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Development of Glass Formulations Containing High-Level Nuclear Wastes

by
Wayne A. Ross

February 1978

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SUMMARY

The effects of Na_2O, K_2O, B_2O_3, TiO_2, CaO, and ZnO contents were determined on the leach rate and homogeneity of a potential high-level waste glass. It was found that the two characteristics are in competition, with variations of CaO having the greatest effect and TiO_2 having the least effect.

Boron oxide content is important in control of Na_2MoO_4 formation and separation. Sodium molybdate formation can also be controlled by the use of reducing agents.

The waste glass discussed in this report can tolerate from 0-50% waste content with minor effects on leachability and viscosity.

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INTRODUCTION

The incorporation of high-level radioactive wastes into a glass structure or matrix has been long recognized as an attractive method for handling these wastes and preparing them for disposal. Many of the attractive features which make glass the current choice as a waste form were recently reviewed.(1) Work on glass formulations has been carried out both in this country(2-4) and abroad(5-7) for many years. During the early development phosphate glasses were utilized, however, most of the recent formulations have been based on borosilicate type compositions.

Since high level wastes vary widely in composition due to different process conditions and fuel types, various glass compositions are optimum for each waste and process. The results of this study apply to a high level waste (PW-4b) and are based on the properties discussed below.

IMPORTANT PROPERTIES

The ultimate objective of waste solidification is to prevent significant release of radiation and radioactive materials to the environment. The most likely method is through contact of the waste with water, with subsequent leaching of radioisotopes, and migration into the environment. The leachability of waste forms is therefore a major concern. Long-term stability of the glasses with regard to thermal and radiation effects is also necessary to maintain the desired low leachability of the waste form. These effects are discussed in more detail in a separate report.(8) To obtain a consistent product with the desired properties, homogeneity of the waste glasses is important, though the degree of homogeneity required is much less than that of commercial glasses.

The processing equipment and allowable process conditions must also be carefully considered in developing glass formulations. Two melting systems, the ceramic melter and the in-can melter have been developed, each with its own set of specific requirements.(9,10) The ceramic melter originally selected for this development requires that the melts be oxidized (i.e., no reducing agents can be used) and that maximum melting temperature be limited to 1150°C. At this temperature the viscosity of the glass must be below 200 poise to allow refining of the glass melt. At temperatures greater than 1150°C the volatility of Cs_2O and corrosion of the electrodes become a concern.

Since long periods of time are required to determine glass behavior under thermal and radiation conditions, the glass development has proceeded under the two-step plan shown in Figure 1. Development undergoes two iterative loops, beginning with numerous iterations around the inner loop, with optimization based on leachability, viscosity, and homogeneity. The optimal glass from this loop is then subjected to a much more detailed test plan.(11) This report will be confined to a specific set of experiments in the inner melt formulation loop.

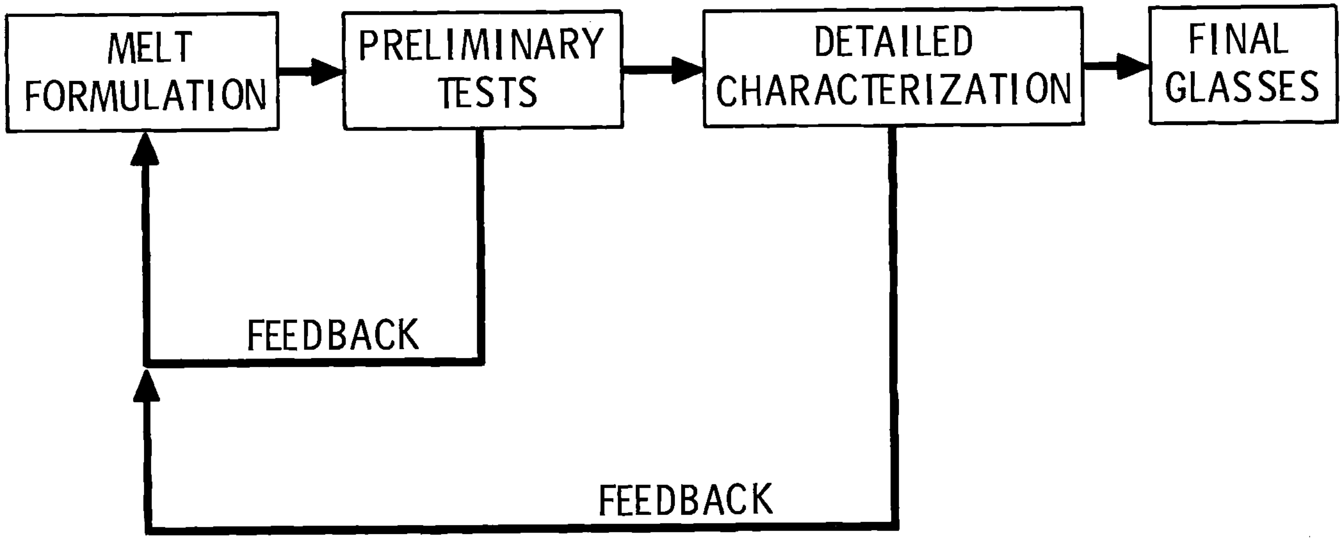


FIGURE 1. Task Relationships

WASTE COMPOSITION

As shown in Figure 2 high-level waste is composed of elements from three sources: fission products, actinides and process chemicals. Fission products are produced from burnup of the fuel in reactor and represent over 99% of the radioactivity in all nuclear wastes; actinides result from neutron capture and uranium and plutonium that have not been completely removed during solvent extraction in the reprocessing plant, (typically 0.1 to 1.0 wt%); and process chemicals are added during reprocessing. Examples are gadolinium for criticality control and addition of the intermediate level waste stream from cleanup of the solvent extraction solutions, which add sodium and phosphate. The concentrations and types of inerts vary with each reprocessor. The simulated composition used for our series of tests is called PW-4b-1 (shown in Table 1), a very clean waste with low inerts and low uranium and plutonium content. The PW-4b-1 composition contain chemical substitutions for radioisotopes in PW-4b. The referenced PW-4b composition and other simulations are detailed in Table 2 of Reference 8.

