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OPTIMIZATION OF GLASS COMPOSITION FOR THE VITRIFICATION OF NUCLEAR WASTE AT THE SAVANNAH RIVER PLANT

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* E. I. du Pont de Nemours & Company
Savannah River Laboratory
Aiken, South Carolina 29808

** Ferro Corporation/Technical Center
7500 East Pleasant Valley Road
Independence, Ohio 44131

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ABSTRACT

Waste glasses of different compositions were compared in terms of leachability, viscosity, liquidus temperature, and coefficient of expansion. The compositions of the glasses were determined by statistical optimization. Waste glass of the optimized composition is more durable than the current reference composition but can still be processed at low temperature.

The information contained in this article was developed during the course of work under Contract No. DE-AC09-76SR00001 with the U.S. Department of Energy.

INTRODUCTION

In its 25 years of production of defense materials, the Savannah River Plant has generated about 25 million gallons of radioactive waste byproducts. This waste is currently stored in large underground tanks on the plant site. Processing of the waste into a form suitable for long-term disposal has been the subject of intense research efforts at the Savannah River Laboratory and elsewhere.

The waste consists of three fractions: an insoluble sludge, a salt cake, and a saturated supernatant solution. The sludge contains 95% of the total radioactivity of the waste, including virtually all of the actinides and long-lived radionuclides. The primary radionuclide in the salt and supernatant solution is Cs-137, only 5% of which is found in the sludge. The sludge consists primarily of hydroxides and hydrous oxides of aluminum, iron, and manganese. These dictate the chemical and physical properties of the waste and play a major role in defining the limits of the solidification process.

Incorporation of the waste into borosilicate glass is the current reference process for immobilization. The properties of the resulting product glass are determined not only by the waste, but also by the glass-forming materials added during melting. This borosilicate glass frit is roughly 70% by weight of the final glass waste form. This frit must accommodate the entire range of waste compositions, which varies significantly (see Table 1).

Currently, melting temperatures are limited to 1150°C by the volatility of radionuclides such as cesium and ruthenium, which means that the frit must be able to dissolve the waste at this temperature. The resulting waste glass must be highly resistant to aqueous attack.

An optimum frit was defined as one which produced waste glass with a leachability as low as possible, with a maximum viscosity at 1150°C as near 150 poise as possible, with a liquidus temperature as low as possible, and with a coefficient of thermal expansion as low as possible.

Such a frit composition was found after only 25 trials, in spite of the fact that eight chemical components were studied. This was achieved through application of the Nelder-Mead simplex algorithm, which uses the frits themselves to point toward the direction of improvement.

Properties and Optimization Criteria

Waste glasses made from each frit were compared in terms of their viscosities, coefficients of thermal expansion, leachabilities and liquidus temperatures. Since the glass frit must accommodate the entire range of waste compositions, each property was measured under "worst case" conditions -- high aluminum waste for viscosities and high iron for the other three properties. The waste compositions and concentrations are shown in Table 1. The high-aluminum and high-iron waste simulations correspond to the highest concentrations of these two components in any SRP waste. The TDS-3A and Stage 1 simulations represent average compositions. The Stage 1 simulation is more recent and represents a somewhat larger data base than was available when TDS-3A was developed. The melting temperature, 1150°C, and waste concentrations were held fixed at the values specified for use with the current reference frit, Frit 131.

- **Viscosity**

  High aluminum waste was used to prepare glasses for viscosity measurements since it produces melts of higher viscosity than other waste types.[1] An optimum viscosity at 1150°C of 150 poise was chosen. This represents a practical maximum for processing SRP waste. Increasing melt viscosity should reduce melter corrosion and volatility. Viscosities were measured using a Brookfield rotating spindle viscometer.

- **Liquidus**

  High iron waste was used to prepare glasses for liquidus determinations since it is the most likely to form spinels during glass melting.[2] The liquidus temperature with high iron waste was to be minimized, to reduce the tendency to form slag in the melter. The liquidus was based on 24-hour tests in a gradient furnace.

