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**ROUND-ROBIN TESTING OF A REFERENCE GLASS FOR
LOW-ACTIVITY WASTE FORMS**

by

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W. L. Ebert and S. F. Wolf

ABSTRACT

A round robin test program was conducted with a glass that was developed for use as a standard test material for acceptance testing of low-activity waste glasses made with Hanford tank wastes. The glass is referred to as the low-activity test reference material (LRM). The program was conducted to measure the interlaboratory reproducibility of composition analysis and durability test results. Participants were allowed to select the methods used to analyze the glass composition. The durability tests closely followed the Product Consistency Test (PCT) Method A, except that tests were conducted at both 40 and 90°C and that parallel tests with a reference glass were not required. Samples of LRM glass that had been crushed, sieved, and washed to remove fines were provided to participants for tests and analyses. The reproducibility of both the composition and PCT results compare favorably with the results of interlaboratory studies conducted with other glasses. From the perspective of reproducibility of analysis results, this glass is acceptable for use as a composition standard for nonradioactive components of low-activity waste forms present at >0.1 elemental mass % and as a test standard for PCTs at 40 and 90°C. For PCT with LRM glass, the expected test results at the 95% confidence level are as follows:

at 40°C:	pH = 9.86 ± 0.96	at 90°C:	pH = 10.92 ± 0.43
	[B] = 2.30 ± 1.25 mg/L		[B] = 26.7 ± 7.2 mg/L
	[Na] = 19.7 ± 7.3 mg/L		[Na] = 160 ± 13 mg/L
	[Si] = 13.7 ± 4.2 mg/L		[Si] = 82.0 ± 12.7 mg/L

These ranges can be used to evaluate the accuracy of PCTs conducted at other laboratories.

I. INTRODUCTION AND BACKGROUND

In this report, we summarize and discuss the results of a round robin testing program conducted to measure the interlaboratory precision for measuring the composition of a borosilicate glass and conducting a standardized dissolution test with that glass. The round robin was conducted under the auspices of the DOE Tanks Focus Area program - Immobilization. The glass used in the round robin program was formulated at Argonne National Laboratory (ANL) for possible use as a standard material in acceptance testing of vitrified waste forms made by a contractor with the low-activity waste stream generated during pretreatment of Hanford tank wastes [EBERT-1998]. The composition of the glass was formulated based on the anticipated composition of the low-activity waste stream to be generated during pretreatment of Hanford tank wastes and includes chemicals that may be added by the contractor to facilitate vitrification of the waste. A small amount of glass with that composition was made in the laboratory and evaluated to determine the adequacy of several of the tests specified in the initial contract and the possible use of the material as a test standard [DOE-1996]. Those scoping tests indicated the material was suitable for use as a standard [WOLF-1998]. About 1000 pounds of the material was then procured from Ferro Corporation (Cleveland, Ohio) for use as a standard test material. The glass is referred to as the low-activity test reference material (LRM).

The round robin testing program discussed in this report was conducted to measure the interlaboratory precision for use of LRM as (1) a composition standard for nonradioactive components of the waste forms and (2) a standard material for the chemical durability test. The data base generated in the round robin provides a measure of the precision that can be expected from laboratory analyses of waste glasses and a measure of the reliability of the results of contractor laboratories during waste acceptance testing.

The composition of the waste product must be known for product acceptance from the vendor and for disposal [DOE-1996]. The chemical durability test is used to provide a measure of the dissolution rate of the waste form that can be used to bound the fractional release rates of radionuclides. The method specified in the contract to measure the glass dissolution rate in acceptance testing is the Product Consistency Test (PCT). Two PCT methods have been standardized by the American Society for Testing and Materials [ASTM-1285]: Method A is used to monitor the consistency of high-level waste glasses under specific test conditions; Method B is used to study waste form behavior under other tests conditions. The contract requires that PCTs be conducted with low-activity waste products at 40 and 90°C. It was determined in a previous study that the conditions specified in PCT Method A were appropriate for acceptance testing of low-activity waste glasses at both 40 and 90°C [WOLF-1998].

The target composition of the LRM glass is given in Table 1. The glass components are identified as those anticipated to be present in the waste streams and those that may be added by the contractor to facilitate the vitrification or durability of the immobilized low-activity waste products. The glass also contains small amounts of Ba,

Cd, Cr, and Pb, which are regulated by the Resource Conservation and Recovery Act (RCRA). The prototype glass was shown to be nonhazardous per the Toxicity Characteristic Leach Procedure (TCLP) [WOLF-1998]. Although the prototype composition used in the scoping tests contained small amounts of chlorine and mercury [EBERT-1998], they were excluded from the target composition of LRM; vendors would not agree to make a glass with chlorine to avoid contaminating the melters, which are used to make chlorine-free glasses for other customers. The absence of chlorine and mercury is not expected to affect the dissolution behavior of the glass. The target concentration of SiO_2 was increased slightly from the original formulation to account for the exclusion of Cl and Hg.

It was specified that the LRM glass be fully oxidized and water quenched to avoid devitrification during cooling. The glass was to be crushed to provide the $-4 +100$ mesh fraction, with less than 2.5 mass % -100 mesh, and to be homogenized.

The LRM glass was shipped to ANL in September 1998. The glass was examined and found to meet the particle-size specification. Chemical analyses were conducted for Ferro Corp. by Corning Engineering Laboratory Services (CELS) as a part of the procurement contract. The glass was also analyzed by the ANL Analytical Chemistry Laboratory (ACL). The two analyses indicated that the composition of the glass was acceptable. The analyses by CELS and ANL/ACL are evaluated with the composition analyses conducted as part of the analytical round robin, and are not identified separately.

II. DESIGN OF ROUND-ROBIN PROGRAM

The round robin was conducted following the approach given in ASTM standards E1601, *Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method* [ASTM-1601], and E691, *Conducting an Interlaboratory Study to Determine the Precision of a Test Method* [ASTM-691]. For example, as recommended by the procedure, the laboratories invited to participate in the round robin were known to have personnel experienced in performing chemical analyses of glasses and PCTs, the necessary facilities to complete the study, and the likelihood of using the methods being tested in the future. Because funds were not provided to participants, the scope of the study was tightly focused on the use of LRM as a composition and test standard. Additional analyses were considered for inclusion in the study to gain added insight, but were not included in the round robin so as to not overburden laboratories and possibly lower the number of laboratories that participate in or complete the study. For example, we considered providing a standard solution to all laboratories to distinguish interlaboratory variations in solution analysis from variations due to the methods used to dissolve the glass for composition analysis and from test execution. This was not done because it was viewed as unessential to the purpose of the study, since only the overall results of testing and analysis will be considered in product acceptance testing.

Compliance with ASTM standards for an interlaboratory study requires that at least six laboratories participate in the round robin. To meet this requirement, as many as two laboratories may submit independent sets of data generated by independent experimenters using separate analytical equipment and standards. In the present study, six different laboratories participated in the study (a participant from a seventh laboratory did not complete the study). Two laboratories provided two independent sets of composition results and one laboratory provided three independent sets of PCT results. The participating laboratories and scientists are listed in Appendix A. Letters to solicit participants and provide instructions for the program and for data submittal are included in Appendix B.

Standard E1601 provides three test patterns for interlaboratory studies wherein replicate tests are run sequentially as either single or duplicate tests. None of these patterns were followed for this study. Instead, the ASTM protocol for the PCT that calls for conducting triplicate tests in parallel was followed. In the case of the composition analysis, participants were only requested to provide a single composition analysis. This is because the focus of the study was to evaluate the use of the glass as a composition standard rather than to evaluate the methods that were used. Indeed, several different procedures were used by the participants to dissolve and analyze the glass. Some participants did provide the results of replicate analyses.

All the glass used in the study was prepared at ANL to eliminate the effects of crushing, sieving, and the efficiency of washing fines from the glass from the response in the PCTs. The glass was received from Ferro Corp. in four 250-pound barrels. About 1 kg of glass from each barrel was placed in a large container and mixed by rolling the container. About 500 g of glass was then crushed and sieved to isolate the -100 +200

mesh size fraction, which was then washed to remove fines and dried at about 90°C. This glass was used in the round robin program. About 17 g of glass was placed in a plastic screw-top container and provided to each participant for use in both composition analysis and PCTs. All samples were identified as "TFA Reference Glass LRM (ANL GS 713)." Seven 17-g sample aliquots were held in reserve. Additional aliquots were later provided to two participants on request.

Participants were provided with the composition of the prototype glass to help guide the procedures used to dissolve and analyze the glass (see Appendix B). No restrictions were placed on the method used to dissolve the glass for chemical analysis or the analytical techniques to be used, except that the methods used to dissolve and analyze the glass had to be reported with the analytical results. This is because the purpose of this interlaboratory study is to evaluate the use of LRM glass as a composition standard, not to evaluate the techniques that are used to determine the composition.

The participants were instructed to follow the PCT Method A procedure for tests at both 40 and 90°C as closely as possible, except that tests with a standard reference glass were not required. Key aspects of the test procedure used in the round robin are summarized below:

- Three replicate tests were to be conducted at both 40 and 90°C with between 1 and 1.5 g of crushed glass and a mass of demineralized water (ASTM Type I water) exactly 10 times the mass of glass used.
- Tests were to be conducted for 7-day durations. The times of day a test vessel is placed in the oven and removed from the oven had to agree within 3.4 hours.
- The use of unsensitized Type 304L stainless steel vessels was preferred, but tests could be conducted with Teflon vessels if steel vessels are not available. Vessels were to be cleaned according to the PCT procedure for Type 304L stainless steel or Teflon vessels. The vessel type used in the tests had to be reported.
- Duplicate blank tests with demineralized water were conducted simultaneously with the tests with glass. The vessels used in the blank tests were to have been cleaned in the same batch as the vessels used in the tests with glass.
- The oven temperature was to remain within 2°C of the test temperature during the test.
- For the results of the PCTs to be accepted, the total mass of the test vessel could not change during the test by more than 5% of the mass of demineralized water that was initially added to the vessel.
- Solutions generated in the tests with glass and the blank tests were to be filtered through 0.45- μ m-pore filters, acidified with concentrated nitric acid, and then analyzed for B, Na, and Si. Estimated detection limits were to be reported.
- The model PCT data sheet provided in Figure 2 of the ASTM procedure could be used, as could other data sheets containing the test information.
- While a particular analytical method was not required for solution analysis, the method(s) used had to be reported.

