
Low Leach Rate Glasses for Immobilization of Nuclear Wastes

L. A. Chick
C. Q. Buckwalter

October 1980

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Richland, Washington 99352

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SUMMARY

Improved defense and commercial waste glasses are shown to have about one order of magnitude lower leach rates at 90°C in static deionized water than corresponding reference defense and commercial waste glasses. This durability difference diminishes as the leaching temperature is raised; at 200°C there is no significant difference in leach rates. However, at repository temperatures less than 150°C, the improved compositions would have considerable advantages over reference glasses, based on our tests in deionized water. The formulation changes that cause increased durability are not yet fully understood but may involve increased SiO₂ as well as the presence of Al₂O₃ and Fe₂O₃ and their proportion with respect to the alkali oxides.

Melting temperatures of some of the high-durability glasses (1350-1450°C) are higher than those for borosilicate glasses currently being investigated for process application (1050°C). However, there is at least one high-durability glass that is compatible with extended use in existing equipment; others could be processed in the higher temperature equipment commonly in use in the commercial glass industry.

As expected, at the melting temperatures necessary for most of the high-durability glasses, volatility was found to be higher than that experienced in processing current reference glasses. Higher volatilities might be compensated for by specific design of the off-gas system for improved off-gas treatment and volatile materials recovery.

It is concluded that certain waste glass formulations are significantly more durable than current reference waste glasses at temperatures of less than 150°C in deionized water and that, although processing of these glasses would require some changes in current nuclear waste glass processing equipment, the formulations deserve further investigation because they provide viable improvements over current reference waste glasses.



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INTRODUCTION

Radioactive waste incorporated in glass is one of the major waste form options under investigation for immobilization of nuclear waste. Other alternatives include alumina-, silica- and titanate-based crystalline assemblages cement, and multibarrier waste forms. Perhaps the most important property of a nuclear waste form is its chemical durability since leaching and migration of radionuclides into repository ground waters and subsequently into the biosphere is the most credible transport scenario. This priority is indicated by proposed Nuclear Regulatory Commission (NRC) regulations for nuclear waste forms,⁽¹⁾ which place primary emphasis on control of the leach rate once the waste form reaches the repository.

Investigations of the chemical durability of a wide range of waste glass compositions have been progressing at Pacific Northwest Laboratory (PNL)^(a) under sponsorship of the U.S. Department of Energy (DOE). The purpose of this report is to present data demonstrating that waste glasses can be formulated that are more durable than well-known reference waste glasses. Over 100 compositions have been investigated leading to an improved class of glasses. Data are presented on nine representative compositions in this report, including leaching results, viscosity, and volatility.

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EXPERIMENTAL

SAMPLE PREPARATION

The glass compositions included in this report are shown in Table 1. Glass 79-339 is a simplified base-glass composition that was found to have excellent chemical durability. Glass TDS+79-339 contains 28 wt% of a simulated Savannah River Laboratory (SRL) defense waste (TDS^(a) waste) that was generated by the Purex process; it is similar to glass 79-339. Glass TDS/211 is an SRL reference glass containing 28 wt% TDS waste. The RHO Purex glass was developed by Rockwell Hanford Operations (RHO) and contains 30 wt% simulated Purex reprocessing Hanford defense waste. RHO also developed the RHO Residual Liquor glass, which contains 24 wt% of a potential sodium- and aluminum-rich Hanford defense waste.

The other four glasses are commercial power reactor waste glasses. Glass 76-68, a widely reported reference waste glass, contains 33 wt% of a simulated power waste (PW-8a) rich in sodium and iron. The full composition of glass 76-68 is reported elsewhere.⁽²⁾ Glass 77-260 was formulated to contain 33 wt% of PW-7c--a power waste containing significant amounts of sodium, gadolinium, phosphorous, zirconium, and molybdenum. The full composition of 77-260 is also reported elsewhere.⁽³⁾ Glasses 79-417 and 79-418 were patterned after glass 79-339 (Table 1) to establish that this high-durability base glass could contain simulated power wastes. Glass 79-417 contains 34 wt% of simulated power waste PW-4b--a waste containing significant amounts of zirconium, molybdenum, ruthenium, cerium, and cesium. Glass 79-418 contains 10 wt% of PW-4b waste; the full composition of PW-4b is reported in Reference 2.

Samples were prepared by melting batches composed of oxides, nitrates, and carbonates in platinum crucibles in electric resistance-heated furnaces with air atmosphere. The melting temperatures used are listed in Table 1, and melting was carried out for 2 hours. Chemical durability tests utilized samples that were diamond-sawed from annealed bars poured from the melt into a steel bar mold. Volatility and viscosity tests were performed on remelted crushed glass.

