

LEACH MECHANISMS OF BOROSILICATE GLASS  
DEFENSE WASTE FORMS -- EFFECTS OF COMPOSITION

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ABSTRACT

The study described below concerns the mechanisms which control the leaching of two Defense Waste-forms, viz. SRL TDS-131 glass and MCC Defense Waste Reference Glass. It is shown that both the leach mechanisms and the structure of the leached surface are strongly dependent not only on the composition of the leachant and the contact time between the leachant and the glass but also on the exact composition of the glass. The relatively minor differences in composition between the two glasses investigated here were observed to give rise to large differences in leach behavior, in particular upon prolonged contact with water, rather than to limited changes in leach rate alone.

COMPOSITION OF TESTED GLASSES

The two glasses used in this study were Savannah River Laboratory TDS-131 glass<sup>1</sup> and Materials Characterization Center Defense Waste Reference Glass (DWRG),<sup>2</sup> which is generically similar in composition to SRL TDS-165 glass.<sup>1</sup> The major components of the two glasses as determined by the originating laboratories and in the authors' laboratory are given in Table I. The two glasses do not appear very different; DWRG contains (on a relative basis) about 40% less Na<sub>2</sub>O, 30% less Fe<sub>2</sub>O<sub>3</sub> and 25% less B<sub>2</sub>O<sub>3</sub> and MnO, than SRL TDS-131, and on the other hand it has about 15% more SiO<sub>2</sub> and 40% more Al<sub>2</sub>O<sub>3</sub>, as well as an appreciable quantity of U<sub>3</sub>O<sub>8</sub>.

SHORT-CONTACT-TIME TESTS

At high flow rates or high dilutions, corresponding to short contact times between the glass and the surrounding water, resulting in little effect of glass dissolution products on the composition and reactivity of the solution, the leach behavior of the two glasses is not very different. A modified IAEA<sup>3</sup> frequent-exchange test at 90°C on polished rectangular samples using de-ionized water as the leachant shows that at the end of 30, 120 or 360 days the leach rates of DWRG are lower by a factor of about 1.5 than the leach rates of TDS-131 (Table IV). These results agree with findings concerning the relative durabilities of

SRL glasses TDS-131 and TDS-165 (the latter is generically similar to DWRG) in short-term (3-day) tests carried out in neutral solutions.<sup>1</sup>

PULSED-FLOW TESTS

Most of the leach tests reported here were carried out using a partial-exchange dynamic test,<sup>5,8</sup> which permits investigating the effects of flow over a wide range of contact times (or flow rates). Representative results of this test carried out at 70°C on polished rectangular samples at several flow rates, using both de-ionized water and simulated Grande Ronde basaltic ground-water<sup>9</sup> as leachants, are given in Table III. The data show that under flow conditions equivalent to a relatively high flow rate of about 11 m/yr the leach rates obtained with DWRG are lower only by 40% than those obtained with SRL TDS-131 glass, in agreement with the IAEA test results. However, when the flow rates are reduced to about 2 m/yr the leach rates of the major components of SRL TDS-131 glass decrease only by 15-20%, while those of DWRG drop by a factor of 3-3.5. In other words, the leachate concentrations in the case of DWRG approach saturation at much shorter residence times than those at which saturation is observed in the case of SRL TDS-131 glass. The results in the case of leaching DWRG in ground-water are indicative of complete saturation, since a decrease by a factor of 7 in flow rate leads to a corresponding decrease in the leach rate, as shown in Table III.

Another indication of the difference between the behavior of the two glasses at low leach rates is provided by monitoring the leachate concentrations as a function of time. As previously discussed,<sup>8</sup> when in the course of a flow test the leachate concentrations are observed to rise steadily with time until they level off at a constant level, this observation indicates that the leaching process is controlled by a simple corrosion mechanism and that the surface layer is not protective. On the other hand, when the leachate concentrations rise until they reach maximum values and then decrease again, reflecting a decrease in leach rates, before steady-state is achieved, this is indicative of the formation and build-up of an altered surface layer which acts as a protective barrier. In the case of SRL TDS-131 glass at 70°C the second type of behavior only appears at flow rates of 0.12 m/yr or slower.<sup>8</sup> On the other hand, in the case of DWRG a kinetic pattern showing a maximum when leachate concentrations are followed as a function of time, indicative of the growth of a protective layer, is apparently observed at a flow rate of 2.42 m/yr, though not at 10.8 m/yr. (The appearance of this maximum at intermediate flow rates (around 2 m/yr) is particularly prominent in the case where the leachant is groundwater, and this provides further evidence for the conclusion that in this case the leachate composition becomes determined by saturation with respect to the leached, altered surface layer.) Furthermore, at flow rates below 2 m/yr the normalized leach rate based on Si becomes much slower than those of the alkalis and boron, as a stable silica-rich (and alumina-rich) leached layer is formed. It has been concluded that the formation of this layer, which controls the rate of further glass dissolution, involves alteration and re-precipitation processes.<sup>8-10</sup> The solubility of the silica in the altered layer in water, for instance, is much lower than the solubility of the silica present in the original glass.