A copy of the ASTM PCT procedure C1285 was provided to all participants.

III. RESULTS AND DISCUSSION OF COMPOSITION ANALYSIS STUDY

Glass from the same source was used for composition analysis and PCTs. Several participants used more than one method to dissolve the glass for composition analysis and conducted replicate dissolutions using the same methods. Several participants also conducted replicate analyses of the same solutions. The analytical methods used by the participants to dissolve the glass for analysis and then to analyze the solutions are summarized in Tables 2 and 3, respectively. Independent analyses are grouped by letter according to the participant and analytical technique used to analyze the solutions. The analytical techniques are listed in Table 3 as reported by the participants (inductively coupled plasma - atomic emission spectroscopy (ICP-AES), inductively coupled plasma - emission spectroscopy (ICP-ES), and inductively coupled plasma - optical emission spectroscopy (ICP-OES) refer to the same technique). The results for analysis of the glass composition are given in Table 4. The values are given with the same number of significant figures reported by the participants. Some laboratories used more than one dissolution procedure or conducted replicate dissolutions by the same procedure. Some participants reported the results of each individual analysis; other reported the average and standard deviation of replicate analyses, but not the results of each analysis.

The average values for results of samples analyzed by a participant with the same analytical technique and the overall average results for all participants are given in Table 5. The data were analyzed in terms of laboratory-method combinations, where the method was the analytical method used by the participant. In the case of participant F, the data were analyzed in terms of the method used to dissolve the glass for analysis. The deviation of the compositions measured by the participants (of the average of replicate determinations) from the overall average and the standard deviation for all participants are listed in Table 6. The results of the statistical analyses for the glass composition analysis are summarized in Table 7. They include the measures of repeatability and reproducibility as calculated in ASTM Standard E691: "Repeatability concerns the variability between independent test results obtained within a single laboratory in the shortest practical period of time by a single operator with a specific set of test apparatus using test specimens taken at random from a single quantity of homogeneous material obtained or prepared for the interlaboratory study. Reproducibility deals with the variability between single test results obtained in different laboratories, each of which has applied the test method to test specimens taken at random from a single quantity of homogeneous material obtained or prepared for the interlaboratory study" [ASTM-691]. The reader is referred to that standard for a detailed description of the calculations. The intralaboratory and interlaboratory components of variance (s_r and s_L) and the interlaboratory estimate of precision (s_R) are plotted against the average concentrations of various glass components in Fig. 1. Regression lines are drawn for the three measures of precision. All increase with the concentration of the element that is analyzed.

The objective of the composition analysis portion of the round robin was to determine if the LRM glass was suitable for use as a composition standard for waste forms made with the low-activity waste streams generated from Hanford tank wastes. The objective was not to evaluate particular analytical methods used at different laboratories;

specific methods were not required for either dissolving the glass for analysis or analyzing the resulting solutions. Hence, the reported results include the uncertainties due to both the dissolution and analytical procedures. The results generated by this interlaboratory study with LRM glass can be compared with the results of previous interlaboratory studies that were conducted with other glasses to determine if the analyses of the composition and test results have similar analytical uncertainties for LRM glass and for other glasses. These comparisons can be used to judge the suitability of the LRM glass as a composition standard material.

The measured and the target compositions are compared in Table 8. These results show that the difference between the measured glass composition and the target composition exceeds the 95% confidence limit for Ba, Cd, Cr, and Ni. The concentration of I was below detection levels for most participants, so the agreement with the target concentration could not be evaluated. The measured and target values of the major glass components Al, B, Na, and Si agree very well.

Several analytical round robin studies have been administrated by the Materials Characterization Center (MCC) at Pacific Northwest National Laboratory in which the analysis of reference nuclear waste glasses have been studied. The results of seven analytical round robins are available for comparison with the results for LRM glass [SMITH-1994; SMITH-1997]. The MCC analytical round robins 1 and 4 were conducted with ATM-WV/205 glass, which is a reference glass for the West Valley Demonstration Project. Round robin 2 was conducted with a mixture of ATM-WV6/205 glass and ARM-1 glass, which is a standard glass. Round robins 3 and 5 were conducted with batches of ARM-1 glass. Round robin 6 was conducted with ARG-1 glass. Round robin 7 was conducted with ARM-1 glass, West Valley reference glass 6 (WV6), and Environmental Assessment (EA) glass. In MCC analytical round robin 7, the results of analysis of the ARM-1 glass were used to bias-correct the results for the other glasses.

The results for ARM-1 in MCC analytical round robin 5 and the results of WV6 and EA glasses in MCC analytical round robin 7 were compared with the present results with LRM glass; the results of WV6 and EA glass without the bias correction were used [SMITH-1994; SMITH-1997]. In these MCC analytical round robins, participants were provided with crushed glass for the analysis. While the WV6 and EA glasses are not used as composition standards, the precision with which these glass compositions were measured provides a useful comparison of how precisely the composition of a waste glass can be determined. The percent relative standard deviation (%rsd) was calculated for comparison of the four glasses by dividing the published consensus results by standard deviation for each element and multiplying by 100. The %rsd for analysis of LRM glass is given as v_r in Table 7. The %rsd is plotted against the mean of the measured concentrations for tests with each glass in Fig. 2a-d. The %rsd values for Ba, Cr, Cu, K, P, and Zn for the EA glass and the %rsd value for Nd for the WV6 glass are greater than 50% and have been excluded from the plots to facilitate comparison of the different glasses. The elements not shown in the plots are all present at concentrations less than 0.5 oxide mass %.

Of particular interest are elements present at concentrations of 0.5 elemental mass % or higher, since they must be quantified as a part of product acceptance. In general, the %rsd values are lower for elements that are present in the glasses at higher concentrations. Diagonal lines are drawn in the plots to facilitate comparison of the analytical precision attained in studies with the four glasses. The lines have no statistical or physical meaning, but provide a simple means to compare the data on the four different plots. The number of data that fall above the line for elements present in the glass at concentration greater than 0.01 mass % are as follows: five for the ARM-1 glass (Ce, Cs, Na, Nd, and Si), seven for EA glass (Fe, La, Na, Si, and Ti, plus Cu and P, which were excluded from the plot), five for the WV6 glass (Al, Fe, La, and P, plus Nd, which was excluded from the plot), and one for LRM glass (K). A breakdown of the precision with which individual elements were analyzed in studies with LRM, ARM-1, EA, and WV6 glass is given in Table 9. The precision is similar for quantitation of most elements in all four glasses.

From this comparison, the composition of LRM glass was measured more precisely, on average, than the compositions of the ARM-1, EA, or WV6 glasses in previous interlaboratory studies. The greater precision with which the LRM glass composition was measured in the present study is probably due, in part, to improvements in the analytical methods since the MCC analytical round robin numbers 5 and 7.

IV. RESULTS AND DISCUSSION OF PCT STUDY

The PCT specifies that the glass/water mass ratio be 10.0, but it does not specify the absolute masses to be used. This is because the concentrations that are achieved in the test are presumed to depend on the glass surface area/solution volume (S/V) ratio, which is proportional to the mass ratio, but not on the absolute amounts of glass and water that are used in the test. Three sets of triplicate tests were conducted at ANL to verify that the solution concentrations were independent, within experimental uncertainty, of the absolute masses of glass and water used in PCTs that were conducted at a glass/water mass ratio of 10.0. This was done to demonstrate that solution concentrations measured by different participants could be compared directly, regardless of the absolute amounts of glass and water used, if the tests were conducted with the same glass/water mass ratio. The results and the mean and standard deviation for each set of tests are given in Table 10 and plotted in Fig. 3. These results show no significant effect of the absolute masses of glass and water that are used. Therefore, data from tests conducted with different masses of glass and water at the same mass ratio were compared directly.

The results of PCTs conducted with LRM glass and blank tests at 40 and 90°C are presented in Tables 11 and 12. The mean values and standard deviations for the replicate tests conducted by each participant are summarized in Tables 13 and 14, and the deviations of the mean values for each participant from the overall average values and the interlaboratory standard deviations for each analyte are given in Table 15. The values of mean ($\bar{x}_p$) and standard deviation (s) for the results of each participant are plotted in Figs. 4a and b. (In most cases, the standard deviation is within the size of the symbol.) The mean ($\bar{x}$) and the standard deviation of the means of replicate tests by each participant are included on the right hand side of each plot. Horizontal lines are drawn through the average of the means to show the variation in the measured values.

The results of the statistical analyses of the PCT results are summarized in Table 16. They include the measures of repeatability and reproducibility as calculated in ASTM Standard E691. The reader is referred to that standard for a detailed description of the calculations. The data were analyzed in terms of replicate tests. Note that the values in parentheses in Tables 13-15 were excluded as outliers in the statistical evaluation of the PCT results, as discussed below. The intralaboratory and interlaboratory components of variance (s_r and s_L , respectively) and the interlaboratory estimate of precision (s_R) are plotted against the average values of the pH and the concentrations of B, Na, and Si in Fig. 5.