(a) Technical Data Summary

TABLE 1. Compositions of Test Glasses (mole%), Melting Temperatures, and 28-Day Silicon Releases

Oxide	High-Durability	Defense Waste Glasses				Power Waste Glasses			
	Base Glass	TDS+	TDS/211	RHO	RHO	76-68(a)	77-260	79-417	79-418
	79-339	79-339	reference	Purex	Res. Liq.	reference	reference		
SiO ₂	66.0	63.2	49.8	57.4	67.0	51.7	49.8	61.4	63.7
B ₂ O ₃	10.5	10.0	7.7	9.8	9.2	10.6	10.7	10.5	10.5
Al ₂ O ₃	4.1	3.9	2.1	3.5	4.2	--	1.6	3.5	4.1
Na ₂ O	10.8	10.1	16.3	10.7	10.9	15.7	15.0	10.6	11.4
Li ₂ O	--	--	7.1	10.9	4.3	--	--	--	--
Cs ₂ O	--	0.2	0.2	--	0.1	0.3	0.2	0.5	0.2
K ₂ O	--	--	--	--	--	--	1.8	0.1	--
CaO	1.2	1.2	6.0	1.0	--	2.8	1.5	0.4	0.1
SrO	--	0.3	0.3	0.4	--	0.3	0.2	0.6	0.2
NiO	--	1.7	1.6	--	--	0.2	--	0.1	--
MnO ₂	1.1	3.1	2.9	0.9	--	--	0.1	0.1	--
TiO ₂	--	--	--	--	4.2	2.9	6.3	--	--
Fe ₂ O ₃	6.3	6.3	6.0	5.4	--	4.8	0.6	0.7	6.5
Cr ₂ O ₃	--	--	--	--	0.1	0.2	--	0.2	0.1
ZnO	--	--	--	--	--	4.8	--	0.2	0.1
CuO	--	--	--	--	--	--	3.2	--	--
Gd ₂ O ₃	--	--	--	--	--	--	2.4	--	--
ZrO ₂	--	--	--	--	--	1.1	1.1	2.2	0.6
MoO ₃	--	--	--	--	--	1.0	0.9	2.2	0.6
Minor(a) Components	--	--	--	--	--	3.6	4.6	6.7	1.9
Melt Temperature,(b) °C	1350	1350	1050	1100	1200	1150	1150	1350	1350
Silicon Released(c)	0.5	0.7	8.8	1.1	0.6	14.0	NA	1.1	1.6
Resulting pH(d)	4.2	4.2	7.7	4.8	3.8	8.1	NA	4.7	6.1

(a) Minor components each less than 1 mole%.

(b) Temperature at which leach test samples were melted.

(c) Silicon released (normalized g/m²) after 28 days, 90°C in deionized water leachant at SA/SV = 10 m⁻¹.

(d) Resulting pH measured in leachate after 28 days at 90°C at SA/SV = 10 m⁻¹. Initial pH of deionized water leachant was 5.7.

EXPERIMENTAL PROCEDURES

Chemical durability was measured by two static tests in deionized water-- one at 90°C and one at hydrothermal temperatures (100 to 200°C). In the 90°C test, blocks of glass were suspended in deionized water within sealed, polypropylene containers. The water volume was adjusted so that the geometric sample surface area to solution volume ratio (SA/SV) was 10 m⁻¹, and the containers were placed in a 90°C ±1°C controlled-temperature bath without agitation. At the specified time, the container was pulled from the bath, the sample was removed, and the pH of the leachate was measured. The leachate was then analyzed chemically by inductively coupled plasma atomic emission spectroscopy (ICP).

The hydrothermal leaching tests were conducted in TFE Teflon®-lined acid digestion bombs. Glass block samples were prepared as for the 90°C leach test and placed in the bombs with a measured amount of deionized water to maintain SA/SV = 6 m⁻¹. The bombs were then sealed and placed in temperature-controlled ovens for 9 days at 100, 150, or 200°C ±5°C. Water vapor pressures were 1 atm for the 100°C test and 4.7 and 15.3 atm for the 150 and 200°C tests, respectively. Upon removal from the ovens, the bombs were rapidly cooled by immersion in ~20°C water, the lids were removed, and the leachate was pipetted out for ICP analysis.

Leaching results are presented in a unit appropriate for comparison of the chemical durability of nuclear waste forms. The normalized grams per square meter unit is independent of waste form specimen size, shape, and composition.

$$\text{release of element X}_2 \text{ to solution (normalized g/m}^2\text{)} = \frac{\text{mass of element X in solution (g)}}{\text{weight fraction of element X in waste form} \left(\begin{array}{c} \text{unitless} \\ \text{normali-} \\ \text{zation} \\ \text{factor} \end{array} \right) \cdot \text{geometric sample surface area (m}^2\text{)}}$$

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Viscosity of the glass melts was tested using the rotating spindle method. The system was calibrated using National Bureau of Standards (NBS) standard glasses.

Volatility was determined by weight loss from 2-g glass samples in platinum crucibles suspended in a 2-in. inside diameter (ID) vertical tube furnace for 3 hours with approximately 1 liter/min dry air flowing upward. The surface area of the glass sample was 2 cm².

RESULTS AND DISCUSSION

CHEMICAL DURABILITY

Since the major network former in the glasses is silicon, data on silicon release during leaching provide a good indication of relative durability. Figure 1 shows cumulative silicon release for the static test at 90°C in deionized water. After 28 days, releases for the high-durability glasses fall in a band that is roughly one order of magnitude less than releases for the two reference glasses (76-68 and TDS/211). Average leach rates based on the cumulative silicon release between 14 and 28 days also show about one order of magnitude difference between reference and high-durability glasses (see Table 2).

