LEACHING OF BOROSILICATE GLASSES INCORPORATING SIMULATED HIGH-LEVEL RADIOACTIVE WASTES USING DRAFT ASTM PROCEDURE

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ABSTRACT

The results of three independent experimental runs of aqueous leaching tests conducted on four borosilicate glasses, namely SRL 202-P, SRL 202-G, ARM-1, and SRM-623, are presented. The main objectives of the experimental investigation were to compare the aqueous leaching test protocol for vitrified (glass) wasteforms with other laboratories involved in developing and testing wasteforms for high-level radioactive wastes (HLW), and to verify the sensitivity of the test [draft American Society for Testing and Materials leaching test called the Product Consistency Test] in discriminating between two borosilicate glasses based on the same frit but with slightly different composition of the incorporated simulated HLW. The leaching tests were conducted in deionized water, at 90°C, for a period of 7 days. The mean values of the elements in the leachant ranged from 3 to 62 $m \cdot gL^{-1}$, with the exception of silicon, the predominant matrix element. The silicon release ranged from 39 to 101 $m \cdot gL^{-1}$. The final pH values for the leachant ranged from 8.7 to 10.6. The difference in the elemental release values were much more significant between the different glasses as compared to sample-to-sample variations within the same type of glass. From the results, it is concluded that (i) the precision of the test data was adequate to discriminate between two similar borosilicate waste glasses, based on the same frit but with slightly different waste composition, (ii) the durability rank ordering of the four glasses tested matched that of the round robin conducted earlier among six independent laboratories, and (iii) the laboratory protocols used for the leaching tests are consistent with those used by the other laboratories that participated in the earlier round robin.

INTRODUCTION

The results of three experimental runs of aqueous leaching tests conducted on four borosilicate glasses, namely SRL 202-P, SRL 202-G, ARM-1, and SRM-623, are presented in the paper. The SRL glasses were developed by the Savannah River Laboratory (SRL); the ARM glass was developed by the Pacific Northwest Laboratory (PNL); while the SRM glass is a reference borosilicate glass developed by the National Institute of Standards and Technology (NIST). Only nonradioactive chemical compounds were incorporated into the test glasses to simulate high-level radioactive waste (HLW). The results of the tests are compared with those of a round-robin test conducted earlier in which the participants were mostly from the U.S. Department of Energy (DOE) laboratories.

It is important to point out here that the draft American Society for Testing and Materials (ASTM) procedure provides a method for conducting a leaching test whereby the results of various laboratories and different lots can be compared. As such, it would serve a useful function as a short-term standardized leaching test for determining the variability in wasteforms which are likely to be fabricated over decades. Early detection of variability could then be investigated further to ascertain potential impact on performance using other tests. Therefore, the main function of such a test is to ensure product consistency from batch to batch in a manufacturing environment, and should not be perceived as a product acceptance test for a geological repository. The relationship between the elemental release obtained from the draft ASTM leaching test and long-term performance of vitrified wasteform in a geologic repository has not been established. As such, no conclusions can be drawn from the data, analyses, or interpretations presented in this paper regarding the performance of any of the four glasses tested under any repository-specific condition or their licensability under federal regulations as stated in Title 10 Code of Federal Regulations Part 60 (1).

MATERIAL, SAMPLE PREPARATION, AND LEACHING PROCEDURE

Glass Composition

Tests were conducted per draft American Society for Testing and Materials (ASTM) procedure, called the Product Consistency Test (PCT), for glass wasteforms incorporating simulated or radioactive HLW (2). The composition of the glasses used in the tests are shown in Table I. The SRL 202-P glass was formulated with a larger amount of potassium (twice the amount in SRL 202-G) to represent off-normal composition. The presence of potassium in the SRL glasses is used to simulate the precipitation hydrolysis product that will result from the in-tank precipitation of Cs-137. Analyses of the glass composition were conducted independently by SRL and PNL for the SRL 202-G and *SRL 202-P* glasses. The ARM-1 and SRM-623 glasses were analyzed by PNL only. As shown in the referenced table, the difference between the two independent analyses or the certified/nominal value and the analyses is less than 10 percent for major components of the glass (those greater than 1 wt.% of the total composition). For the minor components, such as NiO, K_2O, Na_2O, etc., the differences in the analyses are much higher, ranging up to 50 percent, e.g., for Cr_2O_3. The compositions of the glass samples used in the investigation reported here were not analyzed and are presumed to have the same general compositions as shown in Table I, since they were a subset of the lots used in the round-robin tests. The glasses used for preparing the test