Comparison of the values of s_r in Table 14 with the values of s_x in Table 15 shows that the interlaboratory precision is statistically poorer than the intralaboratory precision for pH, B, Na, and Si results from the PCTs with LRM glass at both temperatures. It was observed previously in tests with LRM that more precise results were obtained when solutions were analyzed on the same day than when the solutions were analyzed on different days [WOLF-1998]. One laboratory provided the results of analyses of the test solutions that were conducted on two different days. The results as the mean $\pm$ s for each set of analysis and for the combined results are summarized in Table 17. While the

standard deviations for analyses conducted on the two days are essentially the same, the mean values differ by more than two standard deviations for most elements. Thus, the day-to-day variability in solution analysis is greater than the same-day variability. The precision of the duplicate analysis by Participant E is significantly lower than the interlaboratory precision except in the case of boron at 40°C. It may be that the long-term intralaboratory precision will be similar to the short-term interlaboratory precision measured in the present interlaboratory study with LRM glass. Further testing is required to confirm this hypothesis.

The mean and standard deviations for replicate tests conducted by each participant can be compared to the consensus values for all participants using the graphical method recommended in ASTM practice E691. The H-parameter is defined as the difference between the mean value measured by a participant and the consensus mean, divided by the standard deviation for all labs. The H-parameter values were calculated using the values of B, Na, and Si in Table 15 for tests conducted at 40 and 90°C and are plotted in Fig. 6. The horizontal lines drawn at $H = \pm 2.15$ indicate the critical value defined in E691 for an interlaboratory study with eight participants. The H-parameter value for the average Si concentration determined by Participant G in tests at 40°C exceeds the critical value. The H-parameter values for the average concentrations of B, Na, and Si determined by Participant H in tests at 90°C are all higher than the critical value. Those test results with H-parameters exceeding the critical value were excluded from the statistical calculations. The results of Participants G and H agree with the consensus values in tests at the other temperature, which suggests that the differences are due to differences in the test execution rather than the solution analyses.

An initial concern addressed in the scoping tests with LRM-1 glass was whether the concentrations of all elements in the solutions generated in tests at 40°C would be high enough to quantify. The concentrations of Al, B, Na, and Si in tests at 40°C were quantified by all participants; the concentrations in tests with glass were at least 5 times the detection limit or the concentration measured in the blank test. The intralaboratory precision, as measured by the square root of the coefficient of variance (v_r), for PCTs conducted at 90°C is about 3X better than for tests conducted at 40°C. The interlaboratory precision, as measured by the square root of the coefficient of variance (v_R), is about 2X better in tests at 90°C than in tests at 40°C. The intralaboratory precision is about 3X better than the interlaboratory precision at both test temperatures.

The precision for tests with LRM can be compared with the results of other interlaboratory studies cited for two standard glasses in the Precision and Bias section of the PCT procedure [ASTM-1285]. The interlaboratory precision (i.e., v_r) in tests using glasses that were not washed to remove fines was 2.64, 3.22, 7.52, and 7.05% in tests with SRL 202P, SRL 202G, ARM-1, and NIST reference glass SRM 623, respectively, all based on boron (from Table 3.2 in [PIEPL-1989]); the interlaboratory precision (i.e., v_R) for these glasses was 5.02, 5.01, 12.13, and 8.64%, respectively (from Table C.2 in [PIEPL-1989]). (Note that there is an error in the Precision and Bias section of ASTM C1285. The values for these tests given in the PCT Precision and Bias section 27.1.5.1 are the values in Table C.1 in [PIEPL-1989] rather than Table C.2, as cited. The values

given in Table C.1 in [PIEPL-1989] include test results determined by the H-parameter to be outliers.) In a study with only two participants, the intralaboratory precision was 2.2 and 5.3% for triplicate tests with SRL 200R glass and 2.9 and 3.5% for triplicate tests with SRL 165/42 glass [BIBLER-1990]. These glasses were washed to remove fines prior to testing. The interlaboratory precision for boron results in that study was 13% for tests with SRL 200R glass and 14% for tests with SRL 165/42 glass. These values bracket the precision in tests with LRM glass at 90°C based on boron, for which the intralaboratory %rsd (v_r) value is 2.4% and the interlaboratory %rsd (v_R) value is 9.5%. The fact that the results for tests with LRM glass at 90°C lie within the results for other glasses lends credence to the results of tests conducted at 40°C, both from the perspective of the suitability of the glass and the qualifications of the participants. Both the intralaboratory and interlaboratory test precision is poorer in tests at 40°C than in tests at 90°C. The intralaboratory % rsd (v_r) value is 6.8 % and the interlaboratory %rsd (v_R) value is 19%, both based on boron.

V. CONCLUSIONS

A round robin test program has been conducted to evaluate the use of a standard test material developed for use in product acceptance tests with low-activity waste glasses as a composition standard and a standard material for PCTs. The results of nine independent determinations of the glass composition showed that the analytical precision was as good as that measured previously in round robin programs with other reference glasses. The LRM glass appears to be a suitable composition standard for the following nonradioactive components present in the waste form at concentrations of 0.1 elemental mass % and higher: Al, B, Ca, Cd, Cr, F, Fe, K, La, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Si, Ti, and Zr.

Triplicate PCTs were conducted at both 40 and 90°C. The concentrations of B, Na, and Si were quantifiable in all tests. The overall variance of the PCT was measured in this study, but the procedural and analytic components of variance were not distinguished. Both the intralaboratory and interlaboratory precision for tests at 90°C were within the range measured in other studies with other glasses. The intralaboratory and interlaboratory precision for tests at 40°C were about 3X and 2X higher, respectively, than the corresponding values for tests at 90°C. For PCT with LRM glass, the expected test results at the 95% confidence level are as follows:

at 40°C:	pH = 9.86 ± 0.96	at 90°C:	pH = 10.92 ± 0.43
	[B] = 2.30 ± 1.25 mg/L		[B] = 26.7 ± 7.2 mg/L
	[Na] = 19.7 ± 7.3 mg/L		[Na] = 160 ± 13 mg/L
	[Si] = 13.7 ± 4.2 mg/L		[Si] = 82.0 ± 12.7 mg/L

These ranges can be used to evaluate the accuracy of PCTs conducted at other laboratories. A supply of LRM glass is being maintained at ANL for use in product acceptance testing of DOE waste forms.

VI. ACKNOWLEDGMENTS

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Table 1. Formulated Composition of LRM Glass [from EBERT-1998]

Chemical	mass %	Comment
Al ₂ O ₃	10	Amount common to Hanford glasses studied previously; half from waste, half as added chemical
B ₂ O ₃	8	Added chemical, high content facilitates melting
BaO	0.005	Added to LRM as RCRA-regulated component
CaO	0.5	Higher than anticipated from waste; may be used as additive in some waste forms, although Ca has been observed to decrease long-term durability
CdO	0.2	Added to LRM as RCRA-regulated component
Cl	0.8 ^a	Twice the anticipated level for desired waste loading expect 50 % loss to volatilization
Cr ₂ O ₃	0.2	Near solubility limit in borosilicate glasses
F	1	Twice the anticipated for desired waste loading; expect 50 % loss to volatilization; added amount is near solubility limit of F in borosilicate glasses
Fe ₂ O ₃	1	Only trace amounts expected in waste stream; may be additive in pretreatment or ILAW products
HgO	0.002 ^a	Expected to be completely volatilized during vitrification
I	0.002	Expected to be completely volatilized during vitrification
K ₂ O	1.5	Anticipated for desired waste loading
La ₂ O ₃	0.009	Added to LRM for use as composition standard
Li ₂ O	0.1	Added to LRM for use as composition standard; may be additive in ILAW products
MgO	0.1	Higher than expected in waste forms to facilitate analysis; may be additive in ILAW products
MnO	0.1	Added to LRM for use as composition standard
MoO ₃	0.1	Added to LRM for use as composition standard
Na ₂ O	20	Target for desired waste loading
NiO	0.1	Added to LRM for use as composition standard
P ₂ O ₅	0.5	Near solubility limit in borosilicate glasses
PbO ₂	0.1	Added to LRM as RCRA-regulated component
SO ₃	0.2	Target for desired waste loading; expected to be partially volatilized during vitrification
SiO ₂	54	Typical concentration for waste ILAW glasses
SnO ₂	0.1 ^a	May be additive in ILAW products
TiO ₂	0.1	May be additive in ILAW products
ZrO ₂	1	May be additive in ILAW products

^a The prototype glass was made with small amounts of Cl, Hg, and Sn. These elements were eliminated in target composition for the glass provided to Ferro Corp.

