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PRODUCT CONSISTENCY LEACH TESTS OF SAVANNAH RIVER SITE RADIOACTIVE WASTE GLASSES

by

N. E. Bibler and J. K. Bates*

Westinghouse Savannah River Company
Savannah River Site
Aiken, South Carolina 29802

*Argonne National Laboratory
9700 S. Cass Ave.
Argonne, Illinois 60439

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ABSTRACT

The Product Consistency Test (PCT) is a glass leach test that was developed at the Savannah River Site (SRS) to routinely confirm the durability of nuclear waste glasses that will be produced in the Defense Waste Processing Facility. The PCT is a 7 day, crushed glass leach test in deionized water at 90°C. Final leachates are filtered and acidified prior to analysis. To demonstrate the reproducibility of the PCT when performed remotely, SRS and Argonne National Laboratory have performed the PCT on samples of two radioactive glasses. The tests were also performed to compare the releases of the radionuclides with the major nonradioactive glass components and to determine if radiation from the glass was affecting the results of the PCT.

The test was performed in triplicate at each laboratory. For the major soluble elements, B, Li, Na, and Si, in the glass, each investigator obtained relative precisions in the range 2-5% in the triplicate tests. This range indicates good precision for the PCT when performed remotely with master slave manipulators in a shielded cell environment. When the results of the two laboratories were compared to each other, the agreement was within 20%. Normalized concentrations for the nonradioactive and radioactive elements in the PCT leachates measured at both facilities indicated that the radionuclides were released from the glass slower than the major soluble elements in the glass. For

both laboratories, the normalized releases for both glasses were in the general order Li~B~Na>Si>Cs-137>Sb-125>Sr-90. The normalized releases for the major soluble elements and the final pH values in the tests with radioactive glass are consistent with those for nonradioactive glasses with similar compositions. This indicates that there was no significant effect of radiation on the results of the PCT.

INTRODUCTION

A crushed glass leach test, the Product Consistency Test (PCT) [1,2], has been developed at the Savannah River Site (SRS) to confirm the durability of the nuclear waste glasses that will be produced by the Defense Waste Processing Facility (DWPF). As the name of the test implies, the primary purpose of the PCT is to measure the consistency of the durability of the radioactive glasses that the DWPF will produce during its several years of operation. Durability of the glasses will be determined by measuring the amounts of soluble elements such as B, Li, Na, K, and Si that are released from the glass to deionized water when the glasses are leached following the PCT procedure.

At SRS, the DWPF will solidify into solid borosilicate glass the ~50 million gallons of caustic slurries of high-level radioactive waste. These wastes are currently stored in double walled underground tanks. Eventually, this glass will be permanently emplaced in a geologic repository. One of the specifications that the glass will have to meet to be acceptable to the repository is confirmation that the radionuclide release property of the glass (its durability in water) has been controlled during production in the DWPF [3]. Control of the radionuclide release property of the glass will be accomplished by carefully controlling the glass composition [4]. Confirmation of this control will be demonstrated by performing the PCT on samples of actual DWPF glass that will be periodically collected during the DWPF campaign [5].

The development of the PCT utilized both an intralaboratory round robin [1] and an interlaboratory round robin [6] with nonradioactive simulated waste glasses. The emphasis during this test development was on generating a methodology that could be routinely performed and yield results that could evaluate comparable durabilities. The main purpose of those two round robins was to determine the precision of the PCT when performed by a single investigator and when performed by different investigators in different laboratories. Both round robins indicated that for a single investigator a relative precision of 3-5% could be expected in triplicate tests. For different investigators and different laboratories, the relative precision decreased to 10-15%.

This paper describes the results of another interlaboratory application of the PCT. In this application, two laboratories, Argonne National Laboratory (ANL) and SRS, performed the PCT on two radioactive glasses containing actual radioactive sludge from the SRS storage tanks. At each laboratory, the PCT was performed remotely in a shielded cell environment using master slave manipulators exactly as it will be performed for the actual high-level radioactive glasses produced by the DWPF. The main purposes of this interlaboratory comparison were to determine whether the methodology that had been developed for routine hands-on operation was directly transferable to remote operation, and to determine the precision of the PCT when performed remotely. Other purposes were to compare the normalized releases for the radionuclides with the major nonradioactive elements in the glasses and to determine if radiation from the glass affected the results of the test.