TABLE I
Composition Analyses of the Glasses Tested in Weight Percent

Compound	SRL 202-G			SRL 202-P			ARM-1			SRM-623		
	SRL Anal.	PNL Mean Anal.	Diff. %	SRL Anal.	PNL Mean Anal.	Diff. %	Cert. Val.	PNL Mean Anal.	Diff. %	Cert. NIST Nom.	PNL Mean Anal.	Diff. %
Al_2O_3	4.40	4.59	+4.14	4.14	4.27	+3.04	5.59	5.78	+3.29	6.3	6.14	-2.61
B_2O_3	5.82	6.00	+3.00	8.25	8.49	+2.83	11.30	11.80	+4.24	10.7	10.10	-5.90
BaO	0.15	0.14	-7.14	0.20	0.18	-11.11	0.658	0.65	-1.23	2.2	2.00	-10.00
CaO	1.32	1.31	-0.76	1.32	1.30	-1.54	2.24	2.32	+3.45	0.7	0.69	-1.45
CeO_2	—	—	—	—	—	—	1.51	1.42	-6.33	—	—	—
Cr_2O_3	0.15	0.10	-50.00	0.15	0.10	-50.00	—	—	—	—	—	—
Cs_2O	n.d.*	0.03	—	n.d.*	0.13	—	1.17	1.08	-8.33	—	0.03	—
CuO	0.40	0.37	-8.11	0.67	0.64	-4.64	—	—	—	—	—	—
Dy_2O_3	—	—	—	—	—	—	—	0.02	—	—	—	—
Eu_2O_3	—	—	—	—	—	—	—	0.02	—	—	—	—
Fe_2O_3	12.53	12.03	-4.16	11.51	10.63	-8.28	—	0.05	—	—	0.09	—
K_2O	2.83	3.24	+12.65	4.82	6.06	+20.46	—	—	—	0.6	1.00	+40.00
La_2O_3	—	—	—	—	—	—	—	0.02	—	—	—	—
Li_2O	3.67	3.80	+3.42	3.29	3.41	+3.52	5.08	4.82	-5.39	—	—	—
MgO	3.30	3.13	-5.43	3.07	2.84	-8.10	—	—	—	—	—	—
MnO_2	—	0.02	—	—	0.01	—	—	0.01	—	—	—	—
MoO_3	—	—	—	—	—	—	1.66	1.91	+13.09	—	—	—
Na_2O	6.92	8.28	+16.43	7.52	9.13	+17.63	9.66	9.73	+0.72	6.4	6.46	+0.93
Nd_2O_3	—	—	—	—	—	—	5.96	5.52	-7.97	—	—	—
NiO	0.74	0.64	-15.63	0.78	0.66	-18.18	—	—	—	—	—	—
P_2O_5	—	—	—	—	—	—	0.65	—	—	—	—	—
RhO_2	—	—	—	—	—	—	—	—	—	—	—	—
RuO_2	—	—	—	—	—	—	—	—	—	—	—	—
SiO_2	56.63	53.97	-4.93	52.91	50.83	-4.09	46.50	45.60	-1.97	73.0	71.40	-2.24
SrO	—	0.01	—	—	0.01	—	0.453	0.47	+3.62	—	0.03	—
TiO_2	0.67	0.65	-3.08	1.22	1.16	-5.17	3.21	3.32	+3.31	—	0.02	—
Y_2O_3	—	—	—	—	—	—	—	—	—	—	—	—
ZnO	—	—	—	—	—	—	1.46	1.47	0	—	—	—
ZrO_2	—	0.03	—	—	0.03	—	1.80	1.87	+3.74	—	0.05	—

*Not determined.

samples were supplied by SRL in quantities of approximately 100 g each. The glasses SRL 202-G and 202-P were in the form of 4 to 10 pieces each, while ARM-1 and SRM-623 glasses were in much smaller pieces (several dozen to hundreds) ranging from fines to 0.5 cm and larger in diameter. Only pieces larger than 0.5 cm in diameter were used for preparing samples for leaching tests.

