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DISSOLUTION TEST FOR
LOW-ACTIVITY WASTE PRODUCT ACCEPTANCE

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ABSTRACT

We have measured the mean and standard deviation of the solution concentrations of B, Na, and Si attained in replicate dissolution tests conducted at temperatures of 20, 40, and 70°C, for durations of 3 and 7 days, and at glass/water mass ratios of 1:10 and 1:1. These and other tests were conducted to evaluate the adequacy of the test methods specified in privatization contracts and to develop a data base that can be used to evaluate the reliability of reported results for tests performed on the waste products. Tests were conducted with a glass that we formulated to be similar to low-activity waste products that will be produced during the remediation of Hanford tank wastes. Statistical analyses indicated that, while the mean concentrations of B, Na, and Si were affected by the values of test parameters, the standard deviation of replicate tests was not. The precision of the tests was determined primarily by uncertainties in the analysis of the test solutions. Replicate measurements of other glass properties that must be reported for Hanford low-activity waste products were measured to evaluate the possible adoption of the glass used in these tests as a standard test material for the product acceptance process.

I. INTRODUCTION

As a part of the U.S. Department of Energy (DOE) privatization initiative, private contractors will perform pretreatment and immobilization functions for remediation of

radioactive tank wastes at the Hanford site [1, 2]. The DOE will provide waste to the contractors, then purchase waste products for subsequent handling and disposal. The waste products will be required to meet contractually specified acceptance criteria, which include chemical, radiological, and physical requirements. The criteria were developed to provide confidence that waste forms will perform acceptably in the disposal system. The acceptance specifications identify maximum allowable release rates of ^{99}Tc , ^{79}Se , ^{129}I , ^{237}Np , and U. The release rate of each radionuclide is to be calculated based on the amount that is immobilized and the average release rate of silicon measured with the Product Consistency Test Method B (PCT-B) at 20°C [3].

The work discussed in this paper was conducted to evaluate the precision and accuracy of glass dissolution rates measured with the PCT method. Under the auspices of the DOE Tanks Focus Area (TFA), we have measured the mean response (i.e., the solution concentrations of B, Na, and Si) and the standard deviation of replicate dissolution tests under various test conditions to (1) evaluate the adequacy of the durability test requirements that are contained in the existing contracts, (2) develop a data base that can be used to evaluate the credibility and validity of test results reported by private contractors for actual waste products, and (3) evaluate the possible adoption of the glass used in these tests as a standard material for demonstrating compliance with test requirements.

The composition of the glass used in these tests was formulated based on the anticipated compositions of immobilized low-activity waste (ILAW) forms for Hanford tank wastes. Since the compositions of waste products to be made by the contractors had not been announced when this program was started, and because the reference glass may be used as a composition standard, we included small amounts of several components that could be used in vitrified waste forms for Hanford or other sites. Some elements regulated by the Resource Conservation and Recovery Act (RCRA) were added to the glass, but no radioactive elements or their surrogates were added. We refer to the glass as Low-Activity Reference Material-1, or LRM-1.

Details regarding the formulation and physical evaluation of the LRM-1 glass have been published elsewhere [4]. A detailed report of the testing, analysis, and statistical results has also been published elsewhere [5]. In this paper, we summarize the results of replicate tests that we conducted to measure the mean and variability of the response under different test conditions.

II. EXPERIMENTAL METHODS

The LRM-1 glass was made by melting a mixture of oxides, carbonates, chlorides, fluorides, iodates, and sulfates that had been mechanically well-mixed. Small batches of a precursor glass were made by heating about 200 g of the mixture in Pt/Rh crucibles stepwise at temperatures of 800, 900, and 1000°C for one hour each, and then at a temperature of 1250°C for two hours. The glass was quenched by submersion in water. The precursor glass was removed from the crucible and crushed. The crushed glass was mixed and then remelted in six 250-g batches following the same procedure. The glass was quenched, crushed, and homogenized for use in the dissolution tests. One batch was prepared by annealing the glass for several hours at about 500°C. This glass was used to make monolithic samples that were used to characterize the microstructure and measure the density.