These findings all indicate that at high flow rates the leach behavior of the two glasses is similar; however, at moderate flow rates DWRG begins to show a slowdown in leach rates as a result of saturation and the formation of a protective alteration layer while a similar effect in the case of SRL TDS-131 glass only takes place at extremely slow flow rates. This is likely to be the result of the larger extent of the pH excursion in the case of SRL TDS-131, which originates in its higher Na content (see Table I). As shown in Table III, at a relatively high flow rate of 11 m/yr the pH values of the leachates of the two glasses are not very different (10.0 and 9.65, respectively), but as the flow rate slows down to 2 m/yr the pH of the DWRG leachate remains virtually unaltered while that of SRL TDS-131 leachate rises to 10.7.

#### SOLUBILITY TESTS

The differences between the two glasses are sharpest under very slow flow or stagnant conditions. Static experiments were carried out at 70°C on large amounts (10 g) of powdered glass in a small volume (about 20 mL) of water. The results at the end of one year are given in Table II together with the total extent of depletion of each of the glass components based on its initial abundance in the specimen. It can be seen that the large difference in final pH causes SRL TDS-131 to decompose to a very large extent while

DWRG remains essentially intact under the same conditions. The difference in leachability between the soluble components of the two glasses, respectively, corresponds to a factor of 20-100.

#### CHEMICALLY ACCELERATED TESTS

Flow tests were also carried out on DWRG using de-ionized water as well as two concentrations of NaOH. The results given in Table V show that DWRG, like SRL TDS-131, is much more leachable at high pH values and that the leach rates increase by nearly an order of magnitude when the pH is raised from 9.3 to 12.7. However, when the initial leachant is water, the pH of DWRG leachates is not expected to reach levels above 10-10.5 (see above) unless the flow rates are extremely small, corresponding to contact times exceeding 10<sup>2</sup> years at the expected repository S/V ratios of about 10<sup>2</sup> m<sup>-1</sup>. Such slow flow rates are probably unrealistic due to mass diffusion of water through the repository. Under expected service conditions, the ultimate pH level in the case of DWRG is unlikely to exceed the pH of the ground-water (around 9.7 in basalt formations<sup>9</sup>) by a considerable extent. On the other hand, in the case of SRL TDS-131 the pH is likely to rise to very high levels (11.5-12.5) even when flow rates are not unrealistically slow, overwhelming any buffering by the original groundwater composition. Since both glasses are observed to be subject to rapid matrix dissolution when the pH rises above a critical region around 9.5-10,<sup>6</sup> as demonstrated in the high-pH tests described in this section, the effective durability of DWRG at slow but finite flow rates is likely to exceed that of TDS-131 by as much as two orders of magnitude since only in the case of TDS-131 the pH of the surrounding water is expected to rise beyond the critical region. As mentioned above, the large difference in leach behavior between the two glasses is caused by a relatively small difference in Na content or Na:Si ratio; however, the slightly higher alkali content of SRL TDS-131 glass is apparently sufficient to prevent buffering of the leached alkali by acidic components of the glass (borate, silicate).

#### DISCUSSION: EFFECTS OF DIFFERENCE IN COMPOSITION BETWEEN TDS-131 GLASS AND DWRG ON LEACH RATES

Based on the experimental findings cited in this section, it can be concluded that the leach behavior and the controlling leach mechanisms of borosilicate glasses at a given temperature are strongly dependent not only on leachant composition and on flow/dilution conditions, but also on glass composition.

A relatively small increase in alkali content can cause, according to the findings listed above, a severe enhancement of the pH excursion upon leaching and this can inhibit the onset of saturation at moderately slow flow rates. In such cases leaching is not retarded until contact between the leachant and the glass has lasted for very long periods or until the glass undergoes decomposition to a large extent. Thus SRL TDS-131, although close in composition to DWRG and exhibiting leach rates which are only slightly higher at high dilutions, undergoes massive extraction of its soluble components upon prolonged contact with a given volume of water while DWRG is much more durable at long contact times.