GLASS COMPOSITIONS AND PREPARATION

The concentrations of the major nonradioactive and radioactive components in the two glasses used in this study are given in Table 1. The compositions of the glasses are different because they were prepared from different wastes and from different frits. The 165/42 glass was prepared using Frit 165 and sludge from SRS Tank 42. This glass contained nominally 28 wt% sludge oxides and 72 wt% frit oxides and was used in two earlier studies measuring the leachability of radioactive glass [7,8]. Except for Cs-137, the radionuclide concentrations in this glass are close to those expected in actual DWPF glass [9]. The radiation dose rate from this glass was estimated to be in the approximately 2000 rad/hour using a solid state dosimeter. This dose rate is primarily due to the gamma and beta radiation from Cs-137, Sr-90, and their daughters. The Cs-137 concentration in the 165/42 glass is lower than that expected in the DWPF glass because the actual radioactive waste supernate, which contains ~99% of the Cs-137, was not processed and put into this glass. The 200R glass was prepared using Frit 200 and a mixture of radioactive sludges from SRS Tanks 8 and 12. The final glass contained nominally 28 wt% sludge oxides, 64 wt% frit, and 8 wt% simulated tetraphenylborate precipitate hydrolysis product. The precipitate hydrolysis product simulates the waste that will result from the in-tank precipitation of Cs-137 to remove it from the supernate and put it into the glass. This accounts for the presence of K in this glass. The radioactive composition of 200R is significantly lower than 165/42 glass because 200R glass also contained some simulated sludge which diluted the radionuclides.

Both glasses were prepared in separate campaigns in the Shielded Cell Facility at SRS using a slurry-fed, remotely operated, joule heated melter. In these campaigns, a slurry of frit and waste was fed to the melter at 1150°C. The molten glass was poured from the melter into 500 mL stainless steel cans. Each campaign produced several cans of glass. For this interlaboratory comparison, an appropriate can was selected for each glass and cut into 0.5" slices. A single slice from each can was then chosen for the PCT at SRS and ANL. Leach samples were obtained by knocking pieces of the glass out of these two slices. A portion of these pieces was sent to ANL and a portion was retained at SRS.

The glass compositions were determined at SRS by HCl/HF and Na_2O_2 dissolutions of pulverized pieces of glass from each chosen slice. The resulting solutions were analyzed by ICP for nonradioactive elements and by appropriate counting techniques for radioactive elements. The results presented in Table 1 are averages of dissolutions of twelve to eighteen samples of each glass. For the major oxides (those >1 wt%) and radionuclides (Cs-137 and SR-90), the relative precision was 6% or better. For the minor components such as NiO, Sb-125, and the alpha emitters, the relative precision was 10 t0 20%. The actual glass samples sent to ANL were not analyzed; however, since these samples were taken from the same slices as the glasses analyzed at SRS, they were presumed to have the same compositions as those determined at SRS and presented in Table 1.

PCT PROCEDURES FOR RADIOACTIVE GLASSES

The PCT is a seven day leach test at 90°C in distilled, deionized, ASTM Type I water using washed, crushed (100-200 mesh) glass. Ten milliliters of water are used for each gram of glass leached. Because of the intense radiation from the glass, all procedures that involved the glass had to be performed remotely in a shielded cell facility with master slave manipulators. After the leach testing, the leachates were not very radioactive and could be safely transferred from the shielded cell to a radiochemical hood where they could be handled directly. The various remote and direct operations are summarized in Table 2. Details of the procedure are available elsewhere [1,2], and only pertinent items will be discussed here.