Replicate dissolution tests were conducted following the PCT-B procedure with crushed

glass [3]. All tests were conducted in Type 304L stainless steel vessels with the -100 +200 mesh size fraction of glass and demineralized water. The crushed glass was washed with water and ethanol following the PCT-B procedure. A small amount of glass was examined with the use of a scanning electron microscope (SEM) to ensure that fines had been removed. The vessels were cleaned following the PCT-B procedure. Vessel cleanliness was confirmed by analyzing demineralized water that had soaked in the vessel overnight at 90°C. A matrix of tests was conducted at temperatures of 20, 40, and 70°C for 3 or 7 days at glass/water mass ratios of 1:10 (1 g of glass in 10 g of water) and 1:1 (5 g of glass in 5 g of water). Vessels were sealed with Teflon gaskets and closure fittings. Tests at 20°C were conducted in a water bath with a refrigerated recirculation system, and tests at 40 and 70°C were conducted in convection ovens. Vessels were placed in plastic bags before being placed in the water bath. The water bath and oven temperatures were set with the use of NIST-traceable thermometers and monitored with thermocouples and a data logger. All temperatures remained within 1°C of the set temperature.

Nine replicate tests were conducted under each set of conditions. These were run as three sets of triplicate tests that were initiated on different days to measure the within-day and between-day variability of test execution and solution analysis. One test blank was run with demineralized water with each set of triplicate tests. These were used to detect possible contamination during testing and analysis and determine if background correction was required for the B, Na, or Si concentrations measured in tests with glass.

Care was taken to follow exactly the same methodology in every step of the procedure to minimize random variations in test execution and solution analysis. At the end of a test duration, vessels were removed from the oven and weighed to determine if any mass loss occurred. The vessels were then opened, and a 0.5 mL aliquot of the solution was removed and placed in a capped vial for pH analysis. The remaining leachate solution was removed from the vessel with a pipette and passed

through a filter (0.45- μm pore size) into a pre-cleaned solution bottle. This was done to ensure that no glass particles were included in the solution that was analyzed for dissolved glass components. The filtrate solution was acidified with ultra-pure nitric acid. The leachate solutions were analyzed for pH with a combination microelectrode within one hour of opening the vessels to minimize the effect of absorption of CO_2 by the solution. The acidified leachate solutions were analyzed with inductively coupled plasma-mass spectrometry (ICP-MS). The solution concentrations of B, Na, and Si were used to determine the precision of the replicate tests and calculate the dissolution rate under different test conditions.

III. RESULTS

The composition of LRM-1 glass was measured by dissolving aliquots of the glass, then analyzing the solutions. Two aliquots were dissolved in a closed-vessel dissolution procedure, and one aliquot in an open-vessel dissolution procedure with a mixture of ultra-pure hydrofluoric, nitric, and hydrochloric acids; one of the solutions from a closed-vessel dissolution was taken to dryness and fumed with perchloric acid to dissolve fluorides. Replicate aliquots of these solutions were analyzed with ICP-MS and inductively coupled plasma-atomic emission spectroscopy (ICP-AES). Another aliquot of the glass was fused with potassium hydroxide and then dissolved for anion analysis with ion chromatography (IC). The analyzed composition is given in Table 1 as the mean value and standard deviation of replicate analyses. That the total is less than 100% may be due to the selected gravimetric factors or to incomplete decomposition of carbonates used to make the glass. Why each component was included in the glass is given in Table 1; that is, components are present in the waste, glass-forming additives, RCRA additives, or chemicals added to broaden the usefulness of the glass as an composition standard.