Table 2. Sample Preparation Methods for Composition Analysis

Analysis No.	Sample Preparation Method
A	Digestion in mixture of HNO ₃ , HCl, and HF (analyzed for Al, B, Ba, Ca, Cd, Cr, Fe, Hf, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Si, Sn, Sr, Ti, Zr)
B	Digestion in mixture of HNO ₃ , HCl, and HF (analyzed for Al, B, Ba, Ca, Cd, Cr, F, Fe, K, La, Mg, Mn, Mo, Na, Ni, P, Pb, S, Si, Sr, Ta, Th, Ti, V, Zn, Zr; for La analysis, HF was excluded and sample fumed into HClO ₄).
C1, C2	Na ₂ O ₂ /NaOH fusion in platinum crucible at 600°C followed by acidification with HCl and dilution with water (analyzed for Al, B, Li, Si).
C1, C2	LiBO ₂ fusion in platinum crucible at 900°C followed by acidification with HNO ₃ and dilution with water (analyzed for Ba, Ca, Cd, Cr, Fe, K, La, Mg, Mn, Mo, Na, Ni, P, Pb, Sn, Te, Zr).
D1, D2	CsOH•H ₂ O fusion in zirconium crucible at 500°C followed by dilution with water and added H ₂ O ₂ and acidification with HNO ₃ (analyzed for Al, B, Ba, Cd, Fe, La, Mg, Mn, Mo, Ni, P, Pb, Si, Sn).
D1, D2	CsOH•H ₂ O fusion in nickel crucible at 500°C followed by dilution with water and added H ₂ O ₂ and acidification with HNO ₃ (analyzed for Zr).
D1, D2	Na ₂ O ₂ /NaOH fusion in zirconium or nickel crucible at 675°C followed by dilution with water and added H ₂ O ₂ and acidification with HNO ₃ (analyzed for Cl and F).
D1, D2	Microwave digestion in mixture of HF and HNO ₃ in Teflon vessel followed by acidification with H ₃ BO ₃ and HCl (analyzed for Ca, Cr, Hg, K, Li, Na, Ti).
E	Microwave digestion in mixture of 5:1 vol % HNO ₃ and HF followed by dilution with water (analyzed for Al, B, Ba, Ca, Cd, Cr, Fe, Hg, K, La, Li, Mg, Mn, Mo, Na, Ni, P, Pb, S, Si, Sn, Ti, Zn).
K	Glass was analyzed directly (analyzed for Al, B, Ba, Ca, Cd, Cr, F, Fe, Hg, K, La, Mg, Mn, Mo, Na, Ni, P, Pb, S, Si, Sn, Ti, Zn).
F1	Na ₂ O ₂ /NaOH fusion in zirconium crucible followed by acidification with HNO ₃ (analyzed for Al, B, Ba, Ca, Cd, Cr, Fe, K, La, Li, Mg, Mn, Mo, Ni, P, Pb, S, Si, Sn, Ti).
F2	KOH/KNO ₃ fusion in nickel crucible followed by acidification with HNO ₃ (analyzed for Al, B, Ba, Ca, Cd, Cr, Fe, La, Li, Mg, Mn, Mo, Na, P, Pb, S, Si, Sn, Ti, Zn).
G	Microwave digestion in mixture of HNO ₃ , HCl, and HF in Teflon vessel (analyzed for Al, B, Cd, Cr, Fe, K, Li, Mg, Mn, Mo, Ni, P, Pb, Sn, Ti, Zn).
G	Microwave digestion in mixture of HNO ₃ , HCl, and HF in Teflon vessel followed by acidification with H ₃ BO ₃ and addition of EDTA (analyzed for Ca, Na, Si, Sn).
J	HF/HClO ₄ preparation (analyzed for Al, B, Ba, Ca, Cd, Cr, F, Fe, Hg, K, La, Mg, Mn, Mo, Na, Ni, P, Pb, S, Si, Sn, Ti, Zn).

Table 3. Analytical Methods Used at Different Laboratories

Element	A	B	C	D	E	K	F	G	J
Al	ICP-MS	ICP-AES	ICP-AES	ICP-ES	DCP-AES	XRF	ICP-OES	ICP-AES	ICP/DCP
B	ICP-MS	ICP-AES	ICP-AES	ICP-ES	DCP-AES	not determined	ICP-OES	ICP-AES	ICP/DCP
Ba	ICP-MS	ICP-AES	ICP-AES	ICP-ES	DCP-AES	XRF	ICP-OES	not determined	ICP/DCP
Ca	ICP-MS	ICP-AES	ICP-AES	ICP-ES	DCP-AES	XRF	ICP-OES	ICP-AES	ICP/DCP
Cd	ICP-MS	ICP-AES	ICP-AES	ICP-ES	DCP-AES	XRF	ICP-OES	ICP-AES	ICP/DCP
Cl	not determined	IC	not determined	ISE	not determined	XRF	not determined	not determined	Titration
Cr	ICP-MS	ICP-AES	ICP-AES	ICP-ES	DCP-AES	XRF	ICP-OES	ICP-AES	ICP/DCP
F	not determined	IC	not determined	ISE	not determined	not determined	ISE and XRF	not determined	Pyrohydrolysis
Fe	ICP-MS	ICP-AES	ICP-AES	ICP-ES	DCP-AES	XRF	ICP-OES	ICP-AES	ICP/DCP
Hg	not determined	not determined	not determined	Cold vapor AA	DCP-AES	XRF	not determined	not determined	ICP/DCP
I	not determined	IC	not determined	not determined	not determined	XRF	not determined	not determined	Titration
K	ICP-MS	ICP-AES	ICP-AES	Flame AA	DCP-AES	XRF	ICP-OES	ICP-AES	FES
La	not determined	ICP-AES	ICP-AES	ICP-ES	DCP-AES	XRF	ICP-OES	not determined	ICP/DCP
Li	ICP-MS	ICP-AES	ICP-AES	ICP-ES	DCP-AES	not determined	ICP-OES	ICP-AES	FES
Mg	ICP-MS	ICP-AES	ICP-AES	ICP-ES	DCP-AES	XRF	ICP-OES	ICP-AES	ICP/DCP
Mn	ICP-MS	ICP-AES	ICP-AES	ICP-ES	DCP-AES	XRF	ICP-OES	ICP-AES	ICP/DCP
Mo	ICP-MS	ICP-AES	ICP-AES	ICP-ES	DCP-AES	XRF	ICP-OES	ICP-AES	ICP/DCP
Na	ICP-MS	ICP-AES	ICP-AES	ICP-ES	DCP-AES	XRF	ICP-OES	ICP-AES	FES
Ni	ICP-MS	ICP-AES	ICP-AES	ICP-ES	DCP-AES	XRF	ICP-OES	ICP-AES	ICP/DCP
P	ICP-MS	ICP-AES	ICP-AES	ICP-ES	DCP-AES	XRF	ICP-OES	ICP-AES	ICP/DCP
Pb	ICP-MS	ICP-AES	ICP-AES	ICP-ES	DCP-AES	XRF	ICP-OES	ICP-AES	ICP/DCP
S	not determined	LECO	not determined	IC	IC	XRF	ICP-OES	not determined	LECO
Si	ICP-MS	ICP-AES	ICP-AES	ICP-ES	DCP-AES	XRF	ICP-OES	ICP-AES	ICP/DCP
Sn	ICP-MS	not determined	not determined	ICP-ES	DCP-AES	XRF	ICP-OES	ICP-AES	not determined
Ti	ICP-MS	ICP-AES	ICP-AES	ICP-ES	DCP-AES	XRF	ICP-OES	ICP-AES	ICP/DCP
Zr	ICP-MS	ICP-AES	ICP-AES	ICP-ES	DCP-AES	XRF	ICP-OES	ICP-AES	ICP/DCP

AA	= Atomic absorbance	ICP/DCP	= Inductively coupled plasma-direct coupled plasma	IC	= Ion chromatography
DCP-AES	= Direct coupled plasma- atomic emission spectroscopy	ICP-ES	= Inductively coupled plasma-emission spectroscopy	ISE	= Ion selective electrode
FES	= Flame emission spectroscopy	ICP-MS	= Inductively coupled plasma-mass spectrometry	LECO	= Combustion/infra-red spectroscopy
ICP-AES	= Inductively coupled plasma-atomic emission spectroscopy	ICP-OES	= Inductively coupled plasma-optical emission spectroscopy	XRF	= X-ray Fluorescence spectroscopy

Table 4. Compositions Measured by Participants, in mass %

	Target	A	B ^a	C1	C2	D1	D2	E1	E2
Al ₂ O ₃	10	9.33	9.901	10.1	10	9.85	9.53	8.84	9.08
B ₂ O ₃	8	8.19	7.492	8.07	8.39	7.99	7.73	7.56	7.65
BaO	0.005	0.0009	0.002	not determined	not determined	<0.005	<0.005	<0.004	<0.004
CaO	0.5	0.6	0.518	0.49	0.486	0.52	0.52	0.52	0.53
CdO	0.2	0.13	0.149	0.156	0.152	0.17	0.17	0.17	0.18
Cr ₂ O ₃	0.2	0.15	0.19	0.177	0.169	0.21	0.2	0.20	0.20
F	1	not determined	0.86	not determined	not determined	0.67	0.72	not determined	not determined
Fe ₂ O ₃	1	1.46	1.387	1.39	1.39	1.54	1.48	1.40	1.41
I	0.002	not determined	<0.02	not determined	not determined	not determined	not determined	not determined	not determined
K ₂ O	1.5	1.77	1.409	1.31	1.32	1.28	1.26	1.46	1.51
La ₂ O ₃	0.009	not determined	0.011	not determined	not determined	<0.010	<0.010	0.02	0.02
Li ₂ O	0.1	0.1	not determined	0.168	0.167	0.08	0.07	0.12	0.12
MgO	0.1	0.11	0.103	0.096	0.090	0.11	0.11	0.11	0.11
MnO	0.1	0.068	0.071	0.081	0.076	0.08	0.07	0.10	0.11
MoO ₃	0.1	0.11	0.087	not determined	not determined	0.11	0.11	0.09	0.11
Na ₂ O	20	20.2	17.792	20.6	20.2	20.49	19.91	18.08	18.44
NiO	0.1	0.16	0.178	0.182	0.164	0.19	0.19	0.18	0.19
P ₂ O ₅	0.5	0.54	0.482	0.559	0.540	0.55	0.56	0.48	0.57
PbO	0.1	0.078	0.068	0.123	0.090	0.08	0.08	0.11	0.12
SO ₃	0.2	not determined	0.230	not determined	not determined	0.4	0.47	0.33	0.34
SiO ₂	55	55.7	51.99	54.5	53.9	56.29	54.25	51.51	51.79
TiO ₂	0.1	0.11	0.102	0.087	0.09	0.10	0.09	0.11	0.11
ZrO ₂	1	0.82	0.973	0.864	0.867	1.00	1.01	0.94	0.98

^a Reported values are the average of replicate analyses.