- Leach Rate

The leach rate used was the geometric mean of the leach rates at pH = 4, 7, and 10. It was to be minimized in the optimization. All leach tests were performed at 90°C on -40+60 mesh grains of simulated waste glass, which has a specific surface area of 0.007 m^2/g. For the first twelve frits, TDS-3A simulated waste was used and the tests were based on 2 g of glass in 20 mL of pH = 3, 7, and 11 buffer for three days. Leach rates were calculated from the concentration of silicon in the leaching solutions as measured by inductively coupled plasma emission spectroscopy and on the calculated fraction of silicon in the simulated waste glass. Since the only function of this test is to compare different glasses, a more sophisticated test is not justified. Several changes were made in the leach tests used after the first round to improve the ability of the test to accurately discriminate between the waste glass compositions tested. To minimize possible saturation of the leach solution, the mass of glass was halved (to 1 g), the volume of solution was increased (to 40 mL), and the test was shortened to 24 hours. In the first round, the laboratory buffers used were not always able to buffer the leach solutions, so they were replaced by commercial pH 4, 7, and 10 buffers. Finally, high iron waste was used in place of TDS-3A simulated waste, since this produced glass of lower durability.[2] Because of variations in leach test results, all of the waste glasses to be compared in a given round were tested together, even if earlier data were available.

Coefficient of Thermal Expansion

This property, measured on high iron waste glass, was to be minimized. Since the strength of the glass is inversely proportional to this coefficient, cracking may be reduced as the coefficient is lowered. The coefficient of thermal expansion was measured on an Orton dilatometer. The values reported are those for the range 25 to 300°C.

These four properties were combined into a quality index for the different compositions. Each property was projected on a common scale, and weighted in proportion to its importance. The common scale ran from 0 (intolerable) to 1 (optimum). The relationship of this scale to the measured properties is shown in Slide 1. Except for the viscosity, each scale runs in a linear fashion from the intolerable to the optimum value. The viscosity is treated differently in order to reduce the penalty for small deviations from the optimum value of 150 poise. The following relationship between the viscosity η and its desirability coefficient C_η was used:

$$C_\eta = \left(1 - \frac{|\eta - 150|}{150}\right)^{\frac{1}{2}}$$

Futhermore, C_η was not allowed to drop below 0.258, its value at 10 or 290 poise. If the value of C_η had been allowed to go to zero, this value would have unduly affected the resultant statistical optimization. In practice this made little difference, since a C_η value of 0.258 was enough to eliminate the frit from consideration as the best possible one.

The common scale eliminated the problem of "comparing apples and oranges," e.g., a value of 0.8 on the scale corresponds to a viscosity of 96 or 204 poise, a liquidus temperature of 700°C, a coefficient of thermal expansion of $88 \times 10^{-7}/°C$, or a (geometric) mean leach rate of $0.100\ g/m^2$-day.

These properties are not all of equal importance. The durability is most important with a relative ranking of 40%. Viscosity is next with 30%, then liquidus temperature at 20%, and finally the coefficient of thermal expansion at 10%. Since the overall coefficient of desirability is the geometric mean of the desirability coefficients for the individual properties, these weighting factors appear as exponents -- 1.6 for durability, 1.2 for viscosity, 0.8 for liquidus, and 0.4 for the coefficient of thermal expansion. These exponents are readily calculated from the weights and the requirement that they sum to the number of properties, 4. Slide 2 shows how these exponents shift the desirability coefficients. A desirability coefficient of 0.8 on the common scale corresponds to a weighted coefficient of 0.70 for the durability, 0.77 for the viscosity, 0.84 for the liquidus, and 0.91 for the coefficient of thermal expansion. This is just as one would expect: the weighted coefficient for the durability has a greater ability to reduce the frit's overall desirability coefficient than does that for the coefficient of thermal expansion.

Once the desirability coefficient of each waste glass was calculated, the frits were ranked from best (geometric mean of weighted desirability coefficients closest to 1) to worst. The rankings and properties of the waste glasses included in the first, second and third rounds are listed in Tables 2, 3 and 4, respectively.

The rankings in each round were used to generate the compositions to be studied in the next round as described below.

Compositions

The compositions of all frits studied in this program are shown in Table 5. The first twelve compositions were determined by a Plackett-Burman design.[3] Each of the eight components was assigned a "high" value and a "low" value, e.g., 70 and 60 parts by weight for silica. The compositions were normalized to 100 wt %.

Data were collected on waste glasses made from the first twelve frits and the frits were then ranked as described above. The four worst compositions were replaced. It is this focusing on the <u>worst</u> cases which is the strength of this approach. The optimum frit is hard to identify because its exact properties are unknown. There is certainly no reason to assume that it is one of the compositions initially studied. On the other hand, the less desirable frits are quite easy to pick out -- here one need not worry about whether it is the worst possible frit, but simply whether it is poor.