For radioactive glasses, the PCT is performed in 22mL stainless steel Parr vessels. A weighed amount of glass (1.5 -1.7 grams) is placed into each vessel. Based on the above ratio, the exact amount of ASTM water is added to each vessel. The vessel is then sealed, weighed, and put into a 90°C oven whose temperature is carefully monitored. After seven days the vessel is cooled, weighed, and opened. The leachate is carefully decanted into a clean plastic vessel and transferred to a radiochemical hood. Here the leachate is filtered to ensure removal of all the crushed glass particles. The pH of an aliquot of the leachate is then measured and the leachate is sampled for anion analysis. The leachate is then acidified, and analyzed for radioactive and nonradioactive elements. For each glass, triplicate tests are performed along with duplicate blanks, and all the tests are performed simultaneously in the same oven.

To determine the amount of leached elements that may have sorbed onto the steel leach vessel itself, the vessels were leached in the shielded cell with dilute HNO_3. Before leaching, the vessel was inverted and briefly rinsed with a stream of water to ensure that all the crushed glass was removed from the vessel. Nitric acid was then added and the vessel was heated to 90°C for 16 hours. The rinses were then transferred to the radiochemical hood and analyzed.

RESULTS AND DISCUSSION

Comparison of SRS and ANL Results

The average of the results of triplicate tests at SRS and ANL for 200R glass and 165/42 glass are shown in Tables 3 and 4, respectively. For both glasses, the concentrations of elements, including radionuclides, in the blank tests where glass was absent were negligible compared to the concentrations in the leachates when glass was present. Analysis of the rinses for nonradioactive elements indicated that for the soluble elements, the amounts sorbed on the vessel walls were less than 1% of that in solution except for Si which had 2% absorbed. For Al, the amount on the walls was 5% of that in solution.

For both glasses and for each investigator, the relative standard deviations for concentrations of the major soluble elements (B, Li, Na, K, and Si for 200R glass, and B, Li, Na, and Si for 165/42 glass) ranged from 0.7 to 5.3%. The average of all these relative standard deviations is only 2.6 ± 1.4% indicating very good precision. For Cs-137. the major soluble radionuclide, the relative standard deviations ranged from 1 to 6% again indicating good precision. These results confirm that good precision can be obtained from the PCT even when it is performed remotely on radioactive glass. Precisions for the other elements and radionuclides, Al, Sr-90, Pu-238, and Cm-244 were poorer probably because the concentrations of these elements can be affected by solubility constraints or by localized reactions with the glass or the leach vessel itself.

Comparison of the results of the two laboratories for the major nonradioactive soluble elements indicates that the SRS results are consistently slightly higher than the ANL results, especially those for 165/42 glass. This is also true for the final pH values. Even though the SRS results are higher, the two data sets differ by only ~20% or less. When both data sets are considered a single set, the interlaboratory relative standard deviation for the data for the major soluble elements ranges from only 4 to 12%. This range is comparable to that obtained in the earlier interlaboratory round robin [6] using nonradioactive simulated waste glass.

The Student's T test can be applied to these results to determine whether the differences between the results for the two laboratories can be attributed to random errors or whether a systematic difference exists between the two data sets. For the 200R glass, the T test indicates to the 95% confidence level that the results for Li and K agree for the two data sets within the precision for each set. The results for B, Na, and Si are just outside agreement. For the 165/42 glass the Student's T test indicates that a systematic difference exists between the two data sets. The higher final pH values of the SRS tests is consistent with the more rapid leaching being observed at SRS. The exact reason for the differences in the elemental releases is not known at this time. Minor variances in the procedures used for crushing, sieving, and washing the glass at the two laboratories as well as minor temperature differences in the ovens could result in the variances observed. Both of these potential sources of systematic error will be examined in future tests at SRS and ANL with other radioactive glasses.

The agreement for the radioactive elements between the two data sets is not nearly as good as for the nonradioactive elements. Again, the difference is greater with the 165/42 glass (~2X in some cases) and it appears that greater leaching occurred at SRS. For the 200R glass, the results for the gamma emitters Cs-137 and Sb-125 differ by 22 and 40%, respectively. The results for the alpha emitters and for Sr-90, a beta emitter, are in excellent agreement. Analysis of the HNO_3 rinses of the leach vessels indicated that negligible amounts of Cs-137, Sb-125, and Sr-90 had sorbed on the vessel, while significant amounts of Pu-238 and Cm-244 had sorbed. For Pu-238, nearly as much was on the vessel as in solution. This has been observed before [7,10].