Specimen Preparation

The samples were prepared by crushing pieces of glass in a coffee-mill type grinder equipped with a tungsten carbide blade (to avoid contamination of the glass sample with iron from the standard steel blade typically supplied with such grinders). The procedure involved crushing approximately 20 to 25 g of glass for a few cycles of approximately 5 s duration each. The crushed glass was then placed in sieving equipment with a stack of sieves in the order, from top to bottom, of 70 mesh, 100 mesh, 200 mesh, 270 mesh, and 400 mesh. The sieves were vibrated and tapped with an electric device to fractionate the crushed glass. After about 15 min, the sieve assembly was dismantled, and the crushed glass in the -100 to +200 mesh fraction was transferred to a clean plastic container. This fraction of the crushed glass was then washed with deionized water followed by an alcohol wash using an ultrasonic cleaner. The washed glass was dried overnight in a 90°C forced-air oven. The washed and dried glass, ready for testing, was then transferred to a plastic bottle with a label identifying the glass type, the lot number, date of sample preparation, and the preparer's initials. Additional information related to the test equipment and sample preparation is available in a detailed report of the investigation (3).

Leaching Vessel Assembly

Leaching vessels of 45 mL capacity, fabricated from Type 304L unsensitized stainless steel, were used for the tests. The leaching vessels were cleaned using the draft ASTM procedure for new stainless steel vessels (2). Three sizes of glass specimen were used for the leaching tests. Test Run #1 used triplicate samples of only 1.5 g size, while Runs #2 and #3 used one specimen of 1.5 g, one of 2.5 g, and one of 4.0 g, representing triplicate specimens for each type of glass. The amount of deionized water leachate added to each leaching vessel was proportional to 10 mL for each gram of glass specimen used, as required by the procedure. In addition, three blanks (leaching vessel with deionized water but with no glass specimen) were used in each test run. The leaching vessel assemblies were prepared by closing the cylindrical vessel with a Type 304L stainless steel lid. The lid was equipped with a Teflon® washer and was tightly held to the leaching vessel with an independent threaded nut assembly to provide a steam-tight seal.

Test Procedure

Assembled leaching vessels were weighed. They were then hung vertically in a preheated forced-air oven maintained at 90°C. After 1 day, the leaching vessel assemblies were removed from the oven, cooled to room temperature, and weighed to check for mass loss due to any leaks, according to the procedure (2). The reweighed assemblies were reinserted in the oven for an additional 6 days exposure. They were then removed from the oven, cooled to room temperature, and weighed again for mass loss. (According to the procedure, the results from assemblies with greater mass loss than allowed are to be discarded). The assemblies were disassembled one at a time to extract the leachant for chemical analyses.

Leachant Extraction

The leachant was extracted and transferred to a clean plastic container using a syringe with a stainless steel hypodermic needle. A small quantity (approximately 3 mL) of the leachant was removed for measuring the pH of the solution. The remaining leachant was filtered through a 0.45-micron cellulose acetate filter to remove any glass particles. The filtered leachant was acidified using 1 percent HNO_3 solution in the ratio of one part leachant to 20 parts dilute HNO_3. The required number of splits of the solution were prepared and labeled with appropriate identification for the glass type (or blank) and the run number prior to submitting them for analyses. The chemical analyses of the solution were conducted for a number of elements, including aluminum, boron, iron, potassium, lithium, sodium, and silicon, using inductively coupled plasma (ICP) spectroscopy. The concentration of all these elements in the leachant are reported in Table II. However, the discussion in this paper is focused mainly on the concentrations of boron, lithium, sodium, potassium, and silicon, as these were the only elements on which the earlier round-robin investigation was based.