These four frits are used to generate the next set of trial frits through a simplex algorithm. This algorithm was first developed by Spendley et al[4] and later extended by Nelder and Mead.[5] A lucid but somewhat less technical exposition is also available.[6] The composition of each poor frit is subtracted from twice the average of the eight better frits. This is a reflection of the poor composition through the point of average composition in composition space. There is no absolute guarantee that the new composition will be better (i.e., have a higher desirability coefficient) than the one it replaces. It may overshoot the optimum region altogether. However, as long as there is any progress the optimum region will eventually be approached. Slide 3 shows a two-dimensional respresentation of this reflection for the twelve frits initially studied. Referring to Tables 2 and 5, Frit 141 was replaced by Frit 154, Frit 142 by Frit 155, Frit 148 by Frit 156, and Frit 149 by Frit 157. These twelve frits -- eight older ones and four new ones -- were studied as a group and analyzed in exactly the same way as was the first group. This led to replacing Frits 144, 146, 155, and 157 by Frits 158, 159, 160, and 161 respectively. This evolution can be followed by examining Tables 2 through 4, where the data on the simulated waste glasses made from each frit appears as well as the desirability coefficients used to determine the ranking.

While the statistics work by examining the worst frits, the end result is reached by examining the best. The statistically directed part of this experiment was concluded once it became clear that the region of best composition was not changing significantly. This occurred after the third round. By this time twenty frits had been studied.

The data representing these frits served as the basis for the final step, the proposal of an "optimum" frit. Two different approaches were used to generate this composition. One was to use knowledge attained in general glass manufacture along with experience with the first twenty frits to produce the compositions of Frit 162, 163, and 166. The other approach was an attempt to fit the observed properties to linear combinations of the components. This was reasonably successful in the cases of the two most important properties, the (geometric) mean leach rate and the viscosity. Although a linear fit of the liquidus temperature failed every statistical test for significance, it did suggest that lanthana was the only component to dramatically increase (i.e., worsen) that temperature. Because of its low weighting, the coefficient of thermal expansion was not modeled. The results were applied in developing the compositions of Frits 164 and 165.

Both of these approaches reached similar conclusions as regards the effects of most of the components. In general, higher amounts of silica and zirconia improve durability. The higher viscosity is best compensated for by increasing lithia relative to soda since it is a more effective flux (on a weight percent basis) and has a less deleterious effect on the leach rate. Lanthana has no benefits to recommend its inclusion. Boric oxide, magnesia, and titania have a relatively smaller impact on the measured properties.

The properties of these final five frits are compared to that of Frit 154, the best frit generated by the simplex algorithm, in Table 6. Three of the five -- Frits 164, 165, and 166 -- were significantly better than Frit 154. These three were so similar in desirability coefficient and in their individual properties that an additional test to choose between them was needed. The test used was a simple side-by-side comparison of their resistance to devitrification.

Frit 131 (the current reference frit) and Frits 154, 164, 165 and 166 were used to prepare waste glasses using "Stage 1" simulated waste calcine (see Table 1). After being held at 1150°C for eight hours, they were cooled in a programmed furnace in such a way as to mimic the behavior in the center of an uninsulated stainless steel canister of the kind proposed for nuclear waste glass.[7] Glass in this location sees the slowest cooling rate and spends the longest time between the liquidus and the glass transition temperatures. It represents the worst case for crystal formation. The results of this experiment were striking; Frit 131 waste glass contained the most crystalline material, followed by Frits 166, 154, 164 and 165. In Frit 165, only a few small well-separated cyrstals could be found. As a result of this test, Frit 165 was chosen for further study.

SUMMARY

The compositions and properties of the reference frit, Frit 131, and the best frit of the optimization program, Frit 165, are compared in Tables 7 and 8. Frit 165 waste glass is superior to Frit 131 waste glass with respect to every property measured. It is more resistant to leaching, especially in acid solution; it is more viscous, reducing corrosion of electrodes and refractory materials in the melter; it is less likely to devitrify under adverse cooling conditions; and it has a lower coefficient of thermal expansion, which may reduce the severity of cracking during cooling.

The development of Frit 165 required only 25 test frits, demonstrating the power of the simplex approach even in as complicated a problem as this one.