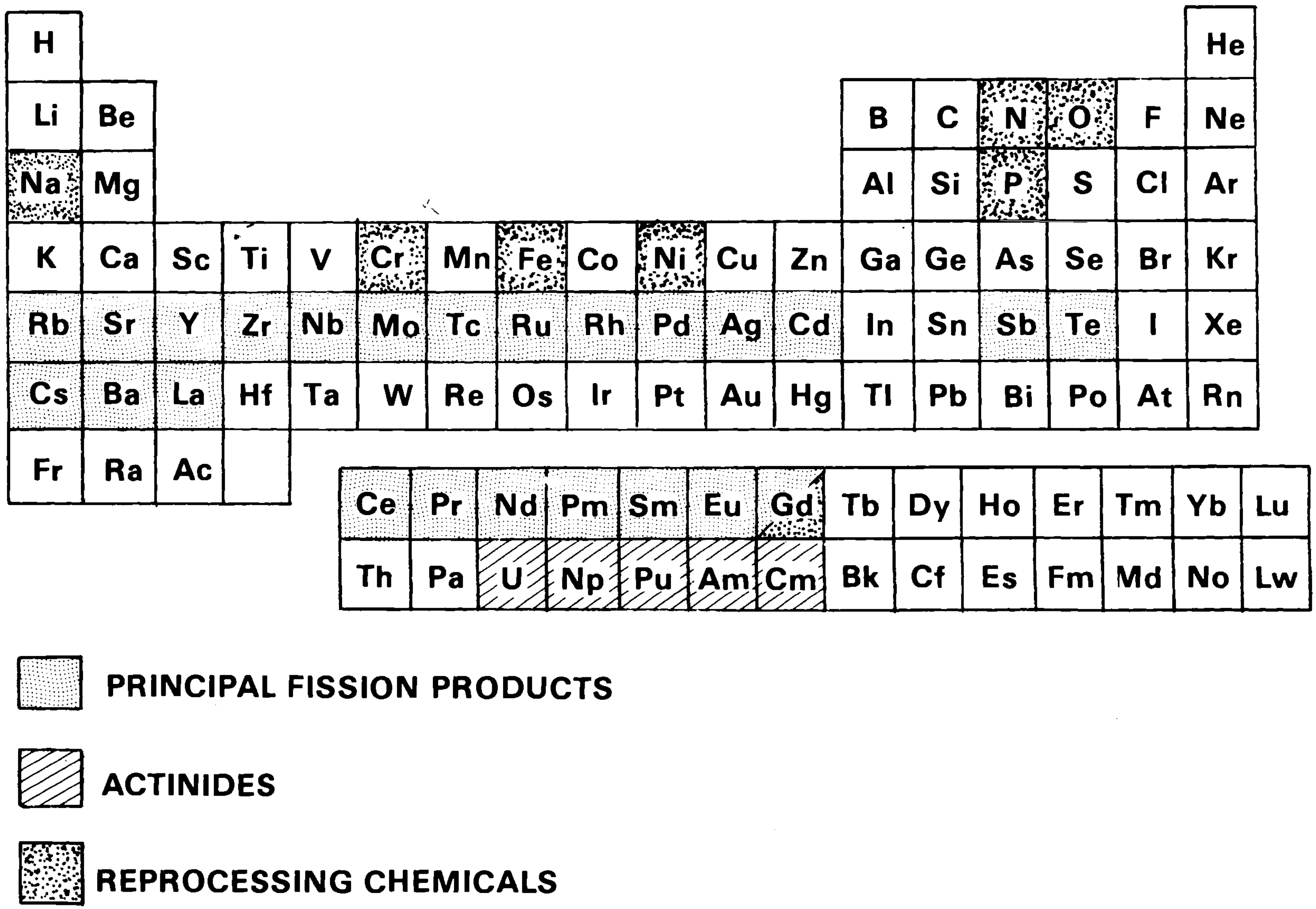


FIGURE 2. Elements in High-Level Waste

TABLE 1. Composition of PW-4b-1 Simulated High-Level Waste

Oxide	wt/o
Fe_2O_3	8.40
Cr_2O_3	0.88
NiO	2.68
P_2O_5	1.71
Rb_2O	0.90
SrO	2.70
Y_2O_3	0.07
ZrO_2	12.61
MoO_3	16.26
CoO	0.78
Ag_2O	0.22
CdO	0.25
TeO_2	1.85
Cs_2O	7.35
BaO	4.00
La_2O_3	8.20
CeO_2	16.39
Pr_6O_{11}	1.71
Nd_2O_3	5.81
Sm_2O_3	1.03
Eu_2O_3	0.27
Gd_2O_3	0.68
U_3O_8	5.30
	100.05

GLASS COMPOSITION

After a series of scouting tests, a tentative glass composition was chosen for the PW-4b-1 waste, the principal criteria were high chemical durability over the pH 4 to 9 range, oxidizing melting conditions, and maximum melting temperature of 1150°C. This composition (76-375) is shown in Table 2. Note that the glass is a borosilicate with quite high mixed alkali and minor additions of ZnO and TiO_2; the 1/3 waste addition adds considerable ZrO_2, rare earth (RE) oxides and Fe_2O_3, which generally increase glass durability. The effects of the high concentration of MoO_3 shown in the table will be discussed later. The waste added to a glass is chosen to minimize the total volume.

To verify that the composition for the glass frit was appropriate, a series of melt tests was performed to observe homogeneity and leach rates. For each test a component oxide concentration of the frit was varied by replacing or being replaced by an equal weight percent of silica. These samples were prepared as 40 gram melts by blending a simulated waste and the raw glass components with mortar and pestle and then heating at 1150°C for 2 hours in platinum crucibles. The glass was air quenched, then photographed and ground to -40 +60 mesh for a consecutive series of leach tests. The first leach test was performed in room-temperature NH_4OH-NH_4Cl solution buffered to pH 9, the second in room-temperature sodium acetate-acetic acid solution buffered to pH 3.9, and the third (Soxhlet test) used distilled water at 99°C. Each leach test was for a 24-hr period. Each sample was contained in a 200-mesh stainless steel envelope, with leach rates determined from weight loss. More details on leach testing are included in another publication.(8)

TABLE 2. Reference Glass Composition (76-375)

Glass Frit	Wt%
SiO_2	35
B_2O_3	13
CaO	2
ZnO	5
TiO_2	3
Na_2O	5
K_2O	4
Subtotal	67
Waste Additions (PW-4b-1)	33
Total	100

The results of tests on the effects of Na_2O content are shown in Figure 3, which also identifies the nominal composition of the standard 76-375 melt. Additions of Na_2O above that of the standard composition further increases the homogeneity of the glass and also increases leachability as measured by all three methods. It is also interesting to note that leach rates were actually decreased by the addition of Na_2O at low levels. As might be anticipated, alkali content must be maintained at moderately high levels to allow melting at the relatively low processing temperatures. Potassium oxide effects, shown in Figure 4, are similar to Na_2O; however, the changes seem to be more significant on a per mole basis. The changes shown are at 2 wt% intervals versus the previous 1 wt%, but on a mole basis, they are very similar. Potassium oxide is used both to take advantage of the "mixed alkali" effects on viscosity and also to retard liquid phase separation, common with borosilicates.