TEST DATA AND ANALYSES

A comparison of the test data from the present investigation and those from the earlier round robin is provided in Table II. The table shows the mean, standard deviation (SD), and percent relative standard deviation (%RSD) for the four borosilicate glasses tested. The round-robin data are based on input from all except one participating laboratory (4). The data from current investigation are reported separately for Run #1 and averaged for Runs #2 and #3, since elemental release values from Run #1 consistently are significantly lower than those from Runs #2 and #3. Run #1 data represents a mean for the six data points per glass type obtained by analyzing the leachant from triplicate samples (two independent analyses of leachant from each leaching vessel), while the data from Runs #2 and #3 are based on six independent solution analyses per glass type (triplicate samples of each glass type for each run). The smaller size of the specimens used in Run #1 does not appear to be the reason for the difference between the leachant concentrations of Run #1 and Runs #2 and #3. Although no definitive explanation for the systematically lower release values in Run #1 as compared to those in Runs #2 and #3 is available at this time, differences in the washing step of the crushed glass samples could be a contributing factor. Also, the possibility of an equipment or operator error in the analyses of the solutions from Run #1 exists. This could, however, be resolved by analyzing the archive leachants from Run #1.

The elemental release data for high-solubility elements, namely boron, potassium, lithium, and sodium, and the matrix element silicon, are shown in Figs. 1 through 5. For plotting purposes, all data are rounded off to the nearest $mg \cdot L^{-1}$ concentration. The round-robin data show mean concentration with error bars of one and two standard deviation, while the data from the current investigation are plotted as mean values with error bars of one standard deviation. Standard deviations of less than 1 $mg \cdot L^{-1}$ (<1ppm) have not been plotted. The mean values of the elements analyzed ranged between 3 and 62 $m \cdot gL^{-1}$, with the exception of silicon, the predominant matrix element. The values of silicon release ranged from 39 to 101 $mg \cdot L^{-1}$. The final pH values for the leachate ranged from 8.7 to 10.6. The pH data are shown in Fig. 6 to the nearest 0.1 pH unit along with error bars of two standard deviation for the round robin results and one standard deviation for the present investigation.

DISCUSSION

It is apparent, from the plots shown in Figs. 1 through 5, that the differences in the elemental release values were much more significant between the different glasses as compared to sample-to-sample variations within the same type of glass. On individual element release basis, for boron, as shown in Fig. 1, the data for Runs #2 and #3 are within one standard deviation for SRL 202-P, 202-G, and ARM-1 glasses. For potassium, as shown in Fig. 2, the results of Runs #2 and #3 are within one standard deviation for SRL 202-P and 202-G glasses (other glasses investigated did not contain potassium). For lithium, as shown in Fig. 3, the releases for Runs #2 and #3 are within two standard deviations of the round-robin data for all glasses except 202-G. For sodium, as shown in Fig. 4, the releases for Runs #2 and #3 are within two standard deviations of the round-robin mean for the glass types 202-P and ARM-1, and are statistically significantly different for SRL 202-G and SRM-623 glasses as shown by the release data which are slightly outside the two standard deviation range. For silicon, as shown in Fig. 5, for all glasses, data for Runs #2 and #3 are within one standard deviations of the round robin except for SRM-623 glass which are within two standard deviations. For SRL 202-P and SRL 202-G glasses, the mean silicon releases