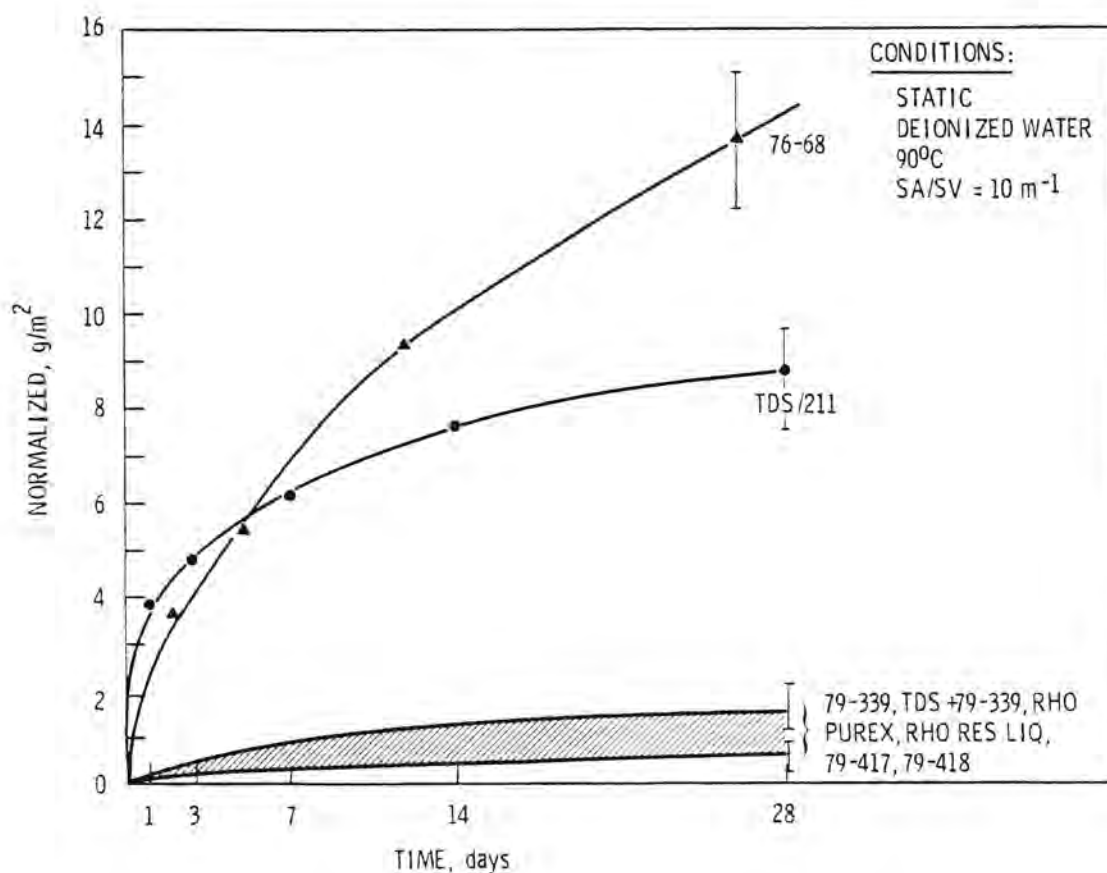


FIGURE 1. Cumulative Silicon Released Versus Time at 90°C

TABLE 2. Silicon and Sodium Release Rates

Glass	Silicon Release Rate, normalized $\text{g/m}^2\text{-d}$	Sodium Release Rate, normalized $\text{g/m}^2\text{-d}$
76-68	0.31	0.42
TDS/211	0.08	0.24
79-339 ^(a)	0.01	0.03
79-418 ^(a)	0.03	0.09

Conditions: Static
 Deionized water
 90°C
 SA/SV = 10 m^{-1}
 Avg rate from 14 to 28 days

(a) Data are shown for the most durable (79-339) and the least durable (79-418) of the six high-durability glasses reported.

Alkali release is usually faster than silicon release from glasses because the alkalis are less strongly bound in the glass structure than silicon. Alkali mobility is a consequence of ion exchange with positively charged hydrogen species from the water.⁽⁴⁾ Figure 2 shows sodium release versus time. Although these sodium release values are higher for all the glasses than the silica release values in Figure 1, the relationships between the glasses are very similar to those in Figure 1. For both the cumulative release after 28 days and for the 14-28 day release rates (Table 2) the high-durability glasses are about a factor of 10 better than the reference glasses.