The properties included in this study are not the only ones of interest in processing nuclear waste glass. The inclusion of all such properties would have made the experiment intractable. Future work on Frit 165 will shift from crucible tests of the sort described here to full-scale studies which will measure its performance in each phase of melter operation -- feeding, melting, and pouring -- as well as examine the quality of the glass produced in longer-term tests. It is anticipated that Frit 165 will continue to show both processing and product quality improvements over earlier frits.

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TABLE 1

Simulated Calcine Compositions

	Amount (Wt %)			
Component	W-Al	W-Fe	Stage 1	TDS-3A
Fe_2O_3	13.8	59.1	41.3	47.3
MnO_2	11.3	4.0	11.5	13.6
Zeolite*	10.2	9.7	6.7	10.2
Al_2O_3	49.3	1.4	15.2	9.5
NiO	2.0	10.1	3.5	5.8
SiO_2	4.5	2.9	10.6	4.1
CaO	0.9	4.0	4.9	3.5
Na_2O	5.0	5.9	--	3.1
Coal	2.3	2.1	1.0	2.3
Na_2SO_4	0.7	0.8	0.4	0.6
Na_2CO_3	--	--	4.9	--
Frit/Calcine Ratio	71.3/28.7	70.2/29.8	69.4/30.6	70.2/29.8

* Linde Ion-Siv IE-95.

TABLE 2

Results of the First Round in Order of Decreasing Desirability Coefficient

	Leach Rates,* (g/m^2-day)							
Frit	pH 3	pH 7	pH 11	Geometric Mean	Viscosity** (Poise)	Liquidus† Temp., °C	Coefficient† of Expansion, 10^{-7}/°C	Weighted Desirability Coefficient
147	0.069	0.034	0.84	0.125	164	804	97.5	0.746
143	0.074	0.036	1.30	0.151	174	835	100.4	0.692
145	0.052	0.063	2.00	0.187	137	820	108.4	0.643
152	0.093	0.055	1.70	0.206	123	840	102.5	0.634
151	0.091	0.055	1.90	0.212	98	860	100.4	0.605
146	0.084	0.032	1.40	0.156	100	927	104.0	0.598
150	0.095	0.070	1.70	0.224	102	790	109.1	0.593
144	0.100	0.093	1.10	0.217	82	784	109.8	0.579
148	0.100	0.069	1.00	0.190	98	937	99.6	0.579
149	0.046	0.009	0.91	0.072	313	848	88.7	0.532
142	0.051	0.011	0.61	0.070	334	870	88.0	0.525
141	0.130	0.096	1.90	0.287	94	862	106.2	0.513

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* Based on 3-day tests at 90°C of glass powder prepared using TDS-3A waste.

** With high aluminum waste.

†† With high iron waste.

TABLE 3

Results of the Second Round in Order of Decreasing Desirability Coefficient

	Leach Rates,* g/m^2-day							
Frit	pH 4	pH 7	pH 10	Geometric Mean	Viscosity** (Poise)	Liquidus Temp.,† °C	Coefficient of Expansion,† 10^{-7}/°C	Weighted Desirability Coefficient
154	0.207	0.112	0.437	0.216	176	850	98.2	0.635
147	0.455	0.084	0.466	0.261	164	804	97.5	0.623
143	0.248	0.110	0.477	0.235	174	835	100.4	0.620
156	0.441	0.084	0.406	0.247	177	840	99.6	0.606
145	0.346	0.110	0.468	0.261	137	820	108.4	0.578
152	0.597	0.088	0.388	0.273	123	840	102.5	0.572
151	0.337	0.115	0.469	0.263	98	860	100.4	
150	0.581	0.083	0.419	0.272	102	790	109.1	
146	0.438	0.096	0.432	0.263	100	927	104.0	
144	0.639	0.098	0.431	0.300	82	784	109.8	
157	0.611	0.116	0.487	0.326	61	805	112.7	
155	0.605	0.159	0.461	0.354	80	800	111.3	

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* Based on 1-day tests at 90°C on powder, with high iron waste.

** With high iron waste.

† With high aluminum waste.