TABLE II
Lechant Elemental Analyses and Final pH

Glass Type	Analyte	Mean[a]			Standard Deviatin[b]			% RDS[c]		
		Round Robin	Run #1	Runs #2 and #3	Round Robin	Run #1	Runs #2 and #3	Round Robin	Run #1	Runs #2 and #3
SRL 202-P	Al	3.508	4.219	5.632	0.288	0.203	0.834	8.22	4.81	14.8
	B	25.28	18.57	24.55	1.269	0.643	0.635	5.02	3.46	2.58
	Fe	4.069	3.669	9.780	1.043	0.640	3.851	25.6	17.4	39.3
	K	32.30	23.50	29.68	2.892	0.312	0.458	8.95	1.32	1.54
	Li	15.87	10.32	13.63	0.817	0.127	0.385	5.15	1.23	2.82
	Na	69.64	45.42	61.88	3.901	0.648	1.542	5.60	1.42	2.49
	Si	109.6	94.01	113.2	3.761	5.000	4.087	3.43	5.32	3.60
	pH	10.63	9.847	10.49	0.408	0.535	0.072	3.84	5.43	0.68
SRL 202-G	Al	3.872	4.391	5.024	0.343	0.247	0.546	8.86	5.62	10.8
	B	14.44	11.57	13.84	0.724	0.263	0.262	5.01	2.27	1.89
	Fe	3.836	3.870	6.891	0.968	0.881	2.328	25.2	22.7	33.7
	K	11.53	9.844	11.21	2.185	0.292	0.674	18.9	2.96	6.00
	Li	15.22	10.37	12.46	0.734	0.089	0.267	4.82	0.85	2.14
	Na	49.86	37.79	43.41	2.493	0.905	0.858	5.00	2.39	1.97
	Si	112.3	100.1	115.2	4.175	1.220	2.391	3.72	1.21	0.20
	pH	10.42	10.25	10.31	0.415	0.022	0.067	3.98	0.21	0.64
ARM-1	Al	4.652	4.742	4.452	0.468	0.166	0.353	10.0	3.50	7.92
	B	27.48	29.46	26.17	3.335	1.347	3.023	12.1	4.57	11.5
	Fe[d]	0.117	0.124	0.293	0.313	0.078	0.195	267	62.9	66.5
	K[d]	0.549	0.356	0.671	0.696	0.119	0.207	126	33.4	30.8
	Li	21.73	20.47	17.63	1.540	0.681	1.486	7.09	3.32	8.42
	Na	55.64	53.56	48.08	5.082	1.911	4.113	9.13	3.56	8.55
	Si	80.16	82.03	73.77	6.031	1.850	3.466	7.52	2.25	4.69
	pH	10.56	10.41	10.38	0.373	0.112	0.080	3.53	1.07	0.77
SRM-623[e]	Al	3.343	—	3.041	0.228	—	0.142	6.83	—	4.67
	B	7.050	—	4.876	0.609	—	0.097	8.64	—	1.99
	Fe[d]	0.056	—	0.240	0.104	—	0.103	184	—	42.9
	K[d]	0.600	—	0.334	0.665	—	0.336	110	—	100
	Li[d]	0.054	—	0.179	0.082	—	0.031	150	—	17.3
	Na	12.73	—	8.075	0.961	—	0.251	7.55	—	3.11
	Si	46.09	—	39.95	4.465	—	1.022	9.69	—	2.56
	pH	8.692	—	8.689	0.280	—	0.080	3.23	—	0.92

(a) Mean values of analyte concentrations, $mg \cdot L^{-1}$
(b) All standard deviations quantify the uncertainty in a single value, $mg \cdot L^{-1}$
(c) %RSD is obtained by dividing the corresponding standard deviation value by the mean and multiplying by 100.
(d) These elements were not present in the glass; therefore, the means and standard deviations characterize detection-limit noise.
(e) SRM-623 glass was not included in Run #1.

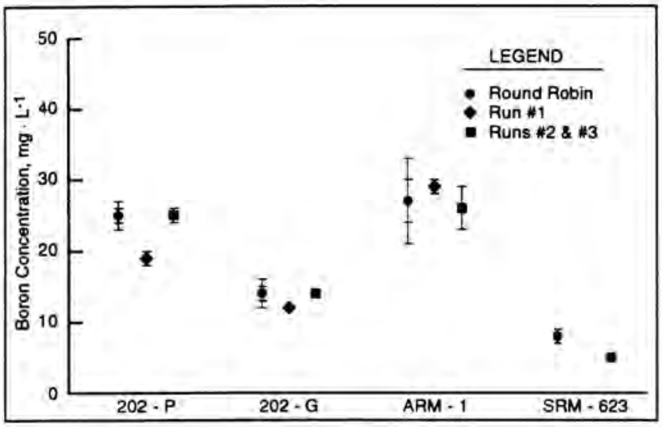

Fig. 1. Release of boron as a function of glass type.