Although the improved glasses are significantly more durable than the reference glasses at 90°C, the differences diminish as the leachate temperature is increased. Figure 3 is an Arrhenius plot of silicon release versus reciprocal temperature for the static hydrothermal test at 9 days. Data for two reference glasses--TDS/211 and 77-260--are plotted along with data for a supercalcine ceramic and two high-durability glasses--79-339 and TDS+79-339. Data on the supercalcine and on glass 77-260 were collected in an earlier study.⁽⁵⁾

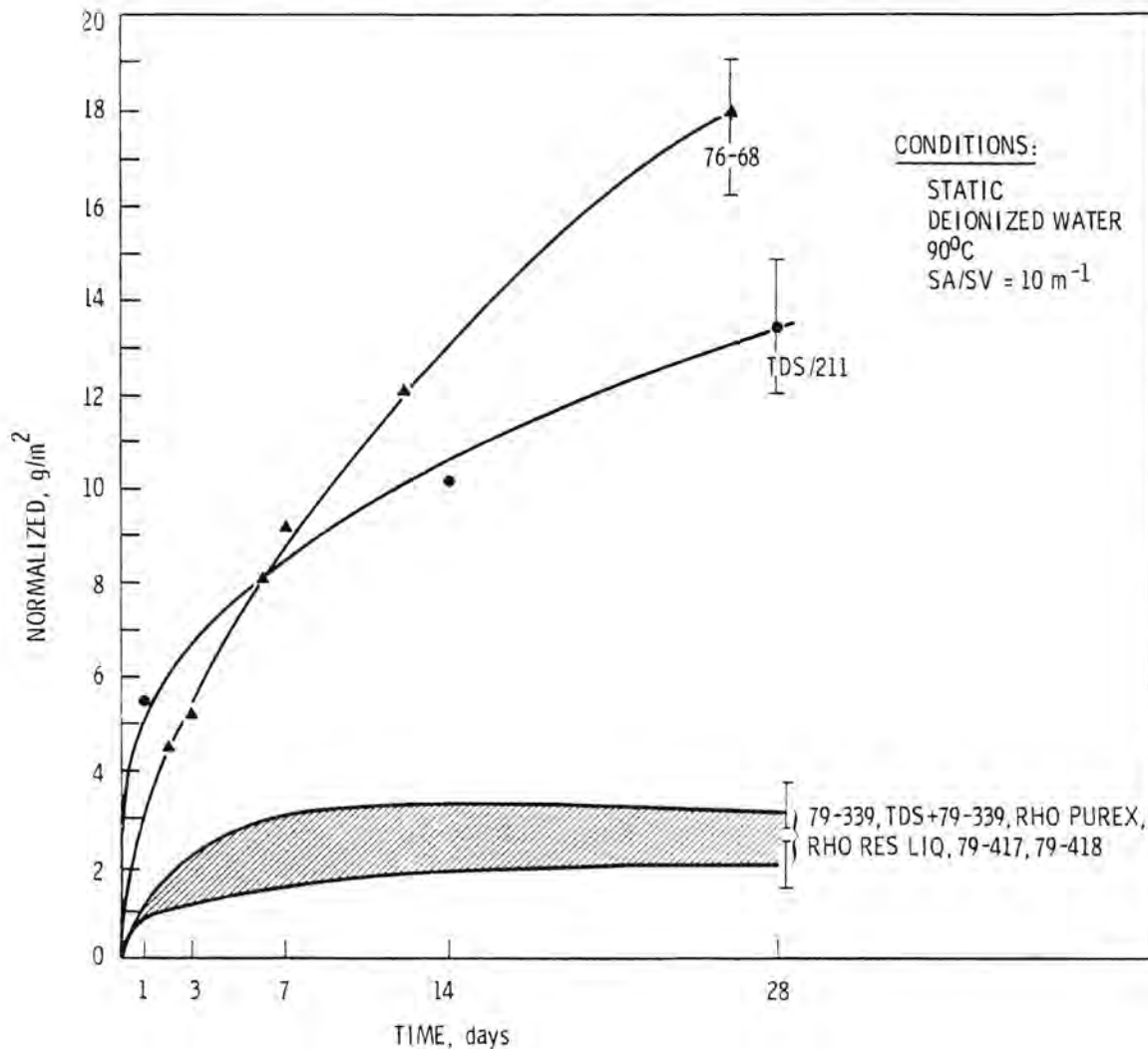


FIGURE 2. Cumulative Sodium Released Versus Time at 90°C

As can be seen from the curves for TDS/211 and TDS+79-339 (glasses containing similar amounts of the same simulated TDS waste) there is a significant difference in silicon release at 100°C; however, this difference diminishes to within experimental error at 200°C. (Curvature in the lines on Figure 3 is utilized for clarity and should not be considered significant in view of the magnitude of error.) The advantage of the high-durability glasses tested thus far is limited to temperatures below about 150°C. Figure 3 shows that the advantage increases greatly as the temperature declines.