Quantitatively, the large differences in leach behavior between TDS-131 and DWRG can be interpreted on the basis of the molar composition of the original glass and of the leachate obtained at the longest residence time in the two cases. The mole fraction  $X_B^g$  of each of the principal components of the glass is divided by  $X_B^g$ , the mole fraction of boron which is always the most leachable element<sup>10</sup>. The difference between the molar amounts of each species relative to boron in the leachate ( $X^l/X_B^l$ ) and in the solid glass ( $X^g/X_B^g$ ), respectively, serves as an indicator of the amount of that species which stays behind in or on the leached surface ( $X^s$ ) during the leaching process, i.e.  $X^s/X_B^g = X^g/X_B^g - X^l/X_B^l$ <sup>11</sup>. The results of this calculation in the cases of TDS-131 and of DWRG are given in Table VI. It can be seen that in the case of DWRG the total relative amount of alkalis in the leachate ( $X^l/X_B^l = 2.5$ ) is smaller than that of the buffering species silicate ( $pK = 9.7$ ) and borate ( $pK = 9.1$ ), for which the combined value of  $X^l/X_B^l$  is 3.7. On the other hand, in the case of TDS-131 the relative amount of alkalis in the leachate, 1.7, exceeds the corresponding combined amount of silicate and borate, 1.6, and this leads to a runaway pH rise under slow flow or stagnant conditions, and to a rapid decomposition of the glass. Under the same conditions DWRG exhibits a stabilized pH level around 10 and a much smaller degree of attack on the glass.

As regards the composition of the two glasses themselves, according to Table VI SRL TDS-131 contains only a combined mole fraction of 0.49 of the two acidic oxides  $SiO_2$  and  $B_2O_3$ . More than one-half of the glass composition is made of basic oxides which are either highly soluble ( $Na_2O$ ,  $Li_2O$ ) or tend at low flow rates to form insoluble silicates and thereby reduce the levels of dissolved silica which can buffer the solution against a pH runaway effect. On the other hand, DWRG contains a significant excess of acidic oxides (a mole fraction of 0.57) over the combined basic oxides in the glass, and this is sufficient to moderate the pH rise as the glass dissolves even at low flow rates.

The calculations shown in Table VI also assist in predicting the compositions of alteration products formed in or on the surface. Neglecting minor species, the surface composition obtained in the case of TDS-131 glass roughly corresponds to  $(Na, Li, Cs)(Fe, Al)Si_2O_6$ . Two frequently cited alteration phases, *aegirine* (aegirine)<sup>10</sup> and *analcime*<sup>12</sup> fall within this composition range. In the case of DWRG the ratio between (Fe, Al) and Si is very similar to the ratio obtained in the case of TDS-131 but the concentration of alkali metal ions is much lower, indicating a more clay-like character of the surface solid. It should be emphasized that the altered surface compositions calculated in Table VI refer only to long contact times, i.e., to slow flow rates. At high flow rates<sup>8</sup> not only boron and the alkalis but also Si and Al dissolve congruently, and only very insoluble species such as Fe, Mn and Ni stay behind (probably as oxides rather than silicates) due to the lower degree of saturation and the lower pH of the corresponding leachates.

#### CONCLUSIONS

The results described above demonstrate that relatively minor changes in glass composition can cause very large differences in the pathway and

rates of corrosion. A similar conclusion has been reported in studies of TDS-131 glass doped with very small amounts of simulated fission products and actinides.<sup>13</sup> The large effects of glass composition are due to the fact that several mechanisms are involved in the corrosion process.<sup>14</sup> A small increase in the amount of alkalis and a corresponding small decrease in silica content can lead to a large pH excursion. This results in a large increase in the extent of the range of conditions where matrix dissolution is the controlling mechanism and to a suppression of the onset of saturation, which limits the concentration of leached species and slows down the net loss of material from the glass.

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Acknowledgement

This study was supported in part by the Materials Characterization Center under Contract No. BA-2832AV.