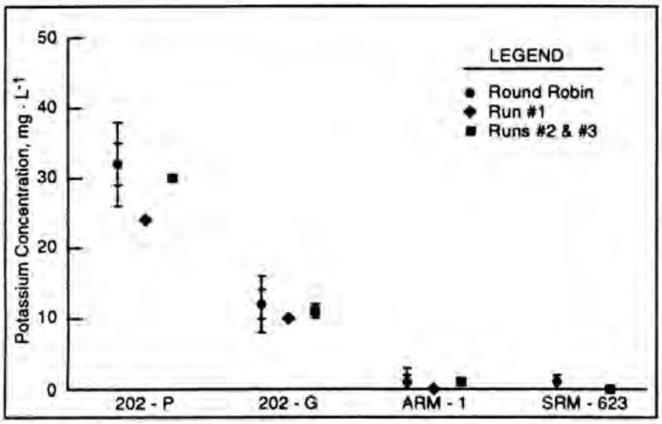

Fig. 2. Release of potassium as a function of glass type.

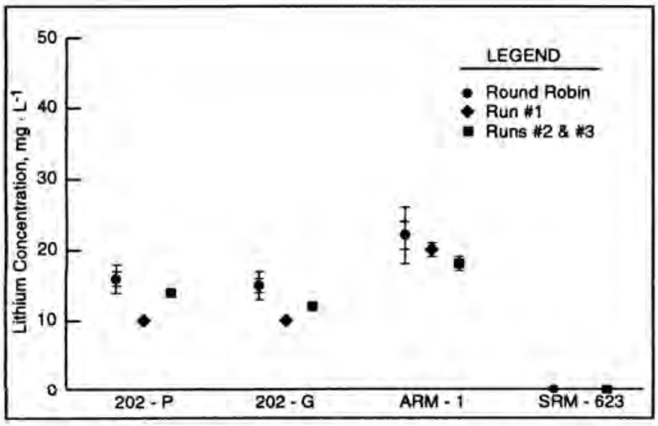

Fig. 3. Release of lithium as a function of glass type.

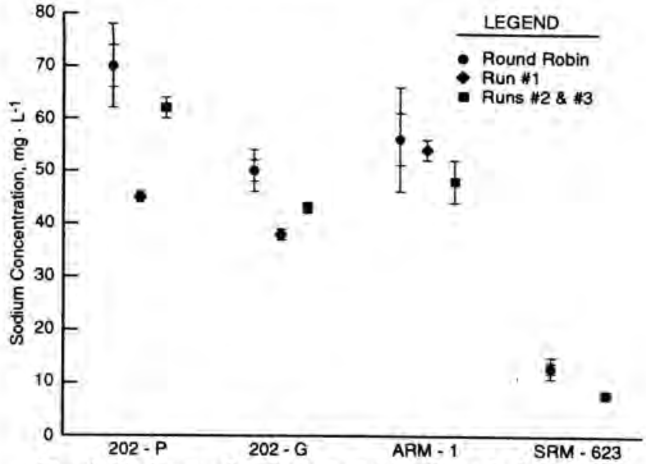

Fig. 4. Release of sodium as a function of glass type.

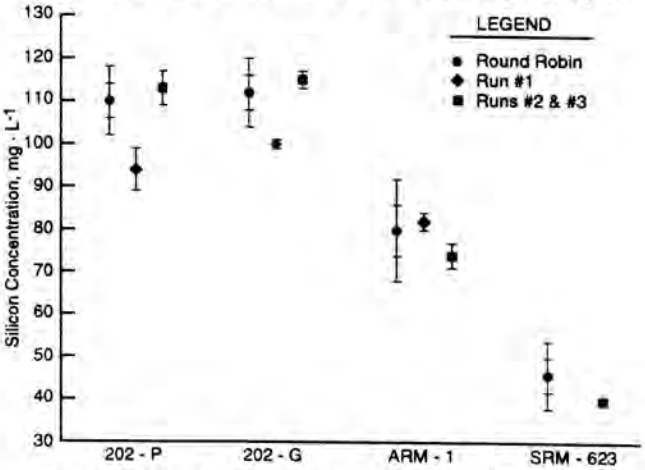

Fig. 5. Release of silicon as a function of glass type.