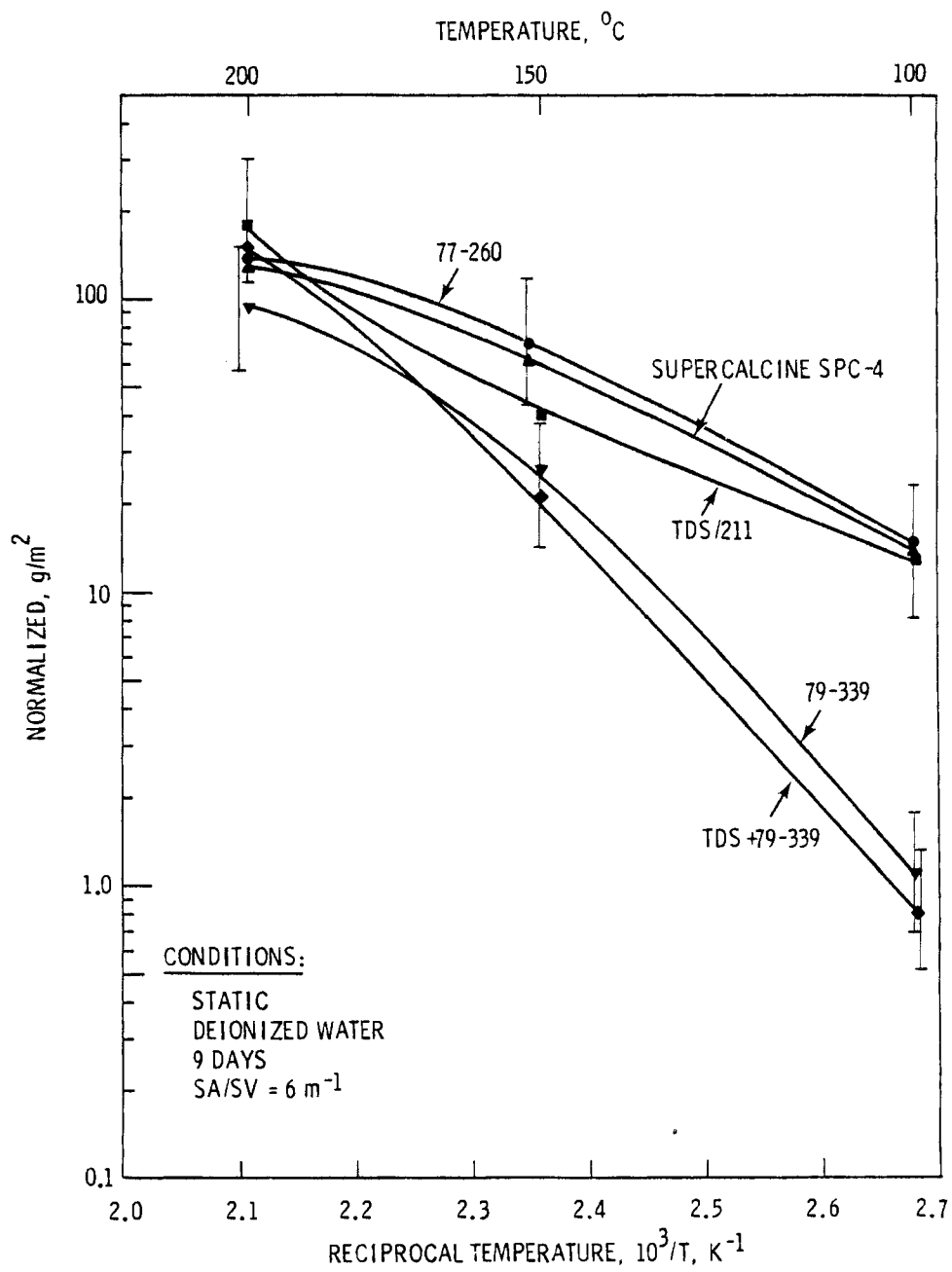


FIGURE 3. Nine-Day Cumulative Silicon Release Versus Temperature

Research is in progress to investigate leaching mechanisms and to determine how glass compositions affect these mechanisms to produce the range of chemical durabilities shown in Figures 1-3. The present studies of elemental release and resulting leachate pH tend to support the contention that there are two mechanisms of glass-water interaction: alkali ion exchange and hydroxyl attack of the matrix.^(4,6,7,8) Table 1 lists resulting leachate pH as well as silicon release after 28 days at 90°C. Notice that those glasses that caused the leachate to become basic (TDS/211 and 76-68) released the larger amounts of silicon. We believe that these glasses are less durable because they are more susceptible to ion exchange. Alkali ions exchange with positively charged water species such as H_3O^+ since isolated H^+ is probably rare in aqueous solutions. Removal of such species from the leachate causes the leachate pH to become basic. Electron sputtering for chemical analysis (ESCA) composition profiles of leached surfaces on those glasses that result in basic leachate pH show that alkali ions are depleted to greater depths than in those glasses that result in acid leachate pH. Transport of ions such as H_3O^+ into the glass could result in matrix attack by hydroxyl groups with a resultant enhanced gel layer formation and expanded lattice to enhance ionic mobilities. In this manner, susceptibility to ion exchange signified by basic leachate pH would result in faster dissolution of the glass and larger gel layer development.

The acidic leachate pHs observed with the more durable glasses may result from the presence of aluminum and iron and their proportions in relation to the total alkali content. Aluminum and iron readily react with OH^- from the leachant to form hydroxides that are soluble only at very acidic pHs. Such OH^- absorption accounts for the acidic leachates. Addition of aluminum has been found to decrease alkali ion mobility in alkali silicate glasses.⁽⁹⁾ Iron as well as aluminum may reduce alkali ion mobility in borosilicate glasses when they are introduced in proper proportion to alkali, thus decreasing the possibility for ion exchange. The result is a near-surface reaction with a thin gel layer due to the fact that ion exchange, which introduces water deeper into the glass, has been significantly inhibited. Thus, astute glass formulation protects the glass from ion exchange, which results in decreased dissolution. Apparently, the degree of protection from ion exchange is a function of

temperature so that by 200⁰C (and maintaining a liquid phase) the large differences in dissolution rates between glasses observed at lower temperatures are diminished (see Figure 3).