Boron oxide is utilized in waste glasses, both to lower viscosity and enhance homogeneity (Figure 5). The 7 wt% melt has several surface phases; one is a yellow phase (Figure 5) around the edge of the melt (sodium molybdate), which is particularly troublesome. It is undesirable both because it is water soluble and also because it contains significant amounts of cesium and strontium--the two most hazardous elements during the early life of the waste. The addition of increased amounts of B_2O_3 is one method of increasing the solubility of the molybdate in the glass. An additional method will be discussed later. The leachability decreases in the pH 9 solutions, with B_2O_3 increasing from 7 to 11%. This is due primarily to the

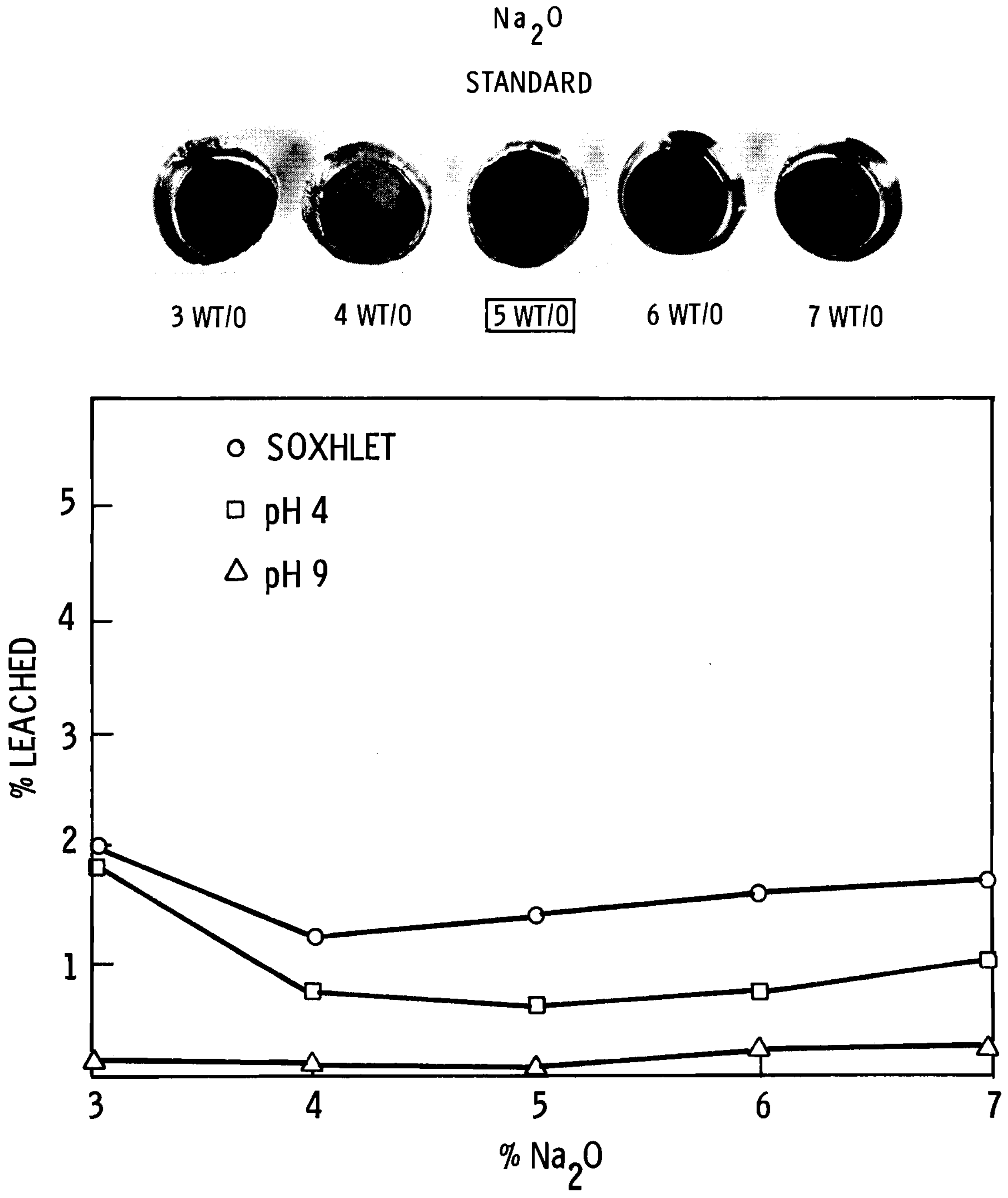


FIGURE 3. Leachability of 76-375 Type Glass with Change in Na_2O Content

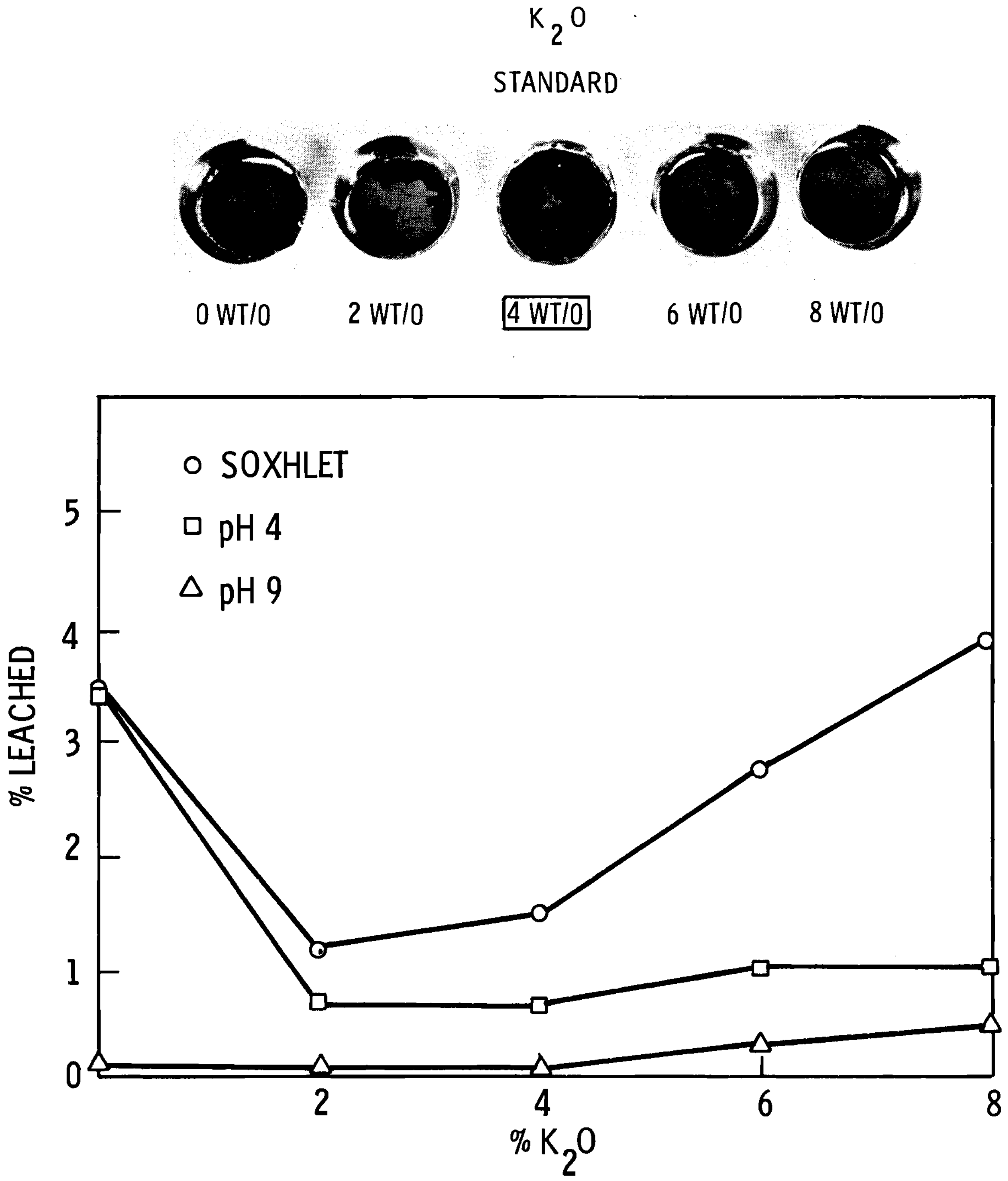


FIGURE 4. Leachability of 76-375 Type Glass with Change in K_2O Content

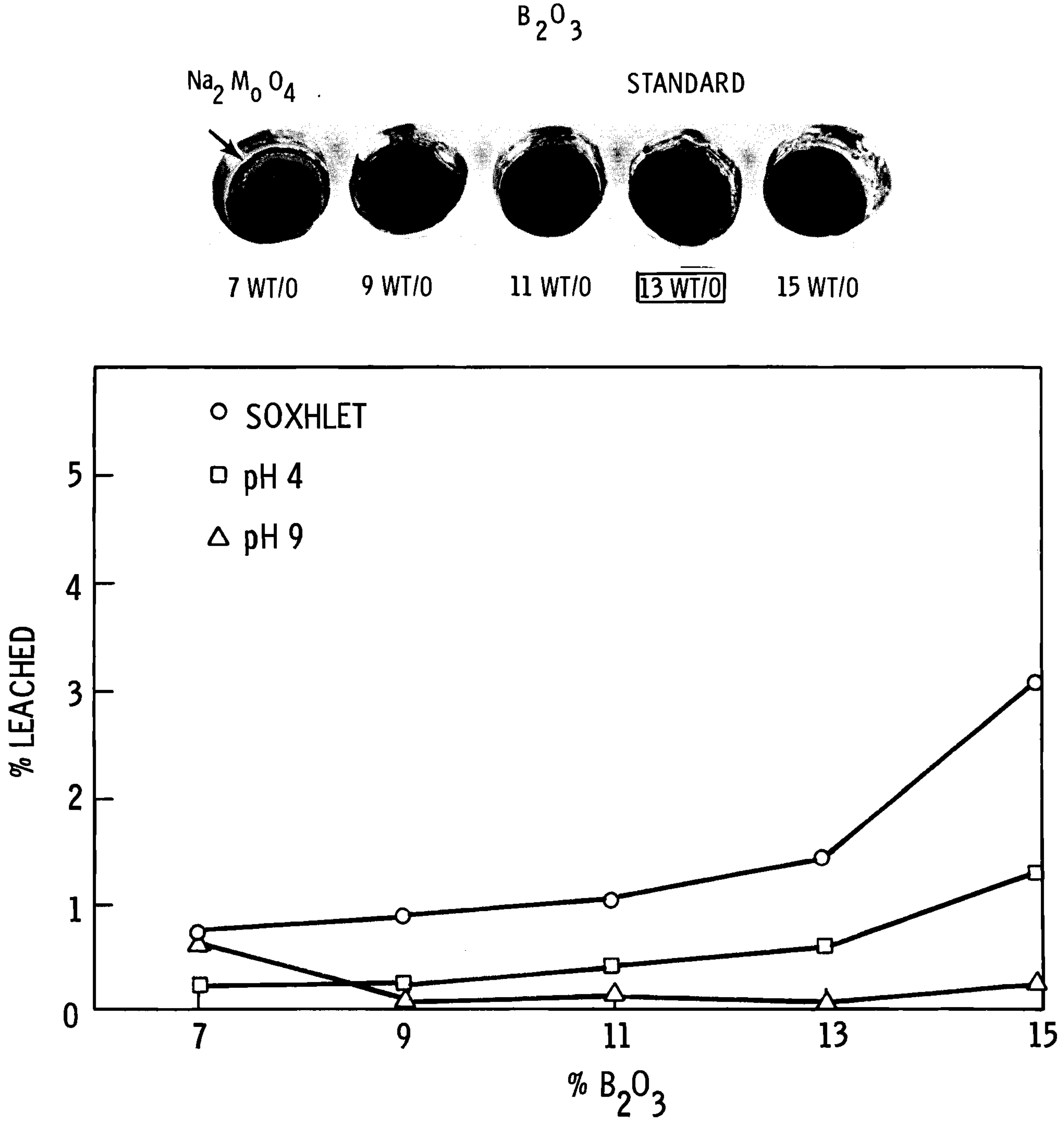


FIGURE 5. Leachability of 76-375 Type Glass with Change in B_2O_3 Content

elimination of the molybdate phase. Since B_2O_3 increases bulk leaching, slightly lower concentrations would be desirable if adequate homogeneity could be obtained.