TABLE 4

Results of the Third Round in Order of Decreasing Desirability Coefficient

	Leach Rates* (g/m^2-day)							
Frit	pH 4	pH 7	pH 10	Geometric Mean	Viscosity,** (Poise)	Liquidus† Temp., °C	Coefficient of expansion,† 10^{-7}/°C	Weighted Desirability Coefficient
154	0.171	0.088	0.451	0.189	176	850	98.2	0.658
147	0.560	0.067	0.428	0.252	164	804	97.5	0.633
151	0.324	0.096	0.491	0.248	98	860	100.4	0.573
152	0.471	0.108	0.426	0.279	123	840	102.5	0.566
160	0.357	0.210	0.427	0.318	158	840	96.7	0.551
156	0.521	0.119	0.461	0.306	177	840	99.6	0.545
150	0.576	0.088	0.426	0.278	102	790	109.1	0.544
145	0.601	0.101	0.479	0.307	137	820	108.4	0.530
143	0.437	0.183	0.610	0.365	174	835	100.4	0.473
161	0.095	0.253	0.521	0.232	1200	890	88.0	0.427
159	0.163	0.282	0.525	0.289	376	953	92.4	0.356
158	0.296	0.288	0.422	0.330	377	900	85.8	0.354

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* Based on 1-day tests at 90°C on powder.

** With high iron waste.

†† With high aluminum waste.

TABLE 5

Composition of Frits

	Weight Percent							
Frit	SiO_2	B_2O_3	Na_2O	Li_2O	MgO	TiO_2	La_2O_3	ZrO_2
141	58.8	14.7	17.6	3.9	2.0	2.0	1.0	0.0
142	66.0	14.2	12.3	3.8	0.9	1.9	0.0	0.9
143	64.8	13.9	12.0	5.6	1.9	1.9	0.0	0.0
144	59.4	14.9	17.8	5.9	1.0	0.0	0.0	1.0
145	64.8	11.1	16.7	5.6	1.9	0.0	0.0	0.0
146	63.1	10.8	16.2	5.4	0.9	1.8	0.9	0.9
147	66.7	13.3	14.4	4.4	1.1	0.0	0.0	0.0
148	61.2	15.3	13.3	6.1	2.0	0.0	1.0	1.0
149	68.0	11.7	12.6	3.9	1.9	0.0	1.0	1.0
150	60.6	12.1	18.2	4.0	2.0	2.0	0.0	1.0
151	63.2	12.6	13.7	6.3	1.1	2.1	1.1	0.0
152	64.2	13.8	16.5	3.7	0.9	0.0	0.9	0.0
154	67.6	10.9	13.8	6.3	0.7	0.0	0.0	0.7
155	60.3	11.3	19.3	6.5	1.8	0.0	0.8	0.0
156	65.0	10.4	17.9	4.2	0.7	1.9	0.0	0.0
157	58.4	13.9	18.7	6.3	0.7	1.9	0.0	0.0
158	68.5	13.1	11.7	3.5	0.8	1.9	0.0	0.4
159	69.3	9.7	13.0	4.1	1.6	1.9	0.5	0.0
160	65.7	13.8	14.3	4.5	1.7	0.0	0.0	0.0
161	70.9	10.6	12.1	3.7	1.8	0.0	0.5	0.4
162	67.5	13.0	13.0	4.0	1.0	1.0	0.0	0.5
163	68.0	13.0	12.0	4.5	1.0	1.0	0.0	0.5
164	67.0	10.0	13.0	7.0	1.0	0.0	0.0	2.0
165	68.0	10.0	13.0	7.0	1.0	0.0	0.0	1.0
166	67.0	12.0	14.0	5.5	1.0	0.0	0.0	0.5

TABLE 6

Results of the Final Round in Order of Decreasing Desirability Coefficient

	Leach Rates,* g/m^2-day							
Frit	pH 4	pH 7	pH 10	Geometric Mean	Viscosity** poise	Liquidus Temp,*** °C	Coefficient of Expansion,*** 10^{-7}/°C	Weighted Desirabilit Coefficient
166	0.215	0.119	0.461	0.228	159	855	97.4	0.636
164	0.135	0.174	0.434	0.217	130	855	99.6	0.632
165	0.165	0.125	0.477	0.214	140††	875	100.4	0.628
154†	0.278	0.151	0.491	0.274	176	850	98.2	0.579
163	0.247	0.145	0.497	0.261	229	865	88.7	0.558
162	0.287	0.138	0.543	0.278	382	845	90.2	
131†	0.586	0.161	0.458	0.351	84	957	110.5	

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* Based on 1-day tests at 90°C on powder, with high iron waste.

** With high aluminum waste.

*** With high iron waste.

† Included for reference.

†† Estimated.

