fluorescence Analyzer Results for Samples from Canister No. 3

Sample No.	Sampling(a) Position, cm	Peak Height for Element Specified Using Ba Target												
		Si	Cl	K	Ca	Ti	Mn	Fe	Ni	Zn	Bi	Sr	Zr	Mo
1	3.8	4.3	2.5	Trace	4.3	9.8	1	17.2	3.6	5.5	8	68	49	Trace
2	14	4.0	2.3	Trace	4.8	9.2	1	16.1	3.5	5.2	7.5	68	49	Trace
3	30	5	2	1	5	9.6	1.5	17.2	4	5	7.5	68	47.5	Trace
4	44	3	3.6	Trace	4.2	9.6	1.2	16.8	3.6	9	7.2	68	48	Trace
5	61	4.8	3	---	4	11.5	3	19.4	4.2	2.4	9.7	68	53.8	---
6	76	4	2	---	3.5	9.5	1.5	17	3.5	2	8	68	46.5	---
7	92	4.2	2	---	3.6	9.9	1.5	15.8	3.7	2	8.4	68	47.2	---
8	107	4.5	2	---	3.5	10	1.5	16.5	3.5	2	7.5	68	45	---
8 Wall	107	4.5	2	---	3.5	10	1.5	17	3	1	7.5	68	44.5	---
9	122	3.1	3.1	---	3.6	10.3	1.5	16.5	3.1	1	8.2	68	45.8	---
10	137	3.7	2.1	---	3.2	9.5	1.5	15.8	3.7	1	7.4	68	46.7	---
11	151	4	2	---	3.5	10	1.5	17	3.5	1	8	68	47	---

(a) Positions stated are heights from bottom of canister in centimeters. Samples except "8 wall" were taken at a radial position about 0.64 cm from canister wall.

glass in the canister, as expected. Zinc is diluted as glass production proceeds, as reflected by height of glass in the canister.

X-ray fluorescence data for canister No. 1 are presented in Table 9. Data shown were obtained using a titanium target, which increases peak height for the elements of lower atomic weights.

The analyses of canister No. 1 glass show that most glass components are uniformly distributed throughout the canister. Exceptions are potassium and zinc, both of which were present in the initial melter inventory but were not present in feed. Hence, these two components decrease in concentration with glass throughput.

Similar results were obtained from analysis of samples removed from canister No. 2.

A third type of analysis was designed to show microscopic homogeneity. Samples of glass were polished, and the x-ray fluorescence analysis was performed at very small points on the polished glass surface. Results obtained for samples withdrawn from canister No. 3 are given in Table 10.

TABLE 9. X-Ray Fluorescence Analysis of Samples from Canister No. 1

Sample No.	Sample(a) Position, cm	Peak Height for Stated Element									
		Si	K	Ca	Fe	Ni	Zn	Sr	Zr	Mo	Bi
1	3.8	17.5	32	67.5	1.5	0.5	17.5	3	5.5	1	---
2	36	18	33	70	1.6	0.6	18	3	6	1	---
3	66	17.2	31	67	1.5	0.5	17	3	5.5	1	---
4	94	17.8	32	68	1.6	0.5	17.5	3	5.5	1	---
5	127	15	27.5	58.5	1.5	0.5	17.4	2.5	4.8	1	---
6	157	17.2	30.5	64.5	1.5	0.5	16.8	3	5.2	1	---
7	188	17.5	25	57	2	0.5	15.2	3.6	5.5	1	---
8	218	21.8	19.8	47	3	0.5	15.2	3.6	5.5	1	Trace
9	244	22.5	19	45.5	3	0.5	10.5	6	6	0.5	0.4
10	267	24	13.2	38	3.5	0.6	8.2	7	6	Trace	0.6

(a) Positions stated are heights from bottom of canister, measured in centimeters.

TABLE 10. Analysis of Polished Glass Specimens Obtained from Canister No. 3

Sample No.	Location, cm	Peak Height for Specified Element (Si=70)											
		Na	Al	Si	Bi	Ca	Ti	Ce	Mn	Fe	Ni	Zn	Sr
1	3.8	5.7	1.3	70	0.8	1.2	5.7	0.6	0.6	4.8	0.6	Trace	Trace
2	14	4.3	1.4	70	0.6	1.7	5.7	0.6	0.8	5.7	Trace	Trace	Trace
3	30	4.6	1.4	70	Trace	1.7	5.1	0.7	1.0	5.0	Trace	Trace	Trace
4	44	5.1	1.2	70	0.6	1.9	5.7	0.5	1.0	5.7	Trace	Trace	Trace
5	61	4.9	1.0	70	Trace	1.6	6.2	0.5	1.0	5.6	Trace	Trace	Trace
6	76	5.1	1.3	70	0.5	1.5	5.7	0.5	1.0	5.1	Trace	---	Trace
7	92	5.3	1.0	70	0.5	1.4	5.3	0.5	1.0	5.1	Trace	---	Trace
8	107	5.0	1.5	70	0.7	1.5	6.0	Trace	1.0	5.5	Trace	---	Trace
9A	122 ^(d)	5.4	1.3	70	0.5	2.0	6.2	0.5	1.0	5.1	Trace	---	Trace
9B	122 ^(d)	5.1	1.3	70	0.6	2.1	6.2	0.5	1.2	5.8	Trace	---	Trace
9C	122 ^(d)	5.6	1.3	70	0.6	1.4	5.6	0.5	1.1	4.8	Trace	---	Trace
9D	122 ^(d)	5.2	1.4	70	0.5	1.6	6.4	Trace	1.0	5.8	Trace	---	Trace
10	137	5.5	1.2	70	0.5	1.6	5.7	0.5	1.0	5.5	Trace	---	Trace
11	151	5.6	1.4	70	0.8	1.7	5.6	0.5	1.0	4.8	Trace	---	Trace

(a) Location stated is centimeters above bottom of canister.