For the 165/42 glass, the results for all the radionuclides for the two data sets differed by nominally 2X with SRS showing the faster leaching. This is much larger than the differences in the nonradioactive elements. The exact reason for the large differences in the leaching of the radionuclides is not known It may be that the glass leached at ANL had less radionuclide content than that at SRS. This appears unlikely, but in future tests, the composition of the glasses tested at each laboratory will be determined at that specific laboratory.

Normalized Concentrations for Radioactive and Nonradioactive Elements

The releases for each element from the glass can be normalized to the concentration of the element in the glass in order to compare how fast the various elements are released from the glass. This information is required to determine whether glass performance and quality can be adequately assessed by glass formers or whether the release of radionuclides must also be routinely meaured for DWPF glass during operation of the DWPF. Normalized concentrations are calculated by dividing the measured concentration of an element in the leachate by its respective concentration in the glass. The normalized concentrations can be considered the concentration of dissolved glass (grams of glass/liter) in the PCT leachate based on that particular element in the glass. Of course this concentration is only the true concentration if all the elements in the glass leach at the same rate, all are soluble in the leachate, and none precipitate or sorb on the leach vessel or on the glass itself. Because of the many different elements in nuclear waste glasses, this is never the case. However, the normalized concentrations of the major soluble elements are good measures of the maximum rates that elements can be dissolved or released from the glass.

The normalized concentrations for radioactive and nonradioactive elements for both radioactive glasses are presented in Table 5 based on the SRS results. Normalized concentrations based on the results at ANL are for the most part lower than the SRS results in proportion to the lower leachate concentrations measured at ANL (see Table 3 and 4). The results in Table 5 for the major soluble elements show that the glass prepared from Frit 165 is more durable than that prepared with Frit 200. This is expected based on the SiO_2 content of the glasses. For the 200R glass, it appears that B, Li, and Na are leaching nearly congruently. This has been observed with a nonradioactive simulated waste glass prepared with SRS Frit 131 [11]. With the 165 glass, data in Table 5 indicate that Li is leaching faster than either B or Na. This has been observed in other leach tests with 165 based glasses [12]. For the most part, K, Si, and Al have lower releases than B, Li, and Na. This order of leaching for the various elements is in agreement with results of leaching nonradioactive SRS glasses [10-12].

The data in Table 5 indicate that all the radionuclides, including Cs-137, leach slower than the major soluble nonradioactive elements in the glass. On this basis, the maximum radionuclide release rate from the glass can be obtained by measuring the normalized release rate for B or Li. This hypothesis will be tested by measuring other radionuclides in PCT tests with glasses containing actual SRS radioactive sludges and with glasses containing simulated nonradioactive sludge doped with specific radionuclides such as Tc-99 and other fission products in the SRS high-level wastes.

Effect of Radiation on the Leaching Process

The effect of radiation on the PCT results can be evaluated by comparing the PCT results for radioactive 200R and 165/42 glasses with results for nonradioactive glasses. Table 6 shows the normalized concentrations for the major elements in tests at SRS and ANL with the two radioactive glasses and two nonradioactive glasses. Data for the SRS 200 glass and the standard ARM-1 glass leached at SRS are given in Reference 1. The PCT was also performed on the standard ARM-1 glass at ANL as part of this study and as part of the earlier interlaboratory round robin [6]. The results in Table 6 are not expected to be in exact agreement, since the glasses have different compositions. The data do indicate for B, Li,and Na, the soluble elements, that as the SiO_2 content of the glasses increases (see the last row of Table 6) the glasses become more durable and leaching decreases. This trend has been observed for nuclear waste glasses as well as commercial and natural glasses [13]. Based on this, it appears that radiation from the radioactive glasses is not significantly affecting the results of the PCT. For Si, saturation effects may be affecting the results.