Table 4. Contd

	Target	E3	E4	E5	F1	F2	G ^a	J	K
Al ₂ O ₃	10	9.23	9.13	8.78	9.03	9.46	9.39	9.91	9.33
B ₂ O ₃	8	7.75	7.81	7.61	7.23	7.58	8.25	7.97	8
BaO	0.005	<0.004	<0.004	<0.004	<0.01	<0.01	not determined	<0.003	<0.012
CaO	0.5	0.54	0.59	0.59	0.72	0.39	0.52	0.46	0.61
CdO	0.2	0.18	0.17	0.17	0.15	0.16	0.18	0.17	0.14
Cr ₂ O ₃	0.2	0.20	0.22	0.22	0.19	0.20	0.21	0.18	0.23
F	1	not determined	not determined	not determined	0.93	1.08	not determined	0.89	1
Fe ₂ O ₃	1	1.43	1.46	1.39	1.07	1.04	1.44	1.4	1.69
I	0.002	not determined	not determined	not determined	not determined	not determined	not determined	<0.01	<0.004
K ₂ O	1.5	1.53	1.54	1.46	1.58	not determined	1.40	1.46	1.62
La ₂ O ₃	0.009	0.02	0.02	0.02	0.02	0.01	not determined	0.016	0.03
Li ₂ O	0.1	0.12	0.13	0.13	0.10	0.12	0.09	0.1	not determined
MgO	0.1	0.11	0.12	0.12	0.10	0.10	0.10	0.11	0.09
MnO	0.1	0.11	0.13	0.13	0.06	0.08	0.08	0.093	0.09
MoO ₃	0.1	0.11	0.12	0.12	0.09	0.09	0.10	0.1	0.09
Na ₂ O	20	18.42	18.44	18.84	not determined	21.00	21.9	20.8	19.53
NiO	0.1	0.19	0.19	0.19	0.16	not determined	0.20	0.19	0.23
P ₂ O ₅	0.5	0.56	0.59	0.53	0.7	0.56	0.51	0.45	0.48
PbO	0.1	0.12	0.12	0.12	0.12	0.16	0.09	0.096	0.08
SO ₃	0.2	0.34	0.35	0.36	0.25	0.34	not determined	0.28	0.24
SiO ₂	55	52.69	51.48	52.15	53.2	54.1	55.2	55.1	55.28
TiO ₂	0.1	0.11	0.11	0.11	0.10	0.10	0.10	0.1	0.14
ZrO ₂	1	0.99	1.02	1.02	not determined	0.92	0.97	0.95	0.84

^a Reported values are the average of replicate analyses.

Table 5. Laboratory-Method Averages of Glass Composition Data, in mass %

	Target	A	B (avg of 2)	C (avg of 2)	D (avg of 2)	E (avg of 5)	F1	F2	G (avg of 3)	J	K	Consensus Average ^a
Al ₂ O ₃	10	9.33	9.901	10.05	9.69	9.01	9.03	9.46	9.39	9.91	9.33	9.51
B ₂ O ₃	8	8.19	7.492	8.23	7.86	7.68	7.23	7.58	8.25	7.97	8	7.85
BaO	0.005	0.0009	0.002	nd ^b	nd	nd	nd	nd	nd	nd	nd	0.0015
CaO	0.5	0.6	0.518	0.488	0.52	0.56	0.72	0.39	0.52	0.46	0.61	0.54
CdO	0.2	0.13	0.149	0.154	0.17	0.17	0.15	0.16	0.18	0.17	0.14	0.16
Cr ₂ O ₃	1	0.15	0.19	0.173	0.205	0.21	0.19	0.20	0.21	0.18	0.23	0.19
F	1	nd ^b	0.86	nd	0.695	nd	nd	nd	nd	0.89	1	0.86
Fe ₂ O ₃	1	1.46	1.387	1.39	1.51	1.422	1.07	1.04	1.44	1.4	1.69	1.38
I	0.002	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
K ₂ O	1.5	1.77	1.409	1.315	1.27	1.5	1.58	nd	1.4	1.46	1.62	1.48
La ₂ O ₃	0.009	nd	0.011	nd	nd	0.02	0.02	0.01	nd	0.016	0.03	0.02
Li ₂ O	0.1	0.1	nd	0.1675	0.075	0.12	0.10	0.12	0.09	0.1	nd	0.11
MgO	0.1	0.11	0.103	0.093	0.11	0.12	0.10	0.10	0.1	0.11	0.09	0.10
MnO	0.1	0.068	0.071	0.0785	0.075	0.11	0.06	0.08	0.08	0.093	0.09	0.08
MoO ₃	0.1	0.11	0.087	nd	0.11	0.11	0.09	0.09	0.1	0.1	0.09	0.10
Na ₂ O	20	20.2	17.792	20.4	20.2	18.44	nd	21.00	21.9	20.8	19.53	20.03
NiO	0.1	0.16	0.178	0.173	0.19	0.19	0.16	nd	0.2	0.19	0.23	0.19
P ₂ O ₅	0.5	0.54	0.482	0.5495	0.555	0.54	0.7	0.56	0.51	0.45	0.48	0.54
PbO	0.1	0.078	0.068	0.1065	0.08	0.12	0.12	0.16	0.09	0.096	0.08	0.10
SO ₃	0.2	nd	0.23	nd	0.435	0.34	0.25	0.34	nd	0.28	0.24	0.30
SiO ₂	55	55.7	51.99	54.2	55.27	51.94	53.2	54.1	55.2	55.1	55.28	54.20
TiO ₂	0.1	0.11	0.102	0.0885	0.095	0.11	0.10	0.10	0.1	0.1	0.14	0.10
ZrO ₂	1	0.82	0.973	0.8655	1.005	0.99	nd	0.92	0.97	0.95	0.84	0.93
Total	100.616	99.627	93.995	98.522	100.120	93.702	74.87	96.41	100.73	100.825	99.74	98.764

^a The average was calculated over the laboratory-method combinations.^b "nd" indicates concentration was either below the detection limit or not measured.

Table 6. Deviation from Average, d_{ij} ^a of Glass Composition Data

	A	B (avg of 2)	C (avg of 2)	D (avg of 2)	E (avg of 5)	F1	F2	G (avg of 3)	J	K
Al ₂ O ₃	-0.18	0.39	0.54	0.18	-0.50	-0.48	-0.05	-0.12	0.40	-0.18
B ₂ O ₃	0.34	-0.36	0.38	0.01	-0.17	-0.62	-0.27	0.40	0.12	0.15
BaO	0.00	0.00	nd ^b	nd	nd	nd	nd	nd	nd	nd
CaO	0.06	-0.02	-0.05	-0.02	0.02	0.18	-0.15	-0.02	-0.08	0.07
CdO	-0.03	-0.01	0.00	0.01	0.01	-0.01	0.00	0.02	0.01	-0.02
Cr ₂ O ₃	-0.04	0.00	-0.02	0.01	0.02	0.00	0.01	0.02	-0.01	0.04
F	nd	0.00	nd	-0.17	nd	nd	nd	nd	0.03	0.14
Fe ₂ O ₃	0.08	0.01	0.01	0.13	0.04	-0.31	-0.34	0.06	0.02	0.31
I	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
K ₂ O	0.29	-0.07	-0.17	-0.21	0.02	0.10	-1.48	-0.08	-0.02	0.14
La ₂ O ₃	nd	-0.01	nd	nd	0.00	0.00	-0.01	nd	0.00	0.01
Li ₂ O	-0.01	nd	0.06	-0.03	0.01	-0.01	0.01	-0.02	-0.01	nd
MgO	0.01	0.00	-0.01	0.01	0.02	0.00	0.00	0.00	0.01	-0.01
MnO	-0.01	-0.01	0.00	-0.01	0.03	-0.02	0.00	0.00	0.01	0.01
MoO ₃	0.01	-0.01	nd	0.01	0.01	-0.01	-0.01	0.00	0.00	-0.01
Na ₂ O	0.17	-2.24	0.37	0.17	-1.59	nd	0.97	1.87	0.77	-0.50
NiO	-0.03	-0.01	-0.01	0.00	0.00	-0.03	nd	0.01	0.00	0.04
P ₂ O ₅	0.00	-0.05	0.01	0.02	0.00	0.16	0.02	-0.03	-0.09	-0.06
PbO	-0.02	-0.03	0.01	-0.02	0.02	0.02	0.06	-0.01	0.00	-0.02
SO ₃	nd	-0.07	nd	0.13	0.04	-0.05	0.04	nd	-0.02	-0.06
SiO ₂	1.50	-2.21	0.00	1.07	-2.26	-1.00	-0.10	1.00	0.90	1.08
TiO ₂	0.01	0.00	-0.02	-0.01	0.01	0.00	0.00	0.00	0.00	0.04
ZrO ₂	-0.11	0.05	-0.06	0.08	0.06	nd	-0.01	0.04	0.02	-0.09

^a d_{ij} = average for participant - consensus average.^b "nd" indicates deviation not calculated because concentration was either below the detection limit or not measured.