Homogeneity and leachability comparisons of glasses containing TiO_2 are shown in Figure 6. Small additions of TiO_2 appear beneficial in lowering viscosity and also leachability. Large concentrations of titania can be expected to increase devitrification potentials and therefore need to be limited. The effects of TiO_2 on glass homogeneity appear to be minor. The standard was melted in a different batch, which may account for its slight decrease in homogeneity in comparison to the other samples.

Calcium oxide has major effects in waste glasses, as can be seen in Figure 7. This is a classic case of trade offs between homogeneity (which is greatly improved by calcia additions) and leachability, which also greatly increases with calcia. The acid durability is particularly affected, with a very rapid increase beyond 4 wt% CaO. The 2% CaO used represents a balance between the two conflicting requirements.

Zinc oxide has been used quite extensively in waste glasses, with some previous compositions containing as much as 21 wt% ZnO. Much smaller amounts are now used because of the rapid increase in acid attack with increasing ZnO content (indicated in Figure 8). Leachability (as measured by the

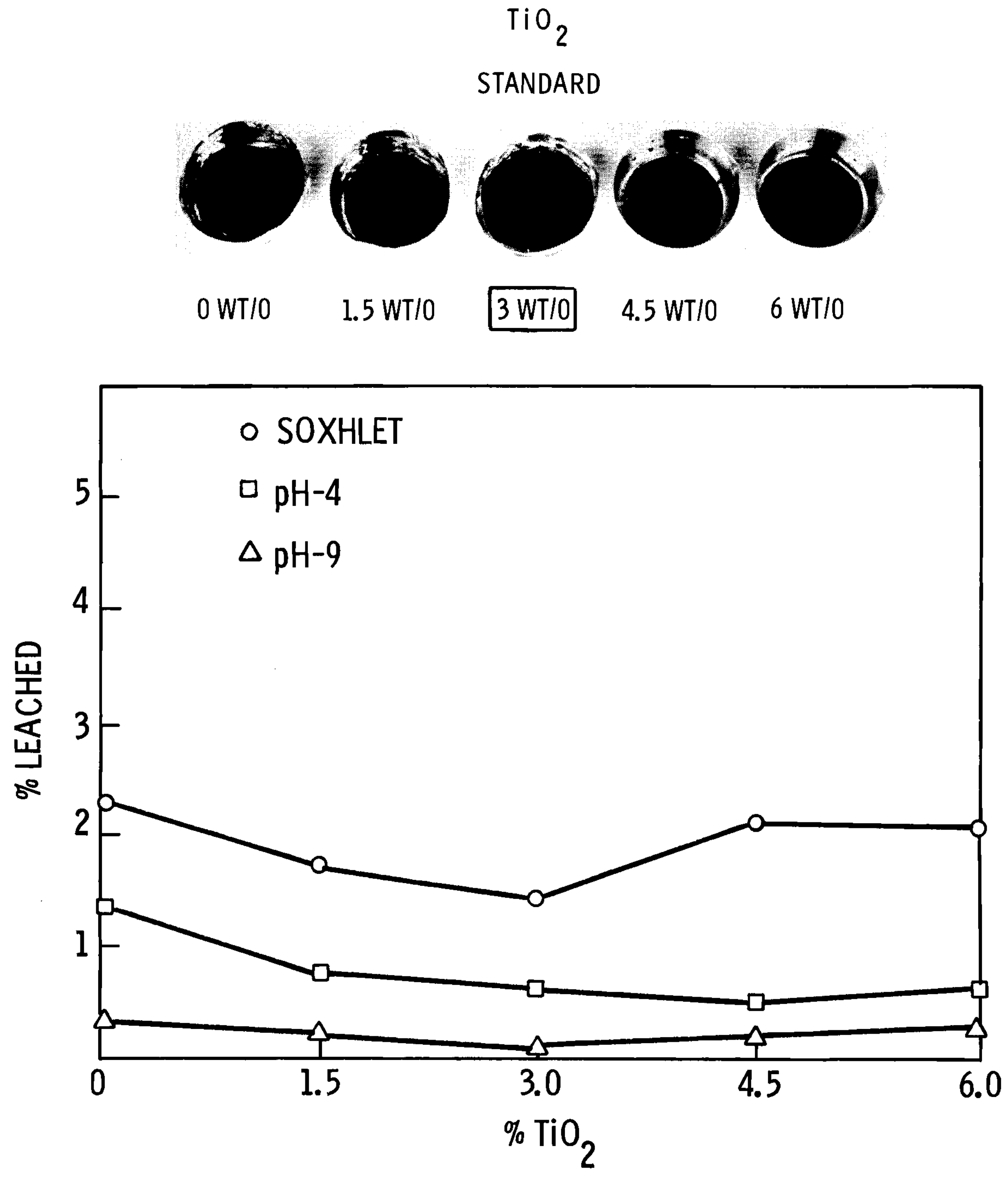


FIGURE 6. Leachability of 76-375 Type Glass with Change in TiO_2 Content

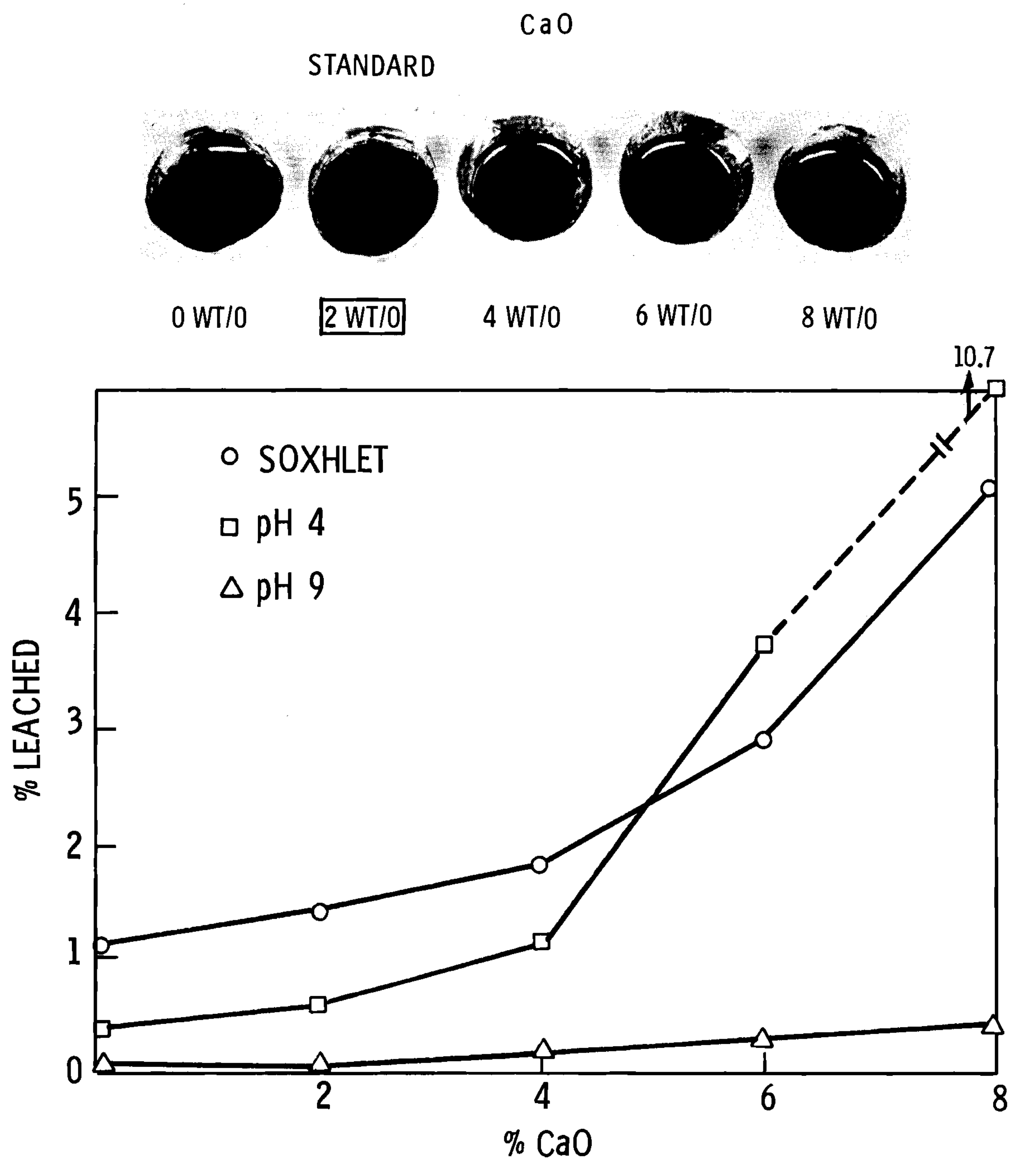


FIGURE 7. Leachability of 76-375 Type Glass with Change in CaO Content

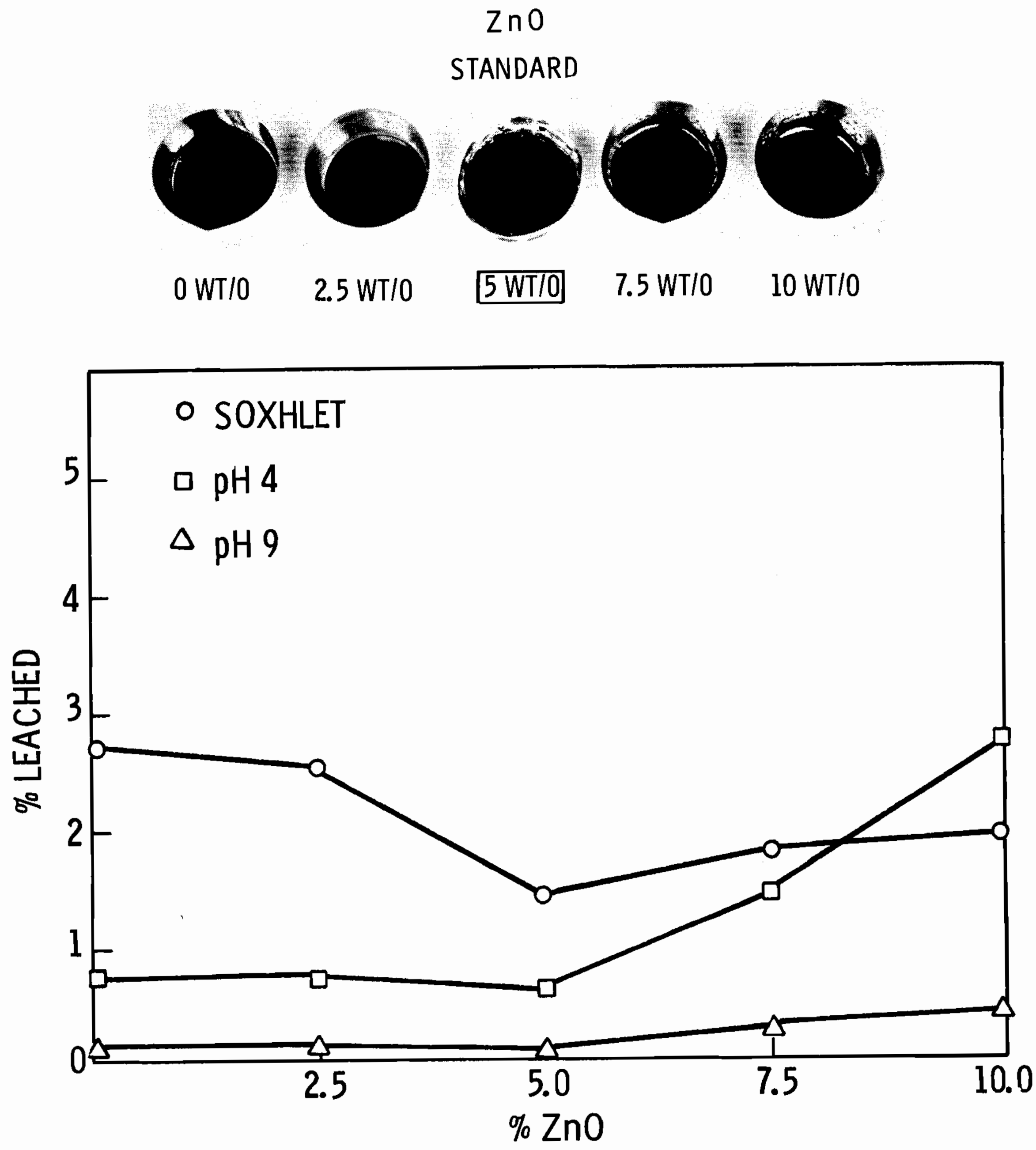


FIGURE 8. Leachability of 76-375 Type Glass with Change in ZnO Content

Soxhlet test), however, continues to decrease even at 21 wt% ZnO. The influence of the type of leach test on leach rate is one of the principal reasons that three leach tests were utilized for these tests. The 5 wt% ZnO content has been chosen as a balance between the various leach requirements. ZnO does not appear to aid homogeneity in this waste glass, but it can generally be beneficial in aiding viscosity. Furthermore, high ZnO content readily forms a Zn_2SiO_4 crystal as a devitrification product, which is discussed in more detail in a separate report.(11)

For all of these reasons, it is apparent that the initially selected frit formulation is near optimum. Further tests have been made to determine the effectiveness of this type of formulation for processing requirements.

VARIABILITY OF WASTE CONTENT