The effect of glass composition on normalized concentrations can best be evaluated by using the hydration thermodynamic model [13]. This model predicts that the logarithm of the normalized concentration for the soluble elements for a series of glasses should be a linear function of the calculated free energy of hydration [13]. This free energy of hydration is calculated from the composition of the glass and the final pH of the leach test [13]. Data obtained at SRS for the soluble elements B, Li, and Na for the two radioactive glasses and two nonradioactive glasses are in Figure 1 along with the corresponding data for the nonradioactive and two radioactive glasses leached at ANL. For each element, good linearity is obtained, again supporting the conclusion that radiation is not affecting the results of the PCT. The results for Si at both SRS and ANL do not fit the model. A possible reason for this is that relatively high Si concentrations (70-90 ppm) are reached in the PCT because of the high surface area of the crushed glass; consequently, saturation effects may affect the release of Si. In tests where monoliths of glass are leached, the Si concentrations are lower (nominally 40 ppm) and the Si results adhere to the model [13]. The slopes of the computer drawn lines in Figure 1 range from -0.11 to -0.22. These slopes are in reasonable agreement with that obtained when the model was applied to the results of leach tests with monolithic saamples where the slope was -0.22 [13]. The free energies of hydration calculated for the ANL results are offset from the SRS values by nominally +1 kcal/mole. This offset results from the lower pH values observed in the ANL tests. In the model, the calculated free energy of hydration is affected by the pH since the pH affects the dissociation of two major hydrated species resulting from the leaching of the glass. These species are silicic and boric acid.

CONCLUSIONS

The data obtained during this study involving the interlaboratory application of the PCT using radioactive glasses supports the following conclusions:

1. The relative precision for a single investigator for major soluble elements of the PCT when performed remotely with a radioactive glass can be expected to be in the range of 2-5%. This range is equal to that obtained for nonradioactive glasses where the PCT was performed directly [1,6].

2. The interlaboratory relative precision for the PCT when performed remotely ranges from 10 to 20% dependending on the glass composition. This range is similar to that observed when the PCT was first evaluated in a round robin

using hand-on operations [6]. In the present study, a systematic error is evident between the results of the two laboratories. Possible sources of this error will be investigated in future tests with radioactive glasses.

3. The normalized releases for B and Li are higher than the radionuclides Cs-137, Sr-90, Sb-125, and the alpha emitters (Cm-244 and Pu-238). This conclusion will be tested with other radionuclides. If this conclusion remains true for the other radionuclides in DWPF glass, then the maximum radionuclide release property of DWPF glass can be determined by measuring the release of B or Li.

4. The effect of radiation from the glass, on either the glass itself or on the leach solution, does not affect the results of the PCT. Future experiments with radioactive glasses will test this conclusion further.

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Table 1. Major Components of Radioactive 200R and 165/42 Glasses.

Nonradioactive Oxides (wt%)

Oxide	200R Glass	165 Glass	Oxide	200R Glass	165 Glass
B_2O_3	9.46	8.16	Al_2O_3	4.62	11.4
Li_2O	3.01	5.30	Fe_2O_3	12.3	6.74
Na_2O	13.29	12.6	CaO	1.47	0.44
K_2O	3.49	0.0	MgO	1.25	1.11
SiO_2	45.1	57.2	MnO_2	2.61	2.13
U_3O_8	0.4	0.17	NiO	0.53	0.51

Radionuclides (mCi/100 g glass)

Isotope	200R Glass	165 Glass
Cs-137	1.76	9.1
Sr-90	50.7	1090.
Sb-125	0.09	0.34
Eu-154	0.23	3.8
Eu-155	0.18	8.6
Alpha[a]	1.0	4.2

[a]Primarily Cm-244 and Pu-238.