Composition differences resulting in increased durability of the present glasses over reference glasses are more subtle than is implied by Table 1. Certainly, increased SiO₂ content is important; notice in Table 1 that the high-durability glasses contain 6-18 mole% more SiO₂ than the reference glasses. Alkali content has some influence on durability, but there are exceptions. For example, the RHO Purex glass contains nearly as much alkaline oxides (21.6 mole%) as the TDS/211 glass (23.6 mole%); however, the former glass is a factor of eight more durable. Glass 79-339 contains half as much alkaline oxides (10.8 mole%) as the RHO Purex glass but is only a factor of two more durable. The beneficial effects on durability of Al₂O₃ and Fe₂O₃ and the detrimental effects of more than about 3 mole% CaO in these glasses are at least as important as the effects of total SiO₂ and alkali contents. Although more work is needed to explain the causes of the improvement in durability, we conclude that waste glasses can be formulated to contain both defense and commercial power wastes that are significantly more durable at likely repository conditions than the widely reported reference waste glasses.

VISCOSITY AND VOLATILITY

The procedure for choosing optimum waste forms for immobilization of nuclear waste must involve tradeoffs between product properties and process feasibility. Although process demonstration for crystalline alternative waste forms is in the early stages, the technology for glass waste form production in the United States has advanced to the point of full-scale nonradioactive testing as well as pilot-scale and some full-scale testing with fully radioactive glasses. The important processing properties are melting temperature, volatility, refractory corrosion, and, for the case of joule-heated melting, electrical conductivity. In this report, melting temperature and volatility of the glasses of interest will be addressed.

For joule-heated melting, the glass must be heated until its viscosity is reduced to about 100 poise so that it will drain from the melter into the

canister. Thus, the required melting temperature of waste glasses can be defined as the temperature at which the viscosity of the glass reaches 100 poise.⁽¹⁰⁾ Present state-of-the-art joule-heated melters for waste immobilization can be operated up to $\sim 1150^{\circ}\text{C}$; the commercial glass industry, however, operates similar melters to much higher temperatures ($\sim 1450^{\circ}\text{C}$).⁽¹⁰⁾ Thus, waste glasses whose viscosity reaches 100 poise at or below 1150°C can be melted in current waste melters whereas higher melting waste glasses would require further melter development, drawing on nonnuclear commercial glass practice.

Viscosity curves for five of the glasses listed in Table 1 are shown in Figure 4. It is apparent that the TDS+79-339 glass and the RHO Residual Liquor glass have melting temperatures well above the 1150°C limit for present waste glass joule-heated melters (~ 1450 and $\sim 1330^{\circ}\text{C}$, respectively). TDS/211 reaches 100 poise at $\sim 900^{\circ}\text{C}$, which is well below the 1150°C limit. Glass 76-68 and RHO Purex glass reach 100 poise near 1100°C ; therefore, both of these glasses can be melted using present equipment. The comparably shallow slope of the RHO Purex glass viscosity curve is interesting; this relative insensitivity to temperature change is not presently understood.

In general, it can be expected that higher melting glasses will be more chemically durable than lower melting glasses. This follows from thermodynamic considerations: that higher energy input will be required to dissolve the more stable glass structures that require higher energy for their formation. This rule of thumb is supported by data in Figure 5--a plot of melting temperature versus 28-day, 90°C dissolution for five glasses. As can be seen, the higher melting glasses generally lose the fewest elements. Two glasses--TDS/211 and RHO Purex--however, lie on the right side of the trend in Figure 4 and have better durability in relation to their melting temperature than do the other glasses.

Volatility of glass components during melting is of some concern to waste glass formulation since the off gas from waste glass melters must be trapped and purified or recycled. Figure 6 shows volatility curves for four glasses. A previous unpublished study showed that there is a negative correlation between viscosity and volatility; that is, for a given temperature,