Table 7. Precision Summary for Glass Composition Data, in mass %

	($\bar{x}$) ^a	(s_r) ^b	(s_x) ^c	(s_L) ^d	(s_R) ^e	v_r (%) ^f	v_L (%) ^g	v_R (%) ^h	I(r) ⁱ	I(R) ^j
Al ₂ O ₃	9.51	0.119	0.346	0.336	0.356	1.25	3.53	3.75	0.335	1.008
B ₂ O ₃	7.85	0.146	0.327	0.311	0.343	1.86	3.96	4.37	0.413	0.971
BaO	0.00	nd ^k	0.001	nd	nd	nd	nd	nd	nd	nd
CaO	0.54	0.003	0.086	0.086	0.086	0.53	15.99	15.93	0.008	0.244
CdO	0.16	0.003	0.015	0.015	0.015	1.80	9.31	9.38	0.008	0.042
Cr ₂ O ₃	0.19	0.005	0.021	0.021	0.022	2.34	10.91	11.58	0.013	0.061
F	0.86	0.035	0.109	0.106	0.112	4.11	12.35	13.02	0.100	0.317
Fe ₂ O ₃	1.38	0.042	0.184	0.181	0.186	3.07	13.12	13.48	0.120	0.526
I	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
K ₂ O	1.48	0.008	0.489	0.489	0.489	0.53	33.01	33.04	0.022	1.383
La ₂ O ₃	0.02	nd	0.008	nd	nd	nd	nd	nd	nd	nd
Li ₂ O	0.11	0.004	0.026	0.026	0.026	3.26	23.76	23.64	0.010	0.074
MgO	0.10	0.004	0.009	0.008	0.009	4.10	7.71	9.00	0.012	0.026
MnO	0.08	0.004	0.013	0.013	0.014	4.91	16.38	17.50	0.011	0.039
MoO ₃	0.10	nd	0.009	nd	nd	nd	nd	nd	nd	nd
Na ₂ O	20.03	0.249	1.202	1.189	1.215	1.24	5.94	6.07	0.705	3.439
NiO	0.19	0.013	0.020	0.018	0.022	6.86	9.89	11.58	0.036	0.063
P ₂ O ₅	0.54	0.008	0.065	0.065	0.065	1.41	12.06	12.04	0.021	0.184
PbO	0.10	0.023	0.026	0.020	0.031	23.37	20.39	31.00	0.066	0.088
SO ₃	0.30	0.049	0.068	0.059	0.077	16.38	19.47	25.67	0.140	0.218
SiO ₂	54.20	0.752	1.318	1.206	1.421	1.39	2.23	2.62	2.128	4.022
TiO ₂	0.10	0.004	0.013	0.013	0.013	3.53	12.42	13.00	0.010	0.038
ZrO ₂	0.93	0.004	0.064	0.064	0.065	0.40	6.95	6.99	0.010	0.183

See next page for footnotes.

Footnotes for Table 7.

^a ($\bar{x}$) is the consensus average of pH, Al, B, Na, or Si concentration, in mg/L.

^b (s_r) is the pooled intralaboratory standard deviation. It gives the estimated repeatability standard deviation for tests within the same laboratory.

$$s_r = [(\sum s^2) / p]^{1/2}$$

^c (s_x) is the standard deviation of replicate tests by the same participant, with a mean $\bar{x}_p$, from the consensus average.

$$s_x = [\sum (\bar{x}_p - \bar{x})^2 / (n-1)]^{1/2}$$

^d (s_L) is the estimated interlaboratory variability, expressed as the square root of the component of variance.

$$s_L = [s_x^2 - (s_r^2 / n)]^{1/2}$$

^e (s_R) is the interlaboratory estimate of precision (reproducibility standard deviation).

$$s_R = (s_r^2 - s_L^2)^{1/2}$$

^f v_r (%) is the estimated intralaboratory relative standard deviation, or coefficient of variation.

$$v_r = 100 \cdot s_r / \bar{x}$$

^g v_L (%) is the estimated interlaboratory relative standard deviation, or coefficient of variation, expressed in terms of the square root of the component of variance.

$$v_L = 100 \cdot s_L / \bar{x}$$

^h v_R (%) is the estimated interlaboratory precision, expressed in terms of the square root of the component of variance.

$$v_R = 100 \cdot s_R / \bar{x}$$

ⁱ $I(r)$ is the estimated 95% repeatability level. On the basis of test error alone, the absolute value of the difference of two test results obtained in the same laboratory will exceed $I(r)$ only approximately 5% of the time.

$$I(r) = 2.83 \cdot s_L$$

^j $I(R)$ is the estimated 95% reproducibility level. On the basis of test error alone (including intra- and interlaboratory components), the absolute value of the difference of two test results obtained in different laboratories will exceed $I(R)$ only approximately 5% of the time.

$$I(R) = 2.83 \cdot s_R$$

^k "nd" means not determined.

Table 8. Difference in Target and Measured Glass Compositions, in mass %

	Target	Average	±	I(R)	Difference ^a	Significant? ^b
Al ₂ O ₃	10	9.51	±	1.01	-0.49	No
B ₂ O ₃	8	7.85	±	0.97	-0.15	No
BaO	0.005	0.0015	±	0.002 ^c	-0.0035	Yes
CaO	0.5	0.54	±	0.24	0.04	No
CdO	0.2	0.16	±	0.04	-0.14	Yes
Cr ₂ O ₃	1	0.19	±	0.06	-0.81	Yes
F	1	0.86	±	0.32	-0.14	No
Fe ₂ O ₃	1	1.38	±	0.53	0.38	No
I	0.002	nd ^d	±	nd	nd	nd
K ₂ O	1.5	1.48	±	1.38	-0.02	No
La ₂ O ₃	0.009	0.02	±	0.02 ^a	0.011	No
Li ₂ O	0.1	0.11	±	0.07	0.01	No
MgO	0.1	0.10	±	0.03	0	No
MnO	0.1	0.08	±	0.04	-0.02	No
MoO ₃	0.1	0.10	±	0.03 ^a	0	No
Na ₂ O	20	20.03	±	3.44	0.03	No
NiO	0.1	0.19	±	0.06	0.09	Yes
P ₂ O ₅	0.5	0.54	±	0.18	0.04	No
PbO	0.1	0.10	±	0.09	0	No
SO ₃	0.2	0.30	±	0.22	0.1	No
SiO ₂	55	54.20	±	4.02	-0.8	No
TiO ₂	0.1	0.10	±	0.04	0	No
ZrO ₂	1	0.93	±	0.18	-0.07	No

^a Calculated as average – target.^b Significant if absolute value of Difference > I(R).^c Calculated as 2.83•s_x.^d “nd” indicates concentration was below detection limit or not measured.

Table 9. Precision of Measurements of Element Concentrations
in LRM (v_R), and in ARM-1, EA, and WV6 Glasses

Element	Percent Relative Standard Deviation			
	LRM	ARM-1	EA	WV6
Al	3.75^a	10.4	24.5	26.6
B	4.37	6.11	3.20	2.61
Ba	nd ^b	11.6	200	17.6
Ca	15.9	10.9	20.2	18.7
Cd	9.38	- ^c	-	-
Cr	11.6	-	192	15.9
F	13.0	-	-	-
Fe	13.5	-	14.8	14.6
I	nd	-	-	-
K	33.0	-	115	14.0
La	40.0 ^d	-	32.7	47.6
Li	23.6	5.58	11.8	11.5
Mg	9.00	-	2.86	22.3
Mn	17.5	-	15	18.3
Mo	9.00 ^d	6.46	-	-
Na	6.07	14.9	12.2	5.13
Ni	11.6	-	17.7	13.9
P	12.0	24.6	354	42.6
Pb	31.0	-	-	-
S	25.7	-	-	-
Si	2.62	13.9	4.41	3.50
Ti	13.0	7.44	22.3	24.4
Zr	6.99	10.2	8.44	15.2

^a Values in bold are minimum values (highest precision) for each element.

^b "nd" means not determined.

^c "-" means not reported.

^d Calculated with s_x .

Table 10. Effect of Amounts of Glass and Water at Mass Ratio 10 on PCT Response at 40°C

Test No.	$g_{\text{Glass}}/g_{\text{Water}}$	pH	[B], mg/L	[Na], mg/L	[Si], mg/L
LRM-1/7	1.0:10	9.72	1.19	16.1	12.0
LRM-1/8	1.0:10	9.54	1.16	16.2	12.2
LRM-1/9	1.0:10	9.65	1.12	15.5	11.6
mean $\pm$ s		9.64 $\pm$ 0.09	1.16 $\pm$ 0.03	15.9 $\pm$ 0.38	11.9 $\pm$ 0.30
LRM-1/10	0.80:8.0	9.85	1.22	15.8	12.4
LRM-1/11	0.80:8.0	9.70	1.09	15.2	11.6
LRM-1/12	0.80:8.0	9.87	0.99	14.4	10.9
mean $\pm$ s		9.81 $\pm$ 0.09	1.10 $\pm$ 0.11	15.1 $\pm$ 0.70	11.6 $\pm$ 0.75
LRM-1/13	1.2:12	9.84	1.07	15.1	11.2
LRM-1/14	1.2:12	9.80	1.11	15.7	11.8
LRM-1/15	1.2:12	9.93	0.97	14.7	10.6
mean $\pm$ s		9.86 $\pm$ 0.07	1.05 $\pm$ 0.07	15.2 $\pm$ 0.50	11.2 $\pm$ 0.60
Overall mean $\pm$ s		9.77 $\pm$ 0.12	1.10 $\pm$ 0.06	15.4 $\pm$ 0.44	11.6 $\pm$ 0.35