Characterization of the microstructure with SEM and transmission electron microscopy (TEM) revealed that the glass was not phase separated and did not contain precipitated inclusions. Energy dispersive X-ray emission spectroscopy showed the glass to be

compositionally homogeneous. The density of the glass was measured by buoyancy to be $2516 \pm 9 \text{ kg/m}^3$. The specific area of the -100 +200 mesh fraction used in the dissolution tests is calculated to be $0.021 \text{ m}^2/\text{g}$ based on sieve size [3].

Table 1. Measured Composition of LRM-1 Glass, mean $\pm 1 \sigma$, in mass %.

Oxide	Role ^a	LRM-1
Al_2O_3	a, b	9.6 ± 0.6
B_2O_3	b	8.1 ± 0.3
BaO	c	0.0084 ± 0.0002
CaO	a, d	0.51 ± 0.06
CdO	c	0.17 ± 0.01
Cl	a	1.1
Cr_2O_3	a, c	0.18 ± 0.06
F	a	0.85
Fe_2O_3	a, d	1.0 ± 0.09
HgO	a	0.00019 ± 0.0001
I	a	0.0025 ± 0.0003
K_2O	a	1.2 ± 0.3
La_2O_3	d	0.0084 ± 0.0001
Li_2O	d	0.081 ± 0.03
MgO	a, d	0.086 ± 0.03
MnO	d	0.091 ± 0.02
MoO_3	d	0.10 ± 0.02
Na_2O	a	17.2 ± 0.6
NiO	d	0.082 ± 0.02
P_2O_5	a	0.47 ± 0.03
PbO_2	c	0.11 ± 0.02
SO_3	a	0.20
SiO_2	b	53.5 ± 1.0
SnO_2	d	0.10 ± 0.01
TiO_2	d	0.10 ± 0.01
ZrO_2	d	0.91 ± 0.3
Total		95.9

^a Role of Component:

a = present in waste stream

b = added glass-forming chemical

c = added RCRA component

d = added for use in composition standard

Table 2. Mean Responses and Standard Deviations for Nine Replicate Tests.

Temp., °C	Mass Ratio	Duration, days	pH	B, mg/L	Na, mg/L	Si, mg/L
20	1:10	3	9.06±0.11	0.09±0.03	5.22±0.22	0.50±0.06
20	1:10	7	9.13±0.25	0.11±0.03	5.87±0.35	1.03±0.19
20	1:1	3	10.19±0.05	0.64±0.21	35.7±2.4	6.35±0.49
20	1:1	7	10.21±0.16	0.69±0.34	40.3±1.2	11.6±2.1
40	1:10	3	9.36±0.04	0.42±0.09	10.06±0.67	4.14±0.51
40	1:10	7	9.41±0.08	0.86±0.21	13.3±0.93	8.90±0.95
40	1:1	3	10.36±0.05	2.96±0.22	62.9±1.48	25.2±1.2
40	1:1	7	10.52±0.08	4.14±0.41	76.5±4.32	35.9±3.2
70	1:10	3	9.69±0.06	3.50±0.17	33.7±1.6	24.7±1.8
70	1:10	7	9.86±0.07	4.67±0.45	43.4±1.8	34.9±1.6
70	1:1	3	10.93±0.01	20.6±1.4	194±14	75.0±6.6
70	1:1	7	11.11±0.08	27.6±3.5	227±14	92.3±8.2

Only Al and Na were detected in the blank test solutions, and the measured concentrations were insensitive to the duration, temperature, or the amount of water used in the test. The mean concentrations from all blank tests were 9.86 µg/L Al and 84.7 µg/L Na; B and Si were below the quantitation limits of <7 µg/L B and <60 µg/L Si.

The mean and standard deviations of the solution concentrations measured in replicate tests with glass are summarized in Table 2. Note that the B, Na, and Si concentrations in all tests are well above the concentrations that were measured in the blank tests. As expected, the mean concentrations of B, Na, and Si all increase with increasing temperature, test duration, and glass/water mass ratio. We used one-way analysis of variance (ANOVA) to statistically determine the effects that these factors have on the mean response of B, Na, and Si [6]. This statistical procedure was used to compare the effects of test parameters

against random error. The between-day variability of triplicate tests was also evaluated.