Process design and instrumentation can provide close control of mixing ratios of waste and frit additives, but at times ratios may not be at the desired 2 frit to 1 waste oxide ratio. Tests have been run on different mixtures to determine how far the ratio can vary without causing major problems with the product. The effect of variations in waste content on leachability and viscosity, two of the most important parameters, is shown in Figure 9. Note that initial waste additions actually reduce both viscosity and leachability. Limits for in-can melting with this composition would be about 15 to 45 wt% waste. The ceramic melter for which this formulation was intended should not have a viscosity problem at the higher 1150°C temperature over

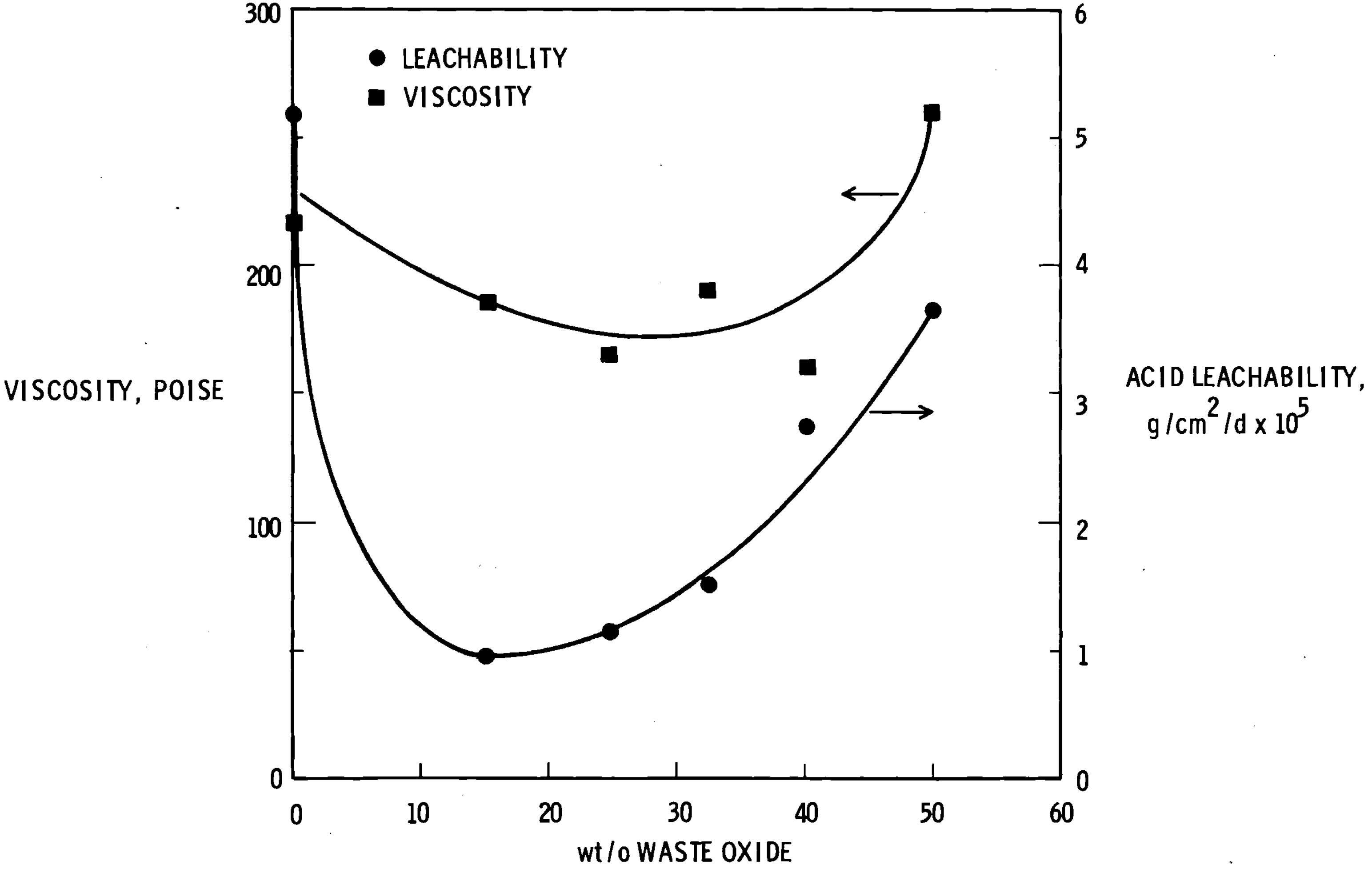


FIGURE 9. Effect of Waste Content on Viscosity at 1050°C and Acid Leachability as a Function of Waste Oxide Content

the total range tested (0-50% waste). However, the acid and soxhlet leachability are higher at the upper and lower limits of waste content, but would not be serious enough to require rework. These initial results indicate that there is a very wide range for acceptable frit-to-waste ratios; more detailed studies are planned to examine thermal and radiation effects.