TABLE 7

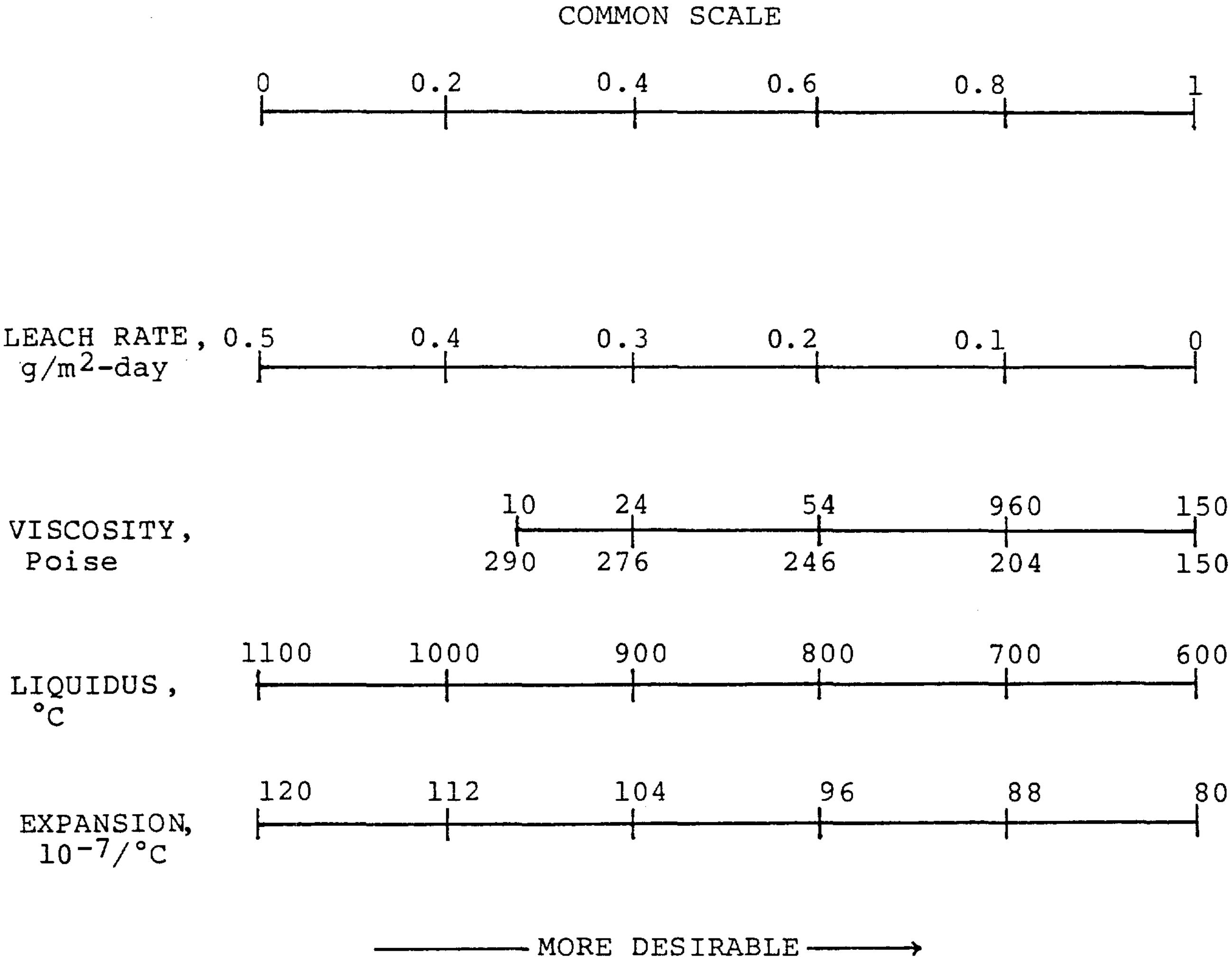
Compositions of Frits 131 and 165

Component	Weight Percent Frit 131	Frit 165
SiO_2	57.9	68.0
B_2O_3	14.7	10.0
Na_2O	17.7	13.0
Li_2O	5.7	7.0
MgO	2.0	1.0
TiO_2	1.0	--
La_2O_3	0.5	--
ZrO_2	0.5	1.0