The results of the one-way ANOVA that we used to evaluate the relative effects of the test parameters and different sets of triplicate tests are summarized in Table 3. An effect is statistically significant at the 95% confidence level if the value of "Prob>F" is less than 0.05. The temperature and glass/water mass ratio significantly affected the mean concentrations of B, Na, and Si. The test duration significantly affected the mean concentrations of Si, but the effects on the B and Na concentrations were only possibly significant. The between-day variability of triplicate tests is insignificant compared to the effects of the other parameters.

The effects of individual parameters on the mean solution concentrations from the nine tests conducted under each set of test conditions were analyzed next. The glass/water mass ratio had a statistically

significant effect on the mean concentrations of B, Na, and Si: higher concentrations of each were generated in tests at a mass ratio of 1:1 than in tests at 1:10 at all temperatures and durations. The solution concentration of B, Na, and Si increased with the temperature at all combinations of mass ratio and duration. The solution concentrations were higher with test duration for all combinations of mass ratio and temperature except for tests conducted at 20°C.

Table 3. One-way ANOVA Results of the Effects of Temperature, Duration, Glass/Water Mass Ratio, and Sets of Triplicate Tests on B, Na, and Si Concentrations.

Boron			
Factor	F-ratio	Prob>F	Significant?
Temp.	88.320	<0.0001	yes
Mass Ratio	72.670	<0.0001	yes
Duration	3.1995	0.0767	no
Triplicate	0.2012	0.8181	no
Sodium			
Factor	F-ratio	Prob>F	Significant?
Temp.	102.45	<0.0001	yes
Mass Ratio	196.44	<0.0001	yes
Duration	3.0241	0.0851	no
Triplicate	0.1181	0.8887	no
Silicon			
Factor	F-ratio	Prob>F	Significant?
Temp.	229.15	<0.0001	yes
Mass Ratio	195.91	<0.0001	yes
Duration	15.731	0.0001	yes
Triplicate	0.1696	0.8443	no

In those tests, the solution concentrations of B in the 3-day and 7-day tests were not statistically different at either mass ratio, but the concentrations of Na and Si were. The difference in the response of B compared to that of Na and Si is likely due to the lower solution concentrations of B generated in those tests.

Differences between the means measured in different sets of triplicate tests under the same conditions were also evaluated. The results of ANOVA analyses for the measured Si concentrations are summarized in Table 4. These results show there are significant differences between the average Si concentrations that are measured in triplicate tests under some test conditions, but not for others. The Na concentrations for the different sets of triplicate tests are also statistically different under some test conditions. The test conditions under which the mean Na and Si concentrations are statistically distinguishable are not always the same. Variance in either the Na or Si concentrations, or both, is observed at all temperatures, mass ratios, and test durations. Analysis of the B concentrations showed that the mean concentrations of different sets of triplicate tests were statistically different under all test conditions.

That sets of tests for which significant differences exist are not correlated with the test parameters indicates that the precision of any one set of triplicate tests is higher than the overall precision of all tests. This suggests that another factor affects the mean results but has not been taken into account. That factor is the day-to-day variability of the solution analysis.

The variability of the solution analysis was measured by reanalyzing the solutions from tests at 40 and 70°C conducted for 3 and 7 days. Only enough solution remained from tests conducted at a glass/water mass ratio of 1:10. The results of one-way ANOVA comparison of the mean concentrations of Na in duplicate analysis revealed that the effect of solution analysis was significant for tests conducted at 40°C at both durations, and at 70°C for 7-day tests. The difference in the results of 70°C at 3 days was found not to be significant, although this is probably fortuitous. The Si concentrations were found

Table 4. ANOVA in Si Concentrations for Sets of Triplicate Tests, Si concentrations in mg/L