Table 11. Results of PCTs at 40°C

Test Number	pH	[Al], mg/L	[B], mg/L	[Na], mg/L	[Si], mg/L	Test Number	pH	[Al], mg/L	[B], mg/L	[Na], mg/L	[Si], mg/L
A-1	10.24	2.41	1.94	21.94	14.79	E-1	9.40	1.865	2.761	17.966	14.805
A-2	10.22	2.30	2.10	22.54	16.00	E-2	9.42	1.914	2.981	17.962	5.151
A-3	10.25	2.34	2.12	22.84	15.59	E-3	9.40	1.900	2.667	18.021	14.831
A-blank1	6.65	<0.1	<0.1	<0.07	<0.08	E-4	9.41	1.907	2.529	17.996	14.790
A-blank2	6.27	<0.1	<0.1	<0.07	<0.08	E-5	9.41	1.914	2.503	17.828	14.943
B-1	9.80	2.36	1.95	18.3	12.2	E-6	9.42	20.89	3.18	17.784	14.729
B-2	9.96	2.34	2.09	19.6	13.4	E-blank1	6.03	<0.1054	<0.5368	<0.0949	0.503
B-3	9.92	2.19	1.95	19.4	12.9	E-blank2	6.05	<0.1054	<0.5368	<0.0949	0.319
B-blank1	>7	0.252	0.130	<0.1	<0.5	F-1	9.91	2.2	2.4	20	12
B-blank2	>7	<0.2	0.110	<0.1	<0.5	F-2	9.90	2.1	2.3	20	12
C-1	9.91	nd ^b	2.77	23.2	13.8	F-3	9.90	2.1	2.3	20	12
C-2	9.98	nd	2.53	21.3	12.9	F-blank1	5.96	0.06	<0.01	0.12	<0.01
C-3	9.96	nd	2.48	22.3	13.7	F-blank2	5.84	<0.4	<0.01	0.10	<0.01
C-blank1	6.78	nd	0.302	1.16	<0.3	G-1	9.3	1.9	1.5	15	8.2
C-blank2	6.76	nd	<0.3	<0.9	<0.3	G-2	9.3	1.4	1.5	16	7.8
D-1	9.95	nd	2.57	21.3	13.7	G-3	9.4	1.6	1.5	15	8.3
D-2	9.99	nd	2.37	21.2	11.8	G-blank ^a	5.5	<0.015	0.090	<0.022	<0.010
D-3	10.00	nd	2.47	22.3	12.1	H-1	10.21	2.473	2.361	17.19	13.87
D-blank1	6.78	nd	0.302	1.16	<0.3	H-2	10.19	2.707	2.493	18.08	14.45
D-blank2	6.76	nd	<0.3	<0.9	<0.3	H-3	10.20	2.423	2.834	19.16	15.54
						H-4	10.21	2.651	2.917	19.68	15.65
						H-blank1		<0.002	<0.001	<0.02	<0.02
						H-blank2		<0.002	<0.001	0.04	<0.02

^a Average value for two tests reported by participant.^b "nd" means not determined.

Table 12. Results of PCTs at 90°C

Test Number	pH	[Al], mg/L	[B], mg/L	[Na], mg/L	[Si], mg/L	Test Number	pH	[Al], mg/L	[B], mg/L	[Na], mg/L	[Si], mg/L
A-1	10.98	13.7	24.71	170.47	82.81	E-1	10.80	13.206	27.860	141.124	85.663
A-2	11.00	13.4	25.89	174.29	84.22	E-2	10.82	13.181	27.924	148.792	85.526
A-3	10.95	13.6	24.31	175.48	82.50	E-3	10.81	13.393	28.196	144.292	86.020
A-blank1	6.42	<0.1	<0.1	<0.07	<0.08	E-4	10.81	13.456	27.846	142.330	85.673
A-blank2	6.53	<0.1	<0.1	<0.07	<0.08	E-5	10.80	13.410	27.704	143.039	86.167
B-1	10.95	14.1	22.9	155	83.8	E-6	10.82	13.168	27.563	142.854	85.399
B-2	11.03	15.0	22.6	164	84.9	E-blank1	6.12	<0.1054	<0.5368	<0.0949	1.280
B-3	11.02	14.3	22.4	156	85.7	E-blank2	6.14	<0.1054	<0.5368	<0.0949	1.321
B-blank1	<7	0.260	0.119	<0.1	<0.5	F-1	11.00	12	28	160	78=
B-blank2	<7	0.288	0.136	<0.1	<0.5	F-2	10.98	12	26	150	76
C-1	11.02	nd ^b	29.2	168	82.7	F-3	10.96	12	26	150	75
C-2	11.02	nd	28.5	170	85.7	F-blank1	5.80	<0.04	<0.01	0.086	<0.01
C-3	11.04	nd	30.0	170	84.0	F-blank2	5.75	<0.04	<0.01	0.098	<0.01
C-blank1	6.72	nd	<0.3	1.40	<0.3	G-1	10.6	13	26	150	74
C-blank2	6.82	nd	<0.3	<0.9	<0.3	G-2	10.6	14	26	150	77
D-1	11.04	nd	30.2	162	82.7	G-3	10.6	13	26	150	75
D-2	11.06	nd	29.7	173	84.7	G-blank ^a	6.4	<0.015	0.044	<0.022	<0.010
D-3	11.06	nd	29.2	175	85.5	H-1	11.01	16.51	32.18	204.1	102.1
D-blank1	6.72	nd	<0.3	1.40	<0.3	H-2	10.88	20.14	37.62	201.2	116.7
D-blank2	6.82	nd	<0.3	<0.9	<0.3	H-3	10.89	20.24	39.04	201.4	116.5
						H-blank1		<0.02	<0.01	0.053	<0.02
						H-blank2		<0.02	<0.01	0.038	<0.02

^a Average value for two tests reported by participant.^b "nd" means not determined.

Table 13. Mean Values for Results of PCTs with LRM Glass at 40°C and 90°C ^{a, b}

	A	B	C	D	E	F	G	H	Consensus Average ^c
PCTs with LRM Glass at 40°C									
pH	10.24	9.89	9.95	9.98	9.41	9.90	9.30	10.20	9.86
Al	2.35	2.30	nd ^d	nd	1.93	2.13	1.6	2.56	2.151
B	2.05	2.00	2.59	2.47	2.77	2.33	1.5	2.65	2.296
Na	22.4	19.1	22.3	21.6	17.9	20.0	15	18.5	19.65
Si	15.5	12.8	13.5	12.5	14.9	12.0	(8.1)	14.9	13.72
Blank Tests at 40°C									
pH	6.46	<7	6.77	6.77	6.04	5.90	5.50	nd	
Al	<0.1	0.252	nd	nd	<0.1	<0.04	<0.02	<0.02	
B	<0.1	0.12	<0.2	<0.2	<0.6	<0.01	0.09	<0.01	
Na	<0.07	<0.1	<0.6	<0.6	<0.1	0.11	<0.03	0.04	
Si	<0.08	<0.5	<0.2	<0.2	0.4	<0.01	<0.01	<0.02	
PCTs with LRM Glass at 90°C									
pH	10.98	11.00	11.03	11.05	10.81	10.98	10.60	10.93	10.92
Al	13.6	14.5	nd	nd	13.3	12.0	13	19.0	14.27
B	25.0	22.6	29.2	29.7	27.9	26.7	26	(36.3)	26.72
Na	173	158	169	170	143	153	150	(202)	159.7
Si	83.2	84.8	84.1	84.3	85.7	76.3	75	(112)	81.97
Blank Tests at 90°C									
pH	6.48	<7	6.77	6.77	6.13	5.78	6.4	nd	
Al	<0.1	0.27	nd	nd	<0.1	<0.04	<0.02	<0.02	
B	<0.1	0.13	<0.2	<0.2	<0.6	<0.01	0.04	<0.01	
Na	<0.07	<0.01	<0.6	<0.6	<0.1	0.09	<0.03	0.04	
Si	<0.08	<0.5	<0.2	<0.2	1.3	<0.01	<0.01	<0.02	

^a Concentrations of Al, B, Na, and Si in mg/L.^b Each cell contains the average for replicate tests by that participant ($\bar{x}_p$).^c The consensus average was calculated from the averages of the independent tests. The results in parentheses were excluded from the consensus average as outliers.^d "nd" means not determined.

Table 14. Standard Deviations for Replicate PCT Results for Each Participant^{a, b}

	A	B	C	D	E	F	G	H	(s _r) ^c
PCT at 40°C									
pH	0.015	0.083	0.036	0.026	0.009	0.006	0.058	0.010	0.0399
Al	0.056	0.093	nd ^d	nd	0.079	0.058	0.252	0.137	0.1313
B	0.099	0.081	0.151	0.100	0.266	0.058	0.000	0.267	0.1558
Na	0.458	0.700	0.918	0.631	0.097	0.000	0.577	1.115	0.6640
Si	0.615	0.603	0.537	1.033	0.152	0.000	(0.265) ^e	0.861	0.6395
PCT at 90°C									
pH	0.025	0.044	0.012	0.012	0.009	0.020	0.000	0.072	0.0326
Al	0.153	0.473	nd	nd	0.131	0.000	0.577	2.122	0.9221
B	0.821	0.252	0.752	0.500	0.214	1.155	0.000	(3.621)	0.6472
Na	2.618	4.933	0.962	6.883	1.620	5.774	0.000	(1.622)	4.0609
Si	0.917	0.954	1.503	1.456	0.294	1.528	1.528	(8.339)	1.2469

^a Values in the cells are the standard deviations (s) for n replicate tests by the same participant:

$$s = [\Sigma(x - \bar{x}_p)^2 / (n - 1)]^{1/2}$$

^b Concentrations of Al, B, Na, and Si in mg/L.

^c (s_r) is the estimated intralaboratory standard deviation for p participants:

$$s_r = (\Sigma s/p)^{1/2}$$

^d "nd" means not determined.

^e Data in parentheses were determined to be outliers and excluded from the calculation of s_r.