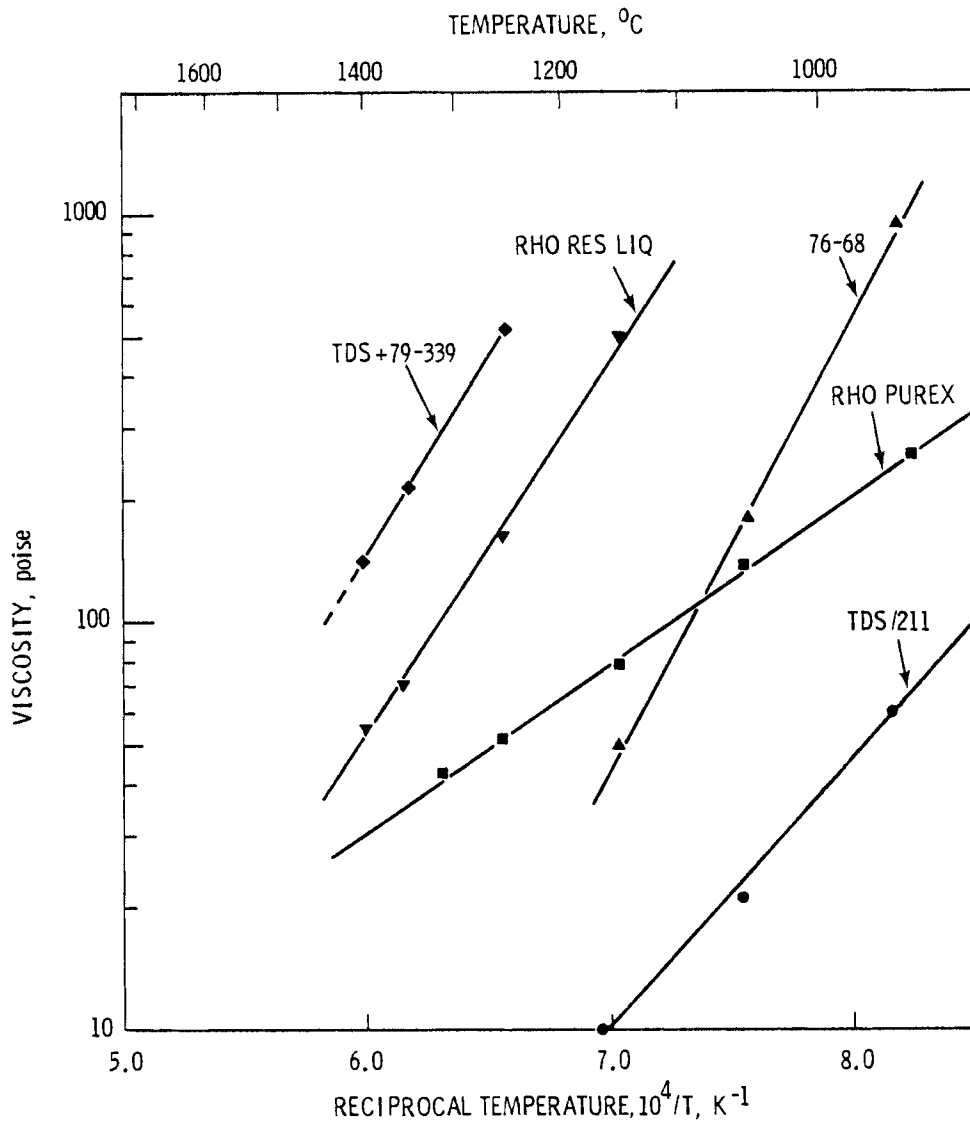
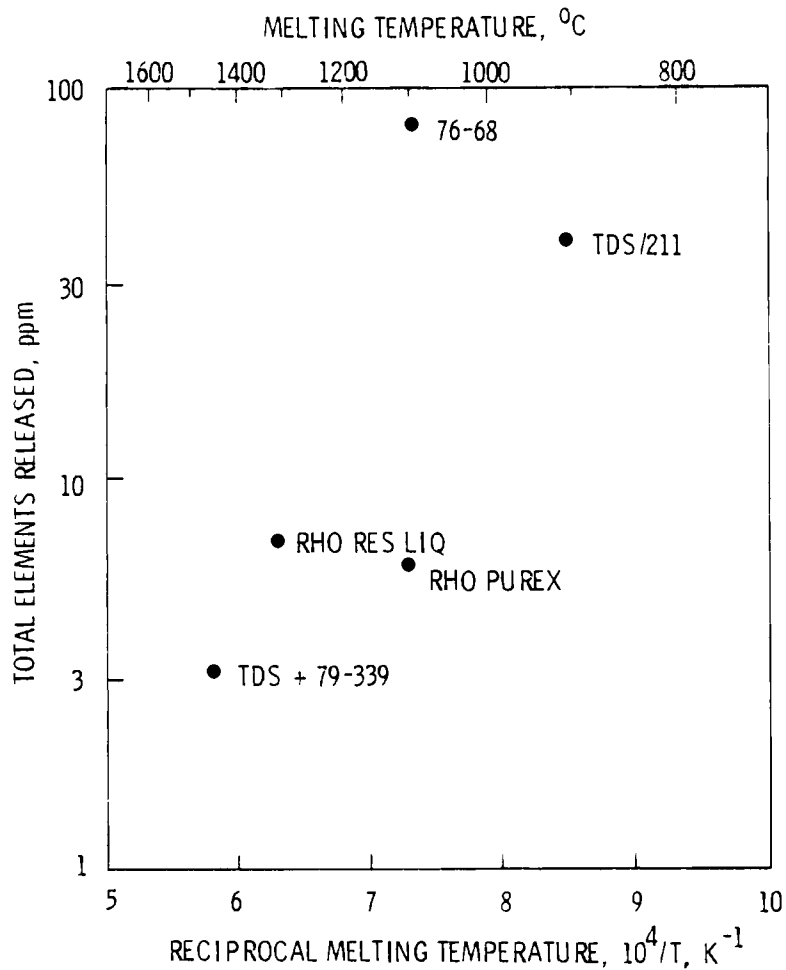