TABLE I

Composition of Nuclear Waste Borosilicate Glasses

	DWRG		SRL TDS-131	
SiO <sub>2</sub>	51.6	49.0	43.9	44.1
Fe <sub>2</sub> O <sub>3</sub>	10.1	10.05	13.6	14.4
Na <sub>2</sub> O	7.7	9.20	12.1	13.7
B <sub>2</sub> O <sub>3</sub>	7.3	6.77	9.9	10.4
Al <sub>2</sub> O <sub>3</sub>	5.5	6.58	3.7	3.4
Li <sub>2</sub> O	4.1	4.85	3.7	4.0
MnO	3.4	3.09	4.5	4.1
U <sub>3</sub> O <sub>8</sub>	2.8	2.61		
NiO	2.1	2.01	2.3	1.7
CaO	1.9	1.95	0.8	1.2
ZrO <sub>2</sub>	1.0	0.94	0.3	0.4
MgO	0.8	0.74	1.4	1.4
SrO	0.5	0.46	0.9 <sup>a</sup>	
Cs <sub>2</sub> O	0.4	0.31	0.7 <sup>a</sup>	
Cr <sub>2</sub> O <sub>3</sub>	0.3	0.06		
P <sub>2</sub> O <sub>5</sub>	0.2			
TiO <sub>2</sub>	0.1	0.11	0.8	0.7
CeO <sub>2</sub>	0.04		1.0 <sup>a</sup>	
K <sub>2</sub> O	0.04			
Nd <sub>2</sub> O <sub>3</sub>	0.03			
ZnO	0.02	0.01		
CuO	0.01	0.02		
CoO	0.01			
La <sub>2</sub> O <sub>3</sub>			0.3	0.4
SO <sub>3</sub>			0.1	0.1
Ref.	3	4	5	6

<sup>a</sup> SRL TDS-131 glass supplied to The Catholic University of America was intentionally doped with Ce, Sr and Cs for leach studies.

TABLE II

Solubility Tests on Defense Waste Glass  
10 g of -60 +200 mesh powder in de-ionized water,  
70°C, 360 days

	Final leachate concentration, mg/L		Total dissolved fraction,	
	TDS-131	DWRG	TDS-131	DWRG
B	8578	106	93.10	0.76
Na	15662	233	59.55	0.61
Li	1137	137	22.93	1.21
U	n.p.	82.7	n.p.	0.43
Cs	56.4	4.66	3.23	0.16
Si	1754	377	3.16	0.25
Sr	7.48	2.12	0.28	0.06
Ca	2.97	27.5	0.16	0.22
Al	2.07	57.8	0.04	0.25
Mn	3.42	43.8	0.03	0.32
Ti	0.28	2.34	0.02	0.38
Fe	4.74	113	0.02	0.20
pH	12.42	10.67		
Final Volume, mL	30	14		

n.p. = not present in specimen

TABLE III

Effects of Glass and Leachant Compositions on the Results of Flow Tests at 70°C  
Normalized Leach Rates x 10<sup>2</sup>, g·m<sup>-2</sup>·d<sup>-1</sup>

Glass Leachant	SRL TDS-131		DWRG
	DI water	DI water	DI water
Flow rate, m/yr	10.7	1.99	10.8
pH	10.0	10.7	9.65
Si	10.5	7.1	6.17
B	17.5	23.2	8.78
Na	21.9	18.7	7.84
Cs	12.5	6.9	9.32
Li	16.4	21.4	9.37
Ca	<0.1	<0.2	2.35
Sr	0.1	<0.1	0.35
Al	6.7	0.3	5.55
Fe	0.06	0.11	0.02
U			0.92
Glass Leachant	DWRG		Ground-water
	DI water	Ground-water	Ground-water
Flow rate, m/yr	2.42	12.2	1.75
pH	9.70	9.57	9.53
Si	1.85	0.80	0.12
B	2.53	1.64	0.26
Na	2.50		
Cs	3.09	2.44	0.34
Li	2.50	2.37	0.39
Ca	0.24		
Sr	0.13	1.39	0.36
Al	1.45	0.65	0.18
Fe	0.10	0.10	0.005
Cr	0.36	<0.01	<0.01
Mg	<0.01	<0.01	<0.01
U	0.67	1.05	0.25