Table 2. Shielded Cell and Radiochemical Hood Prodcedures for PCT with Radioactive Glass

Shielded Cell Procedures performed Remotely

- Crush, Grind, Sieve, Wash, and Dry Glass
- Weigh Vessel, Glass, Water, and Final Test Assembly
- Put in 90°C Oven and Monitor Temperature
- After 7 Days, Remove from Oven, Cool, and Weigh
- Decant Leachate to Clean Vessel and Transfer to Hood
- Remove Glass and Rinse Vessels

Radiochemical Hood Procedures performed Directly

- Filter Leachates (0.45 micron filter), Measure pH
- Submit for Anion Analysis
- Acidify and Submit for Caticn and Radionuclide Analyses
- Filter and Analyze Rinses

Table 3. Average Concentrations, Standard Deviations, and Final pH Values Measured in Triplicate PCT Tests of Radioactive 200R Glass

Nonradioactive Elements (ppm)	SRS Results[a]	ANL Results[a]
B	33.8 ± 1.8	28 ± 0.6
Li	13.6 ± 0.2	13 ± 0.6
Na	97.3 ± 3.5	92 ± 1.0
K	15.5 ± 0.4	17 ± 0.6
Si	98.1 ± 1.1	84 ± 0.6
Al	7.4 ± 1.0	7.1 ± 0.4
Radionuclides (nCi/mL)[a]		
Cs-137	3.5 ± 0.034	2.8 ± 0.1
Sr-90	1.89 ± 0.08	1.83 ± 0.11
Alpha[c]	0.98 ± 0.04	1.0 ± 0.1
Sb-125	0.53 ± 0.01	0.35 ± 0.01
Final pH[b]	10.5	9.9

[a]At both laboratories, the concentrations of nonradioactive and radioactive elements in the blanks were negligible.

[b]Primarily Cm-244.

[c]Initial pH at SRS = 7.3, at ANL, 5.6. Final pH of blanks, 7.5 at SRS and 5.0 at ANL.

Table 4. Average Concentrations, Standard Deviations, and Final pH Values Measured in Triplicate PCT Tests of Radioactive 165/42 Glass

Nonradioactive Elements (ppm)	SRS Results[a]	ANL Results[a]
B	10.4 ± 0.3	8.5 ± 0.3
Li	13.1 ± 0.4	11 ± 0.0
Na	32.1 ± 0.9	27 ± 0.6
Si	87.1 ± 2.8	71 ± 2.3
Al	19.6 ± 0.6	22 ± 0.6
Radionuclides (nCi/mL)[a]		
Cs-137	11.0 ± 0.4	5.3 ± 0.1
Sr-90	210 ± 17	91 ± 7
Alpha[b]	5.5 ± 1.9	2.0 ± 0.1
Eu-154	3.3 ± 0.5	1.8 ± 0.1
Sb-125	1.5 ± 0.2	0.71 ± 0.01
Final pH[c]	10.3	9.3

[a]At both laboratories, the concentrations of nonradioactive and radioactive elements in the blanks were negligible.

[b]Primarily Pu-238.

[c]Initial pH at SRS = 7.3, at ANL, 5.6. Final pH of blanks, 7.5 at SRS and 5.0 at ANL.

Table 5. SRS Results for Normalized Concentrations for Nonradioactive and Radioactive Elements Measured in the PCT on Radioactive 200R and 165/42 Glasses[a]

Nonradioactive			Radioactive		
Element	Norm. Conc. (g/L)		Element	Norm. Conc. (g/L)	
	200R Glass	165/42 Glass		200R Glass	165/42 Glass
B	1.1	0.41	Cs-137	0.20	0.12
Li	0.97	0.53	Sr-90	0.004	0.019
Na	0.99	0.34	Sb-125	0.59	0.44
K	0.57	-	Alpha[b]	0.098	0.13
Si	0.45	0.35			
Al	0.30	0.32			

[a]Calculated by dividing the leachate concentration for a certain element by its concentration in the glass.
[b]Primarily Pu-238 and Cm-244.