SODIUM MOLYBDATE CONTROL

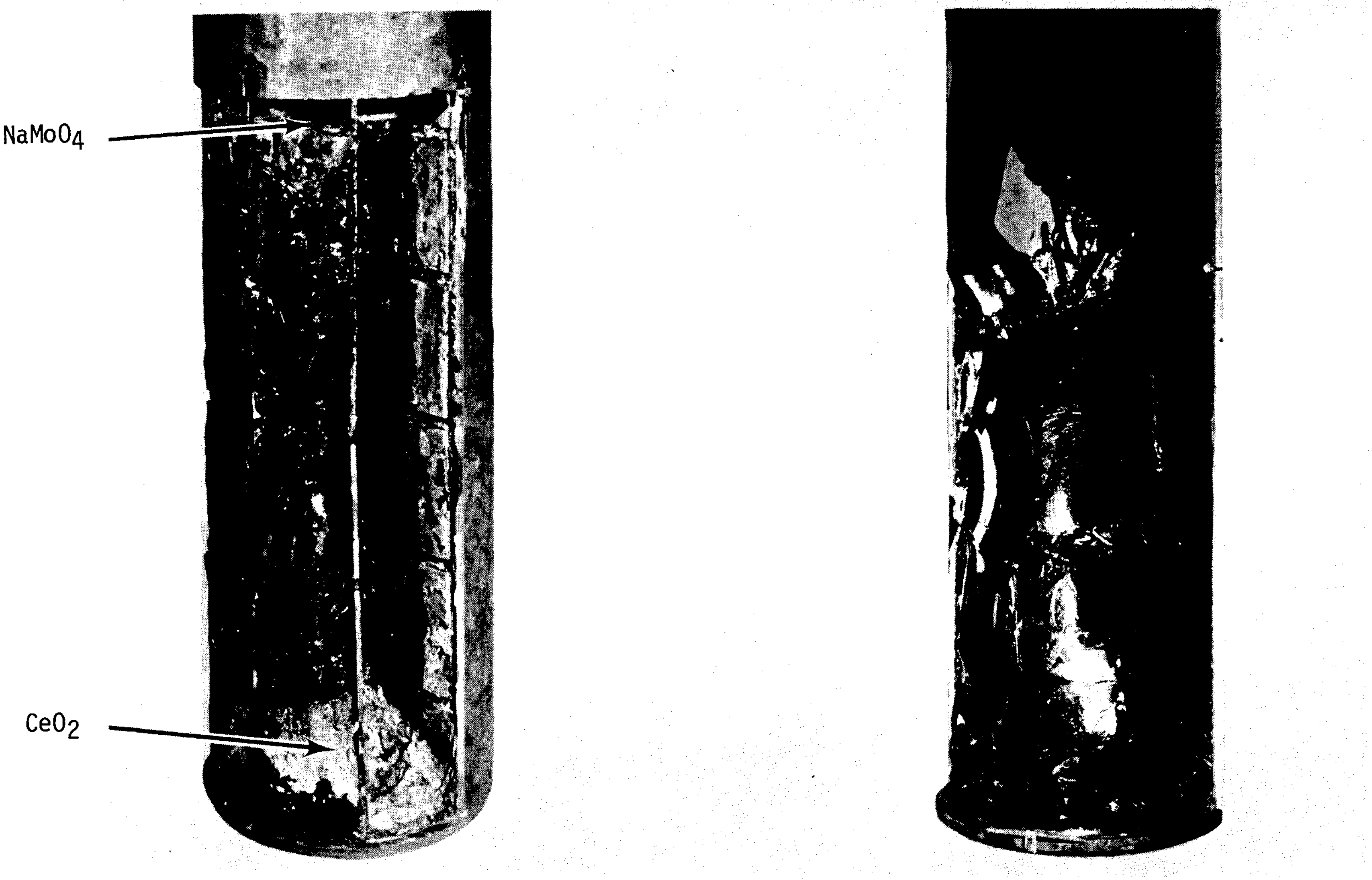
As discussed earlier, Na_2MoO_4 separation can be a problem with some waste types, and B_2O_3 can aid in controlling its behavior. However, additional methods of control are needed since separation may occur at high waste loadings even with higher B_2O_3 content. Na_2MoO_4 may also separate in cooler regions of process equipment since it has lower solubility in the melts at lower temperatures.

The chemical behavior of molybdenum can be controlled by reducing the high valence state (Mo^{+6}) which causes its unique behavior. To reduce the valence of molybdate, reducing agents were added to the glass, e.g., silicon metal powder may react with the Na_2MoO_4 as indicated in the following equation:

$$Na_2O + 2Na_2MoO_4 + 3Si \rightarrow 3Na_2SiO_3 + 2Mo$$

$$\Delta G° \approx -250 \text{ kcal}$$

From standard free energies we calculated that the reaction has a large potential and so proceeds easily. Although the equation shows Mo^{+6} reduced completely to the metal, we have found that it is not necessary to reduce to that extent. Both laboratory and engineering scale tests have verified the effects of reducing agents on the glass behavior (a comparison of engineering scale canisters is shown in Figure 10). The absence of the yellow surface Na_2MoO_4 phase (arrow) can be noted. Also, unmelted material (principally CeO_2) exists in the lower part of the canister (arrow). The reducing agents also appear to act on it to reduce it to Ce_2O_3 which is more soluble in the glass. Other oxides, such as ruthenium, rhodium, palladium, nickel and tellurium, are reduced to the metal state by addition of reducing agents. This is due to their lower free energy of formation compared to molybdenum, and they therefore exist as fine dispersed metallic crystals in the glass. Palladium and rhodium metal crystals have also been observed in nonreduced melts. Therefore, reducing agents have not created a new problem, but have increased the concentrations of insoluble metals in the glass. The overall effects appear very beneficial.



EARLY MELT SHOWING PHASE SEPARATIONS

RECENT MELT SHOWING REDUCTION OF PHASE SEPARATION

FIGURE 10. Comparison of Engineering Scale Canisters Showing the Improvement in Homogeneity with Use of Reducing Agents

CONCLUSIONS

In summary, the homogeneity and leach rates of waste glasses can be controlled by their composition. Since the two properties are generally competing, judgement is required in establishing the composition limits for the glass additives.

- Na_2O, K_2O and CaO have the greatest effect on homogeneity and leachability.
- Boric oxide additions and reducing agents are effective methods for sodium molybdate control.
- There are wide limits on the ratio of frit to HLW oxide which can be incorporated in glasses without serious effect on important properties of viscosity and leach rate.
- The reference composition used in this test is near optimum for the composition ranges tested.

ACKNOWLEDGMENT

Contributions to this study by J. C. Nelson and S. K. Jantz are gratefully acknowledged.

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