Temp., °C	Mass Ratio	Duration, days	Mean of Replicates 1, 2, 3	Mean of Replicates 4, 5, 6	Mean of Replicates 7, 8, 9	P-value	Significant difference?
20	1:10	3	0.54	0.48	0.48	0.4675	no
40	1:10	3	4.78	3.74	3.88	0.0006	yes
70	1:10	3	22.9	24.5	26.7	0.0024	yes
20	1:1	3	6.90	6.12	6.04	0.0255	yes
40	1:1	3	24.5	25.0	26.0	0.3184	no
70	1:1	3	69.0	79.4	76.5	0.1175	no
20	1:10	7	1.28	0.93	0.88	<0.0001	yes
40	1:10	7	9.92	8.68	8.09	0.0208	yes
70	1:10	7	36.4	33.9	34.3	0.1088	no
20	1:1	7	14.4	10.5	10.1	0.0002	yes
40	1:1	7	39.1	34.1	34.5	0.0760	no
70	1:1	7	101	86.4	89.8	0.0456	yes

by ANOVA not to differ significantly, even though the differences in the measured concentrations were similar to those of the Na concentrations. This is attributed to the greater uncertainty of each measured Si concentration inherent with ICP-MS analysis due to isobaric interference from CO and N₂. This probably masked the variance between sample analyses.

It is worth pointing out that, while the finding that temperature, duration, and glass/water mass ratio have a significant effect on the mean response in the PCT may seem trivial or intuitively obvious, the statistically based testing of these factors was required to establish the significance of the variability of the solution analysis procedure.

Test results were compared on the basis of variance to determine which set of parameters resulted in the lowest intrinsic random variability. For this analysis, the effect of the parameter of interest on a test result was isolated from the effects of the other parameters. This was done by normalizing the measured response (i.e., the B, Na, or Si concentration) for each test within a set of triplicate tests conducted under the same conditions to the mean response of that set of tests. The standard deviation was then calculated for the normalized responses. In

this way, the results of the 54 tests conducted at a glass/water mass ratio of 1:10 could be compared directly to the 54 tests conducted at a mass ratio of 1:1, regardless of the temperature or duration of the test, or when the test was run. Likewise, the effects of other test variables were evaluated. The normalized standard deviations thus calculated for each parameter were then compared with the F-test to determine if one parameter value resulted in a greater or smaller test variability than another. None of the parameters resulted in a significantly different intrinsic variability in the Na or Si concentrations at the 95% confidence level. The only significant difference is that solutions from tests run at a glass/water mass ratio of 1:1 have a lower B variability than the solutions from tests run at a mass ratio of 1:10.

IV. DISCUSSION

The results of statistical analyses show that the primary factor influencing the reproducibility of the elemental concentrations is the analytical protocol used to measure them. The intrinsic variability was insensitive to the test parameters of glass/water mass ratio (1:1 or 1:10), temperature (20, 40, or 70°C), or test duration (3 or 7 days). Only the variability of the B solution concentration was

significantly affected by the glass/water mass ratio. This is most likely due to the low concentrations that are generated rather than an effect of the mass ratio. Since analytes present at higher solution concentrations can generally be measured more accurately, test conditions that result in higher solution concentrations should provide higher precision. However, the benefit of selecting test parameters that generate more concentrated solutions may be diminished because some leachate solutions must be diluted to fall within the linear region of the instrumental response. This was the case for our 70°C tests. Dilution may also be required to provide a sufficient volume of solution for analysis. Although tests at a mass ratio of 1:1 generate solutions that are more concentrated than those in tests at 1:10, less solution can be recovered from those tests. These solutions must be diluted to a greater degree than those from tests at a mass ratio 1:10 to provide an ample aliquot for analysis, so that the benefits of conducting tests at a higher glass/water mass ratio are diminished.