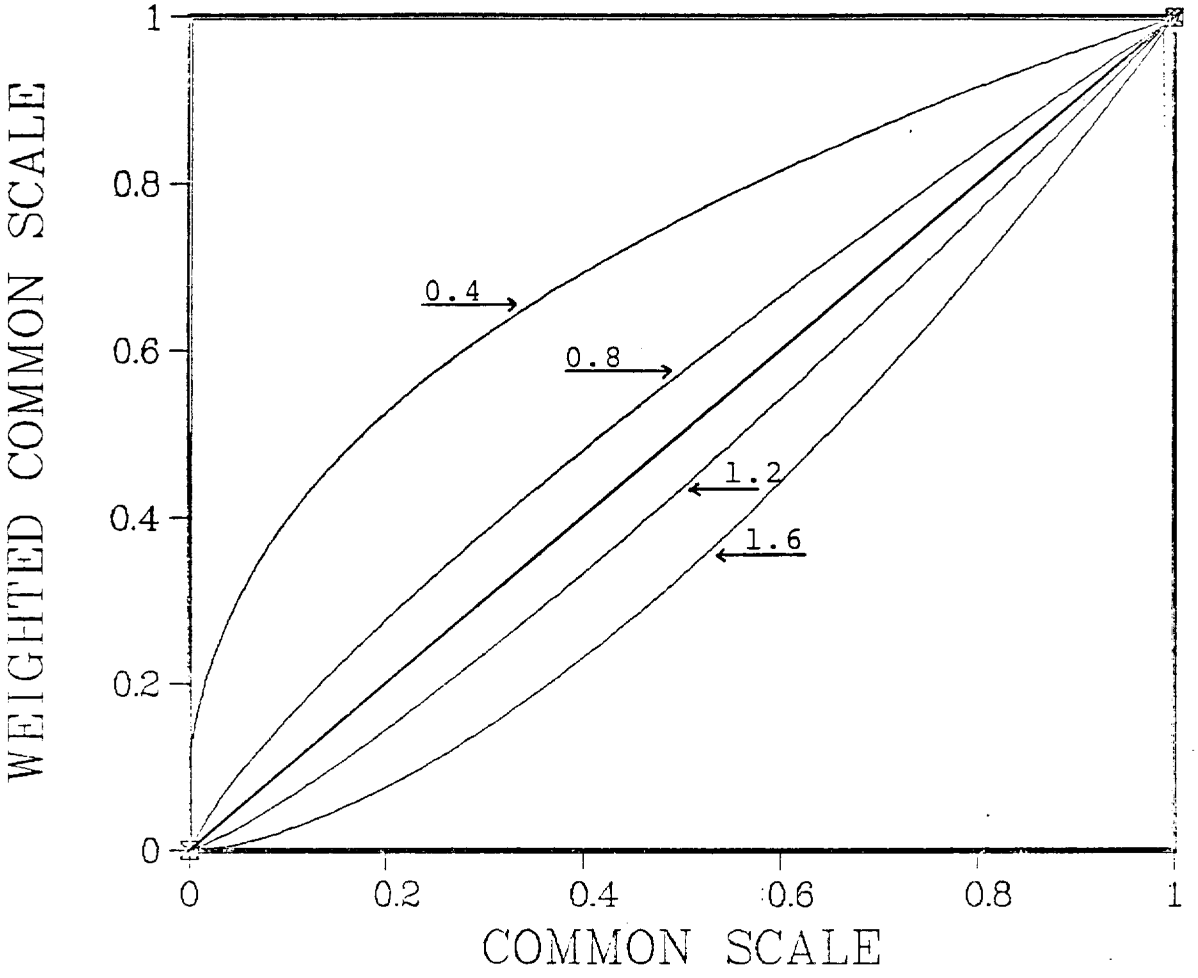
TABLE 8

Properties of Waste Glasses Made from Frits 131 and 165

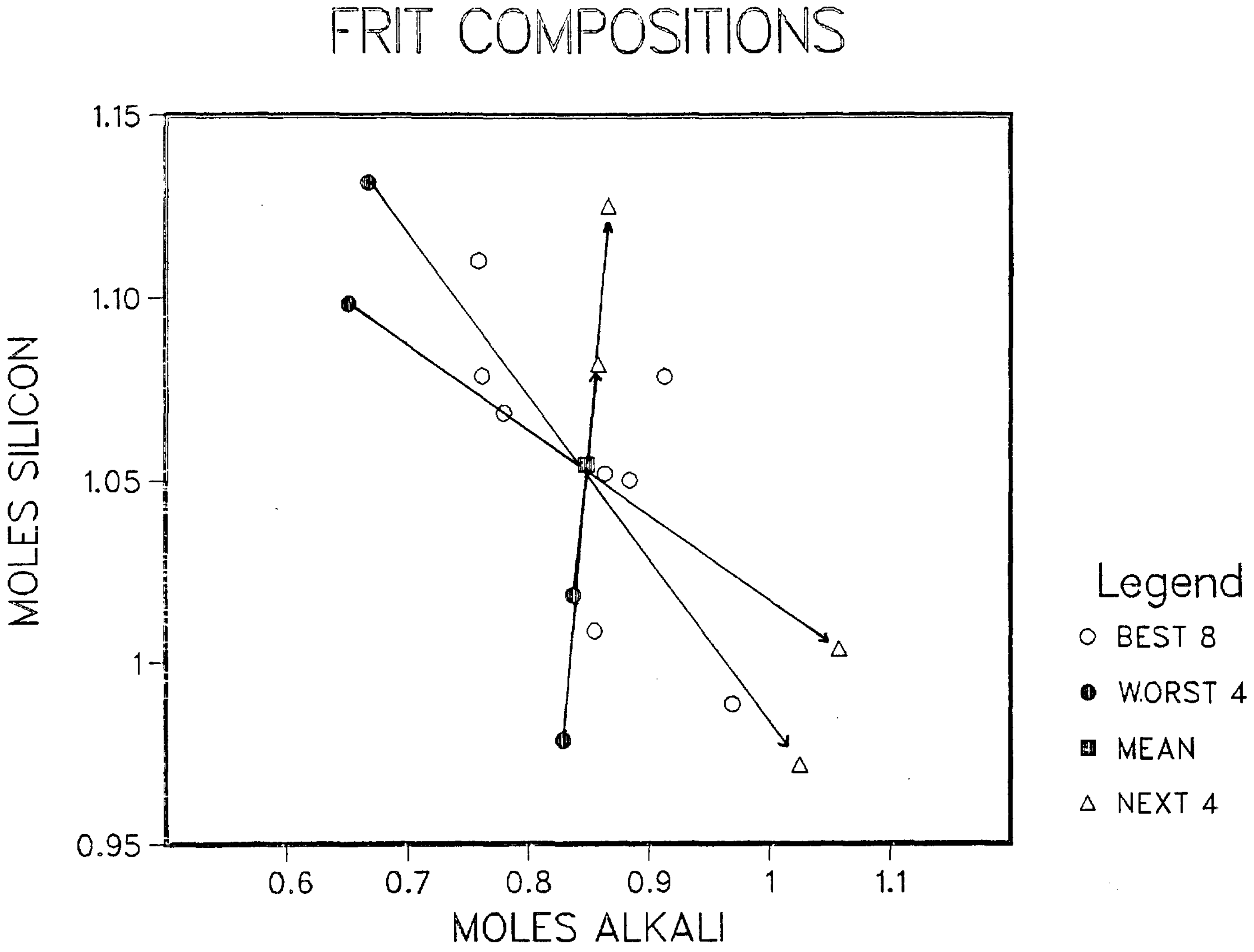
	Waste Glass	
Property	Frit 131	Frit 165
Leach Rates g/m^2-day		
pH4	0.586	0.165
pH7	0.161	0.125
pH10	0.458	0.477
Geometric Mean	0.351	0.214
Viscosity, Poise (at 1150°)	84.0	140.0
Liquidus Temp., °C	957.0	875.0
Coefficient of Expansion 10^{-7}/°C	110.5	100.4
Tg, °C	445.0	449.0
Dilatometric Softening Point, °C	490.0	484.0



SLIDE 1. **Relationship Between the Measured Properties and the Common Scale. A Common Scale Value of 0 is Intolerable while a Values of 1 is ideal.**



SLIDE 2. Relationship Between the Common Scale and the Weighted Common Scale. The Mean Leach Rate Weighting Factor is 1.6, the Viscosity Factor is 1.2, the Liquidus Factor is 0.8, and the Expansion Factor is 0.4.



SLIDE 3. Composition Generation by the Nelder-Mead Simplex Algorithm. The Data on Waste Glasses Made with the Twelve Frits are Used to Rank Them. They are Split into the Best 8 (0) and the Worst 4 (◐). The Average Composition of the Best 8 is Calculated (■). Each of the Worst 4 Compositions is Reflected Through the Average to Give a New Composition (Δ).