Table 6. Normalized PCT Concentrations (g/L) Obtained at SRS and ANL for Major Components of Radioactive and Nonadioactive Glasses[a]

	Radioactive				Nonradioactive		
Glass	200R		165/42		200	ARM-1	
Laboratory	SRS	ANL	SRS	ANL	SRS[b]	SRS[b]	ANL[c]
Element							
B	1.1	0.95	0.41	0.34	0.93	0.62	0.63
Li	0.97	0.93	0.53	0.45	1.4	0.78	0.68
Na	0.99	0.93	0.34	0.29	1.2	0.65	0.64
Si	0.45	0.40	0.35	0.27	0.45	0.32	0.29
SiO_2 Content of Glasses (wt %)	45.1		57.2		42.8	46.5	

[a]Calculated by dividing the leachate concentration for a certain element by its concentration in the glass.
[b]Data are in Reference 1.
[c]Leached at ANL as part of this interlaboratory comparison and the earlier one [6] with nonradioactive glass.

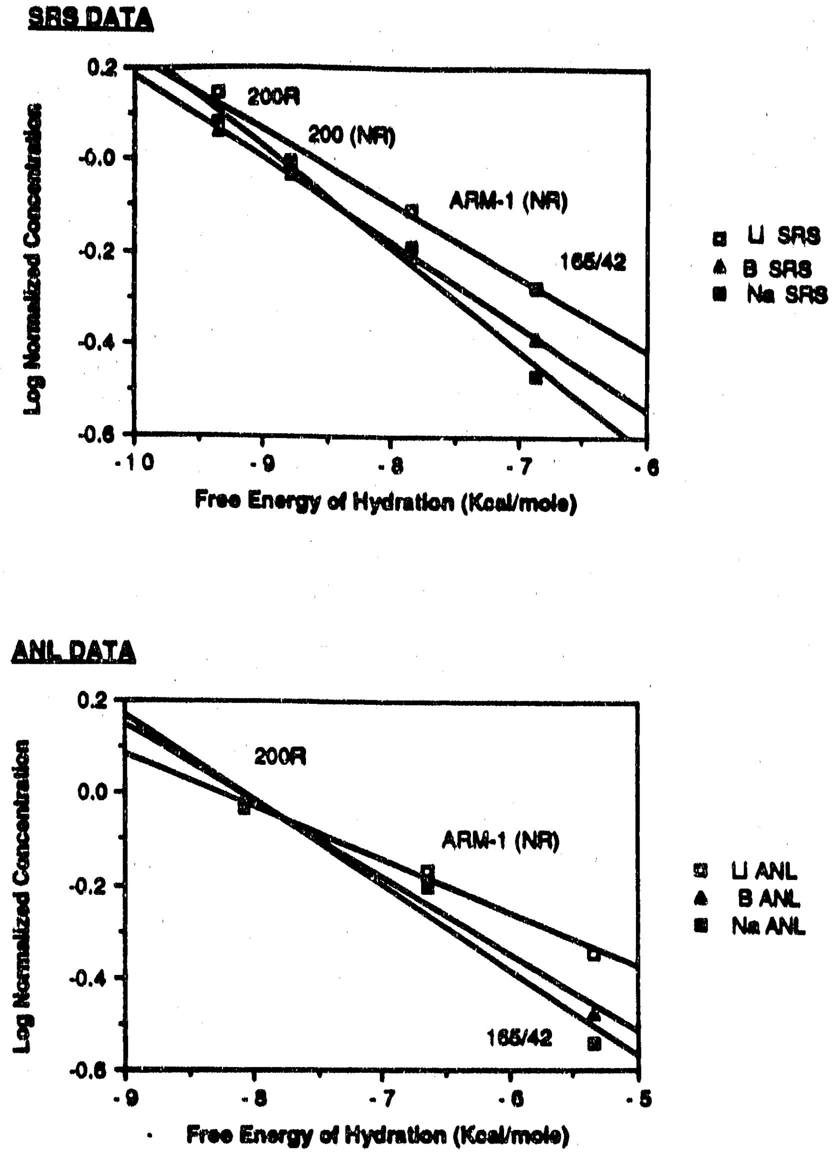

Figure 1. Relation of Normalized Concentrations Measured at SRS and ANL from Radioactive and Nonradioactive (NR) Glasses in the PCT to Glass Composition Expressed as Free Energy of Hydration (kcal/mole).

END

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