The average normalized dissolution rates were calculated from the mean Si concentrations with the following equation:

$$NR(Si) = C_{Si} / (S \cdot f_{Si} \cdot t) \quad (1)$$

where $NR(Si)$ is the average normalized dissolution rate, C_{Si} is the measured concentration of silicon, S is the surface area of the glass, f_{Si} is the mass fraction of silicon in the glass, and t is the reaction time. The rates calculated with the mean concentrations measured under different test conditions are shown in an Arrhenius plot in Fig. 1. The rate increases with temperature and decreases with test duration and glass/water mass ratio. The effect of temperature on the dissolution rate is of interest because the Hanford privatization contracts allow for the rate at 20°C to be estimated based on the rates measured in tests conducted at elevated temperatures. For LRM-1 glass, simple extrapolation of rates measured at 40 and 70°C will clearly overestimate the dissolution rate at 20°C under all test conditions and provide a conservative upper bound.

The dissolution rate of LRM-1 glass measured in these tests provides insight into

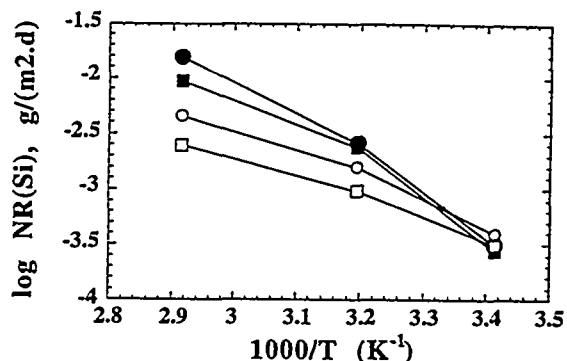


Figure 1. Arrhenius plot of dissolution rates calculated from measured Si concentrations under different test conditions: (●) 3-day test at 1:10, (■) 7-day test at 1:10, (○) 3-day test at 1:1, (□) 7-day test at 1:1.

whether the concentrations that are generated in the acceptance test by a glass that meets the dissolution rate requirement will likely be high enough to be analyzed. Based on the average silicon concentration of 1 mg/L that was measured in replicate tests, the calculated dissolution rate in the tests conducted at 20°C with a mass ratio of 1:10 for seven days is $0.28 \times 10^{-3} \text{ g/(m}^2 \cdot \text{d)}$. The average final pH value was 9.13. In recent sensitivity calculations for a near-surface disposal site at Hanford, a dissolution rate of $6 \times 10^{-3} \text{ g/(m}^2 \cdot \text{d)}$ at pH 9.5 was assumed to be an upper limit for the long-term corrosion rate [7]. The rate measured for LRM-1 glass with the 7-day test at 20°C is about 20 times lower than this rate. Thus, while the required durability in the product specifications will depend on the amounts of radionuclides that are immobilized, the Si concentrations in tests with anticipated waste products will likely be higher than those measured for LRM-1 glass.

Finally, the results of this matrix of tests were used to select conditions in a round robin test program to measure interlaboratory precision. The conditions selected were 7 days, 40°C, and a glass/water mass ratio of 1:10. A duration of 7 days was selected to be consistent with the duration specified in the Hanford contracts. A temperature of 40°C was selected instead of 20°C because we considered the mean concentrations generated in tests at 20°C to be too low to reliably

calculate the dissolution rate. This judgment was made, in part, based on the requirement in the PCT Method A procedure that solute concentrations be greater than ten times the background concentrations for the results to be "valid" [5]. A mass ratio of 1:10 was selected to provide a sufficient volume of solution for analysis.

V. CONCLUSION

Replicate tests following the PCT-B protocol have demonstrated that the temperature, test duration, and glass/water mass ratio values that are used in an acceptance test will all affect the mean concentrations, but none of these parameters will significantly affect the testing precision. The precision is determined instead by the analytical uncertainty associated with the solution analysis. Test parameter values that result in more concentrated solutions may lead to better analytical precision, but the use of some parameter values may be impractical because of instrumental limitations of the quantifiable concentration range and the amount of solution that may be required for multi-elemental analysis. The data base of replicate test results provides insight into the precision that can be expected for the PCT method performed with these test parameters.

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