FIGURE 4. Viscosity Versus Temperature

high-viscosity glasses have low volatility. This is believed to occur because convection currents in the glass play an important role in replenishing the melt surface with volatile elements. High viscosity impedes convection and thus impedes volatility. However, the proper way to compare the volatility for engineering requirements is to examine volatility at the required melting temperatures. On that basis, the two higher melting glasses--79-339 and TDS+79-339--have much higher volatility than the RHO Purex and TDS/211. At 1450°C (the approximate melting temperature of glasses 79-339 and TDS+79-339) these



CONDITIONS:

LEACHING

STATIC
 DEIONIZED WATER
 28 DAYS
 90°C
 SA/SV = 10 m⁻¹

MELTING TEMPERATURE:

TEMPERATURE AT WHICH SHEAR
 VISCOSITY BY ROTATING
 SPINDLE METHOD EQUALS
 100 POISE

FIGURE 5. Total Leaching Release Versus Melting Temperature

glasses have volatilities near 100-mg loss. Data on RHO Purex glass must be extrapolated to estimate a volatility of ~1-mg loss at its 1100°C melting temperature. To estimate the volatility of TDS/211 at 900°C would require an even more drastic extrapolation. However, in practice electrical conductivity requirements result in TDS/211 being melted at about 1050°C where its

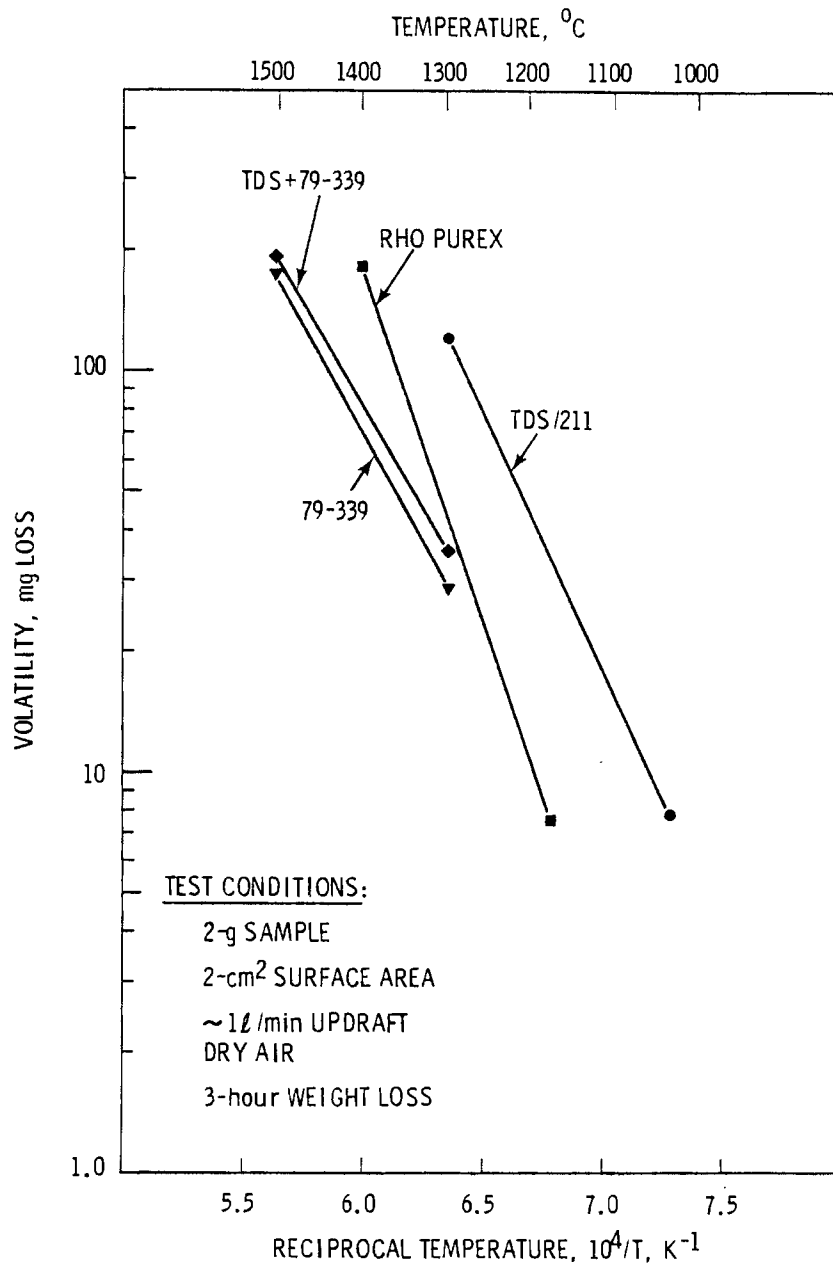


FIGURE 6. Volatility Versus Temperature

volatility is about 3-mg loss. It is obvious then that the higher melting glasses will volatilize at much higher rates than the current reference glass TDS/211 or the RHO Purex glass. However, increased volatility may be reasonably compensated for by keeping a thick "cold cap" of unmelted batch over the melt to act as a reflux or by increasing the capacity of the off-gas system.

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