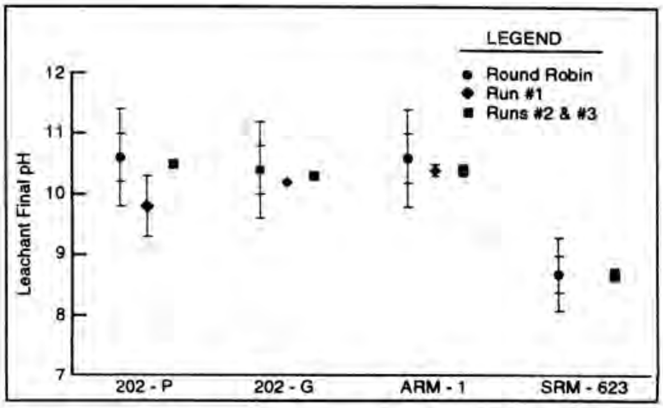

Fig. 6. Leachant final pH as a function of glass type.

for the investigation reported here (Runs #2 and #3) are slightly higher than the round-robin mean, while, for the other two glasses, namely ARM-1 and SRM-623, they are slightly lower. An examination of the data in Fig. 6 indicates that the final leachate pH values for Runs #2 and #3 are within one standard deviation of the round-robin results, while data for Run #1 are within one standard deviation of the round-robin results for all except the 202-P glass.

The cumulative releases for groups of elements are shown in Fig. 7. The first group shows cumulative releases for [B + Li + Na] for the glasses tested. The second group shows releases for the [B + Li + Na + Si] combination, while the third and the fourth groups show elemental releases for the combinations [B + K + Li + Na] and [B + K + Li + Na + Si], respectively. The data in Fig. 7 indicate that the results of the round-robin tests and the average of Runs #2 and #3 are generally within 10 percent of each other. Based on the data shown in Fig. 7, a rank ordering of the durability of the four glasses tested is shown in Table III. The results show that rank ordering using Runs #2 and #3 averaged data agrees with that