TABLE IV

Comparison between TDS-131 and DWRG  
Leach Data, Modified IAEA, 90°C

Leach Rates,  $\text{g}\cdot\text{m}^{-2}\cdot\text{d}^{-1}$

	TDS-131		
	30 d	120 d	360 d
Si	0.82	0.44	0.53
B	1.09	0.54	0.70
Na	1.14	0.59	0.75
Li	n.d.	n.d.	n.d.
Cs	1.02	0.59	0.48
Sr	0.079	0.031	0.057
Ca	0.084	0.053	0.079
Al	0.73	0.30	0.41
Fe	<0.001	<0.001	0.001
U	n.p.	n.p.	n.p.
	DWRG		
	30 d	120 d	360 d
Si	0.67	0.30	0.25
B	0.73	0.34	0.34
Na	0.80	0.35	0.47
Li	0.81	0.41	0.37
Cs	0.86	0.53	0.40
Sr	0.38	0.090	0.072
Ca	0.33	0.044	0.092
Al	0.60	0.25	0.21
Fe	<0.002	<0.001	<0.001
U	0.040	0.031	0.032

n.d. = not determined

n.p. = not present in specimen

TABLE V

Results of Dynamic Leach Tests  
on DWRG, 70°C

Leachant	DI water	0.05 M NaOH	0.2 M NaOH
pH	9.33	12.7	13.3
Flow rate, m/yr	2.4	2.4	2.4
Normalized leach rate, $\text{g}\cdot\text{m}^{-2}\cdot\text{d}^{-1}\times 100$			
Si	1.8	14	22
B	2.7	24	37
P	0.76	8.5	12
Li	1.8	25	37
Cs	4.2	24	45
Ca	1.0	0.62	1.4
Sr	0.21	0.088	0.30
Al	1.8	14	25
Mn	0.096	0.014	0.041
Ti	0.084	0.35	1.1
Fe	0.16	0.089	0.27
Ce	<0.05	14	37
U	0.36	1.7	1.4

TABLE VI

Molar Compositions of the Glass and of the  
Saturated Leachant (Relative to Boron) and  
Resulting Surface Compositions for TDS-131 and DWRG

Component	SRL TDS-131			
	Original glass $\text{X}^{\text{g}}$	$\text{X}^{\text{g}}/\text{X}^{\text{B}}$	Saturated leachate $\text{X}^{\text{L}}/\text{X}^{\text{B}}$	Left behind $\text{X}^{\text{S}}/\text{X}^{\text{g}}$
SiO <sub>2</sub>	0.356	2.55	0.5	2.05
1/2 B <sub>2</sub> O <sub>3</sub>	0.137	1.0	1.0	0.0
1/2 Na <sub>2</sub> O	0.195	1.35	1.05	0.3
1/2 Li <sub>2</sub> O	0.122	0.9	0.6	0.3
1/2 Fe <sub>2</sub> O <sub>3</sub>	0.088	0.6	0.0	0.6
1/2 Al <sub>2</sub> O <sub>3</sub>	0.036	0.25	0.0	0.25
MgO	0.017	0.1	0.0	0.1
MnO	0.024	0.2	0.0	0.2
NiO <sup>2</sup>	0.019	0.15	0.0	0.15
CaO	0.007	0.05	0.0	0.05
SrO	0.004	0.05	0.0	0.05
TiO <sub>2</sub>	0.005	0.05	0.0	0.05
ZrO <sub>2</sub>	0.001			
Component	DWRG			
	Original glass $\text{X}^{\text{g}}$	$\text{X}^{\text{g}}/\text{X}^{\text{B}}$	Saturated leachate $\text{X}^{\text{L}}/\text{X}^{\text{B}}$	Left behind $\text{X}^{\text{S}}/\text{X}^{\text{g}}$
SiO <sub>2</sub>	0.458	4.1	2.3	1.8
1/2 B <sub>2</sub> O <sub>3</sub>	0.111	1.0	1.0	0.0
1/2 Na <sub>2</sub> O	0.137	1.2	1.1	0.1
1/2 Li <sub>2</sub> O	0.142	1.3	1.2	0.1
1/2 Fe <sub>2</sub> O <sub>3</sub>	0.068	0.6	0.05	0.55
1/2 Al <sub>2</sub> O <sub>3</sub>	0.058	0.5	0.2	0.3
MgO	0.005	0.1	0.0	0.01
MnO	0.021	0.2	0.0	0.2
NiO <sup>2</sup>	0.014	0.15	0.0	0.15
CaO	0.017	0.15	0.0	0.15
SrO	0.002	0.02	0.0	0.02
TiO <sub>2</sub>	0.001			
ZrO <sub>2</sub>	0.004	0.05	0.0	0.05
1/3 U <sub>3</sub> O <sub>8</sub>	0.005	0.05	0.0	0.05