(b) Si normalized to 70 unit.

(c) Note: Sr is near the excitation limit for 30 kV, so there is very poor excitation.

(d) Samples 9A, 9B, 9C and 9D were taken 0.64, 15, 28, 41 and 16 centimeters from the wall, respectively.

As shown by the relative peak heights in Table 10, glass composition is quite uniform throughout the canister. The four separate analyses performed on sample 9 show the minor variation found at different points on the same sample.

In summary, the analytical results show that the glass produced in this run was uniform in chemical composition, and no significant amount of crystalline phase was present.

Density of Glass Produced

Samples of glass removed by core drilling of the first three canisters were subjected to a density test employing a water pycnometer. As expected, glass density decreased from 3.0 g/cm³ to 2.65 g/cm³ as the initial glass inventory was displaced from the melter. Measured densities are shown in Figure 28 as a function of the volume of glass drained from the melter.

FIGURE 28. Glass Density Versus Glass Volume Produced

While most of the measurements indicate a uniform density of the glass product, some scattering of data occurred at glass volumes in the range of 0.3 m^3 . This variation from sample to sample is attributable to imperfect mixing of glass in the melter. After the heavier glass is flushed from the melter, the density and composition attained steady-state values.

Leach Rates of Glass Produced

Aliquots of the glass samples removed from the first three canisters were subjected to a 72-h Soxhlet-accelerated test to determine the leach rate. The leach rate is plotted as a function of glass throughput in Figure 29.

The leach rate increased from approximately $2 \times 10^{-5} \text{ g/cm}^2\text{-d}$ to up to $10 \times 10^{-5} \text{ g/cm}^2\text{-d}$ as the initial glass inventory was flushed

FIGURE 29. Glass Leachability Versus Glass Volume Produced

from the melter. The leach rate for the glass produced from the Hanford wastes is average in magnitude for borosilicate glass, and is many orders of magnitude better than the initial waste. Thus, while an even more leach-resistant waste glass could probably be developed, the glass product in the present run demonstrates that a good-quality glass can be produced by the electric melting process.

Temperature of Canisters

Temperatures on the surface of the canisters was monitored at various heights. Typical results are shown in Figure 30, and apply to a point 10 cm (4 in.) above the bottom of canister No. 4. Two temperature spikes occurred some 15 min apart. The first spike was apparently caused when a glass wedge approached the thermocouple site but did not quite reach it. Glass then distributed in a helical pattern, and at the next rotation covered the measurement point. A peak temperature of 410°C was observed for this location.

FIGURE 30. Canister Wall Temperature 10 cm from Bottom

Temperature histories experienced at heights of 61 cm (24 in.), 122 cm (48 in.), 183 cm (72 in.), and 244 cm (96 in.) are shown in Figure 31 through 34. The peaks come later, as expected, and the time slope is less steep. The maximum canister wall temperature was observed to be 460°C, and this occurred 122 cm from the bottom.

FIGURE 31. Canister Wall Temperature 61 cm from Bottom

FIGURE 32. Canister Wall Temperature 122 cm from Bottom

FIGURE 33. Canister Wall Temperature 183 cm from Bottom

FIGURE 34. Canister Wall Temperature 244 cm from Bottom

SUMMARY AND CONCLUSIONS

A four-day test was carried out to demonstrate the feasibility of converting Hanford defense waste into glass using a joule-heated ceramic melter. Some 8200 kg of waste formulated to simulate the waste that would result from the "radionuclide removal" process residue was converted to glass in the LFCM located in the 324 Building. Work completed supports the following conclusions and summary statements:

- The waste formulated to simulate the defense waste with added glass formers melted to produce a uniform glass melt. The properties of the waste and molten glass were entirely compatible with the operation of the joule-heated ceramic melter.
- Four carbon-steel canisters having outside diameters of 0.41 m, 0.61 m and 0.91 m were filled with molten glass. The canisters were exposed to laboratory air during the filling process, and as expected, the natural-convection heat-transfer cooling that occurred caused a degree of fracturing of the solidified glass. The surface-to-volume ratio of glass was equivalent to 3.2-cm-dia spheres, showing that a product of low dispersibility was produced even without annealing the glass product.
- The average feed rate for the fourth waste canister filling period was 132 kg/h (290 lb/h), which demonstrates the high production capability of the joule-heated melter. A peak feed rate of 173 kg/h (380 lb/h) was maintained for 10 hours. This peak rate, along with other observations, suggests the maximum processing rate for the system to be higher than 180 kg/h (400 lb/h).
- Solids entrained in the off gas amounted to 0.044% by weight compared to the feed mass. This low entrainment fraction suggests that an off-gas cleanup system of standard design would be sufficient to remove gas-borne contaminants.
- Electric power dissipation varied from 50 kW to 140 kW in the present run. Electric energy dissipated amounted to 1.0 kWh/kg of batch.
- The success of the present run demonstrates the technical feasibility of vitrifying defense wastes in a joule-heated ceramic melter.

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