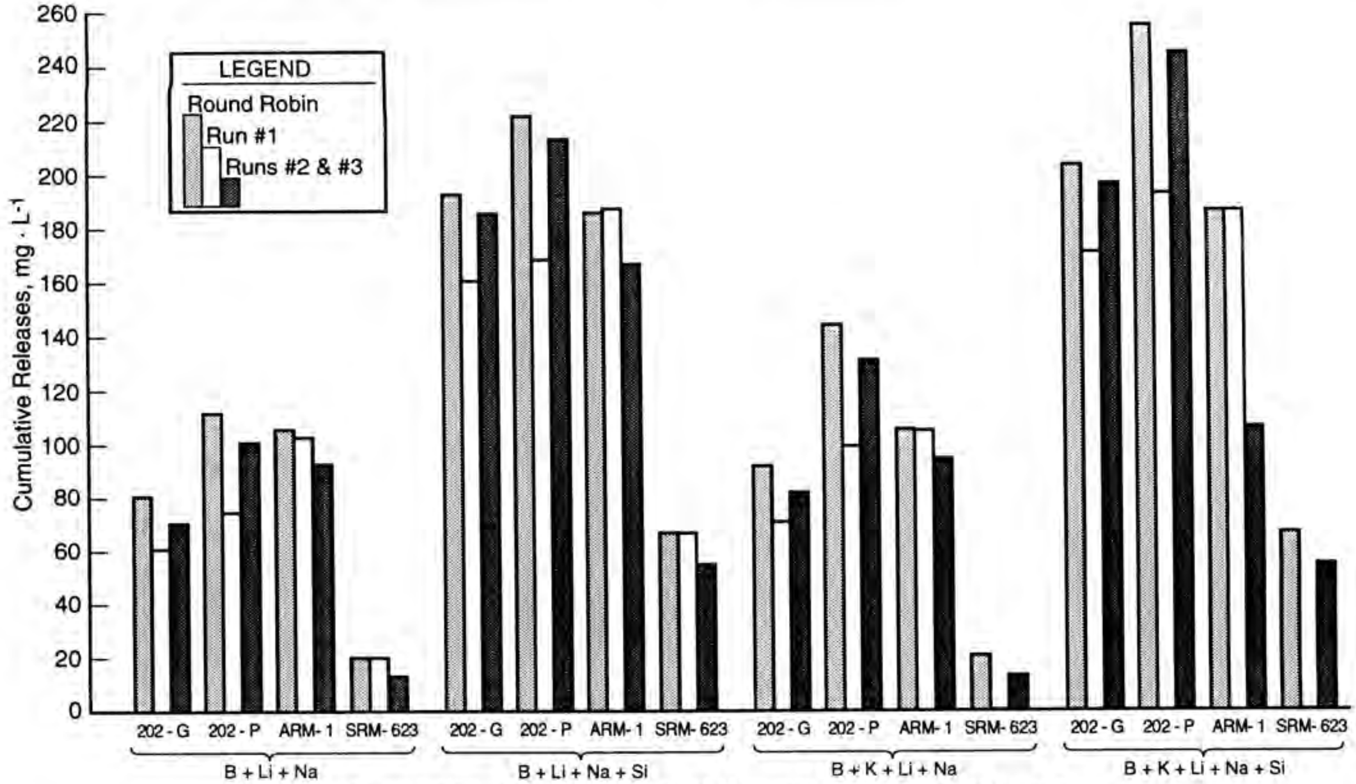

Fig. 7. Cumulative releases of groups of elements as a function of glass type.

of the round-robin tests, while the rank ordering based on Run #1 data agrees with the round robin for the majority of the glasses with the main difference being the interchange of type 202-P and ARM-1 glass rankings.

The data from this investigation show consistently lower individual and cumulative elemental releases as compared to those for the round-robin tests. A significant cause leading to the observed differences is attributed to the change in the procedure for preparing samples. (The procedure was revised, after the round robin tests, to include a step for washing crushed glass samples prior to leach testing. This step leads to reduced sample-to-sample variability in the surface area for the same mass of glass. As a result, lower but more consistent release values are obtained in both intra- and inter-laboratory tests). The round-robin test data reported here are based on samples tested in an unwashed condition (according to the procedure current at the time the round robin was conducted (5), while the current investigation used samples in a washed condition. Information from the participants of the earlier round robin, and limited experimental data (6), indicate that an increase of 10 to 20 percent in the release of the high-solubility elements could be expected from unwashed samples. The precise difference in the elemental release from washed versus unwashed samples, among other factors, will depend upon the amount of fines which, in turn, has been observed to be influenced by the composition of the frit and waste, and waste loading of the glass. The variability in the glass crushing step due to equipment differences or operator handling, within the allowable draft ASTM specifications, also contributes to the amount of fines generated and the morphology of the particles in the crushed glass. If the data are adjusted by increasing the elemental releases by 10 to 20 percent to account for the washing away of the fines, the differences between the results of the present investigation and the round-robin tests would be quite small. Such a match, based on adjusted release values, would be considered to be well within the experimental error associated with aqueous leaching experiments on vitrified wasteforms.

CONCLUSIONS

It is concluded that the sample preparation and leaching protocols used for conducting the leaching tests are consistent with the other laboratories that performed the earlier round-robin tests. This conclusion is supported by a ranking of results taken from the round robin and the data from the present investigation. Furthermore, the results of this investigation agree with those of the round robin in showing that SRL 202-P is less durable than SRL 202-G. These two glasses were specially formulated to investigate the sensitivity of the draft ASTM leaching procedure in discriminating between glasses of slightly different leachability.

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TABLE III
Ranking of Glasses Tested Based on Cumulative Releases for Groups of Elements

Glass Type	B + Li + Na			B + Li + Na + Si			B + K + Li + Na			B + K + Li + Na + Si		
	Round Robin	Run #1	Runs #2 and #3	Round Robin	Run #1	Runs #2 and #3	Round Robin	Run #1	Runs #2 and #3	Round Robin	Run #1	Runs #2 and #3
202-P	A	B	A	A	B	A	A	A	A	A	A	A
202-G	C	C	C	B	C	B	B	B	B	B	B	B
ARM-1*	B	A	B	C	A	C	—	—	—	—	—	—
SRM-623**	D	—	D	D	—	D	—	—	—	—	—	—

*No potassium.
**No potassium or lithium.
A → Least durable (most leachable)
B → Second least durable
C → Third least durable
D → Most durable (least leachable)

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