Table 15. Deviation of PCT Results of Participants from Consensus Average^{a, b}

	A	B	C	D	E	F	G	H	(s _x) ^c
PCT at 40°C									
pH	0.38	0.03	0.09	0.12	-0.45	0.04	-0.56	0.34	0.3383
Al	0.20	0.15	nd	nd	-0.21	-0.01	-0.55	0.42	0.3412
B	-0.24	-0.30	0.29	0.17	0.47	0.04	-0.80	0.36	0.4225
Na	2.75	-0.55	2.65	1.95	-1.75	0.35	-4.65	-1.15	2.5304
Si	1.78	-0.92	-0.22	-1.22	1.18	-1.72	(-5.62) ^d	1.18	1.3719
PCT at 90°C									
pH	0.05	0.08	0.11	0.13	-0.11	0.06	-0.32	0.01	0.1498
Al	-0.67	0.23	nd	nd	-0.97	-2.27	-1.27	4.73	2.4731
B	-1.72	-4.12	2.48	2.98	1.18	-0.02	-0.72	(9.58)	2.4791
Na	13.30	-1.70	9.30	10.3	-16.7	-6.70	-9.70	(42.3)	11.476
Si	1.23	2.83	2.13	2.33	3.73	-5.67	-6.97	(30.0)	4.3614

^a Deviation = laboratory average – consensus average.^b Concentrations of Al, B, Na, and Si in mg/L.^c The values (s_x) are the estimated interlaboratory standard deviations: $s_x = [\sum d^2 / (p-1)]^{1/2}$ ^d Data in parentheses were determined to be outliers and excluded from the calculation of s_x.

Table 16. Summary of Test Precision for PCTs

	($\bar{x}$) ^a	(s_r) ^b	(s_x) ^c	(s_L) ^d	(s_R) ^e	$v_r(\%)$ ^f	$v_L(\%)$ ^g	$v_R(\%)$ ^h	I(r) ⁱ	I(R) ^j
PCT at 40°C										
pH	9.864	0.040	0.338	0.338	0.340	0.404	3.422	3.446	0.113	0.962
Al	2.151	0.131	0.341	0.333	0.358	6.102	15.46	16.62	0.372	1.012
B	2.296	0.156	0.423	0.413	0.441	6.785	17.98	19.22	0.441	1.249
Na	19.65	0.664	2.530	2.501	2.588	3.379	12.73	13.17	1.879	7.324
Si	13.72	0.640	2.372	1.321	1.468	4.661	9.631	10.70	1.810	4.154
PCT at 90°C										
pH	10.92	0.033	0.150	0.149	0.152	0.299	1.361	1.393	0.092	0.431
Al	14.27	0.922	2.415	2.415	2.585	6.461	16.92	18.11	2.610	7.316
B	26.72	0.647	2.479	2.451	2.535	2.423	9.174	9.488	1.832	7.173
Na	159.7	4.061	11.48	11.23	11.95	2.543	7.034	7.480	11.49	33.81
Si	81.97	1.247	4.36	4.302	4.479	1.521	5.248	5.464	3.529	12.67

See next page for footnotes.

Footnotes for Table 16

^a ($\bar{x}$) is the consensus average of pH or the Al, B, Na, and Si concentrations, in mg/L.

^b (s_r) is the pooled intralaboratory standard deviation. It gives the estimated repeatability standard deviation for tests within the same laboratory.

$$s_r = [(\sum s^2) / p]^{1/2}$$

^c (s_x) is the standard deviation of replicate tests by the same participant, with a mean $\bar{x}_p$, from the consensus average.

$$s_x = [\sum (\bar{x}_p - \bar{x})^2 / (n-1)]^{1/2}$$

^d (s_L) is the estimated interlaboratory variability, expressed as the square root of the component of variance.

$$s_L = [(s_x^2 - (s_r^2/n))]^{1/2}$$

^e (s_R) is the interlaboratory estimate of precision (reproducibility standard deviation).

$$s_R = (s_r^2 - s_L^2)^{1/2}$$

^f v_r (%) is the estimated intralaboratory relative standard deviation, or coefficient of variation.

$$v_r = 100 \bullet s_r / \bar{x}$$

^g v_L (%) is the estimated interlaboratory relative standard deviation, or coefficient of variation, expressed in terms of the square root of the component of variance.

$$v_L = 100 \bullet s_L / \bar{x}$$

^h v_R (%) is the estimated interlaboratory precision, expressed in terms of the square root of the component of variance.

$$v_R = 100 \bullet s_R / \bar{x}$$

ⁱ $I(r)$ is the estimated 95% repeatability level. On the basis of test error alone, the absolute value of the difference of two test results obtained in the same laboratory will exceed $I(r)$ only approximately 5% of the time.

$$I(r) = 2.83 \bullet (s_r)$$

^j $I(R)$ is the estimated 95% reproducibility level. On the basis of test error alone (including intra- and interlaboratory components), the absolute value of the difference of two test results obtained in different laboratories will exceed $I(R)$ only approximately 5% of the time.

$$I(R) = 2.83 \bullet (s_R)$$

Table 17. Results of Replicate Analyses of PCT Solutions^a

PCT at 40°C				
Element	Participant E Analysis 1	Participant E Analysis 2	Participant E Overall	All Participants Overall
Al	1.93 ± 0.08	1.96 ± 0.03	1.95 ± 0.06	2.15 ± 0.31
B	2.77 ± 0.27	1.87 ± 0.28	2.32 ± 0.54	2.30 ± 0.40
Na	17.9 ± 0.1	17.7 ± 0.2	17.8 ± 0.2	19.7 ± 2.3
Si	14.9 ± 0.2	14.0 ± 0.1	14.5 ± 0.5	13.0 ± 2.2
PCT at 90°C				
Element	Participant E Analysis 1	Participant E Analysis 2	Participant E Overall	All Participants Overall
Al	13.3 ± 0.01	13.6 ± 0.1	13.5 ± 0.2	14.3 ± 2.2
B	27.9 ± 0.1	27.4 ± 0.2	27.6 ± 0.3	27.9 ± 3.8
Na	143 ± 2	148 ± 2	145 ± 3	165 ± 17
Si	85.7 ± 0.3	79.3 ± 0.5	82.5 ± 3.4	85.7 ± 10.5

^a Cell values give mean ± s, in mg/L.

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Fig. 1. Plot of s_r (●), s_L (◆), and s_R (■) vs. the Average Concentrations of Various Glass Components

Fig. 2. Percent Relative Standard Deviation vs. Mean Oxide Mass Percent Measured in Interlaboratory Studies with (a) ARM-1, (b) EA, (c) WV6, and (d) LRM Glasses

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Fig. 4. Mean and Standard Deviation of Concentrations of B, Na, and Si for PCTs Conducted by Eight Participants at (a) 40°C and (b) 90°C. The mean and standard deviations of the mean values for each participant are shown on the far right. The averages of the means are shown by the horizontal lines.

Fig. 5. Plot of s_r (●), s_L (◆), and s_R (■) vs. the Average Values Measured in PCTs at 40 and 90°C

Fig. 6. Calculated H Parameters for Al (shaded bars), B (filled bars), Na (open bars), and Si (cross-hatched bars) in PCTs at 40 and 90°C. The horizontal lines at $H = \pm 2.15$ identify the critical value for eight participants.

APPENDIX A. Participating Laboratories and Contributors

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(contact: D. D'Anna, analysis of PCT solutions)

ANL Analytical Chemistry Laboratory,

(contact: D. G. Graczyk, analysis of glass composition and PCT solutions)

CELS Corning Laboratory Services, Corning, NY

(contact: L. Adams, analysis of glass composition)

APPENDIX B. Letters to Participants

First Letter to Prospective Participants

Dear Colleague,

You are invited to participate in a round robin testing program that we are conducting under the auspices of the DOE Tanks Focus Area (TFA) for Immobilization. (The Technical Implementation Manager for the Immobilization area is Bill Holtzscheiter-WSRC-STRC). The round robin consists of two separate parts. The first part is being carried out to determine the interlaboratory precision of a test method when conducted with a standard test material to document the response of that material in that test. The test method is based on the Product Consistency Test (PCT) that has been standardized by the American Society for Testing and Materials [ASTM-1285]. (A copy of the ASTM PCT procedure will be provided with the glass.) The test method is being developed for use as an acceptance test for Immobilized Low-Activity Waste (ILAW) products. The second part of the round-robin addresses compositional analysis of the glass. This will require dissolution of the glass by your method(s) of choice followed by chemical analysis. Your participation will support the effort of DOE to develop a meaningful test method and standard for use in ILAW acceptance while at the same time providing your laboratory experience in conducting the test that will likely be used in qualification of ILAW products. It is likely that all contracting laboratories will have to be "certified" to perform acceptance testing for ILAW products. You are invited to participate in one or both parts of the round-robin.

The glass to be used in these tests is a borosilicate glass that contains about 20 mass % Na_2O and small amounts of other components that are either present in DOE waste streams or may be added to ILAW products. The glass also contains small amount of the RCRA metals Ba, Cd, Cr, and Pb. We have shown the glass itself to be non-hazardous per the TCLP. (A paper describing the glass composition and the results of tests conducted with the prototype glass will be provided with the glass [EBERT-1998].) A supply of 1000 lbs. of glass with the composition as the prototype glass was made by Ferro Corp. We have mixed, crushed, sieved, and washed the glass to prepare a single source of glass for testing in the round robin program. The -100 +200 mesh size fraction was isolated for use and washed to remove fines. About 17 g of glass will be provided to each participant for use in the round robin. This amount will be sufficient to run two sets of triplicate tests at each temperature and to provide composition analysis.

The procedure for PCT Method A is to be followed for tests at both 40 and 90°C, except that tests with a standard reference glass are not required. A copy of the ASTM PCT procedure will be provided. Key aspects of the testing are summarized below:

- Three replicate tests are to be conducted at both 40 and 90°C with between 1 to 1.5 g of crushed glass and a mass of demineralized water (ASTM Type I water) that is exactly 10 times the mass of glass used. For example, if 1.15 g of glass is used, then 11.50 ± 0.02 g of demineralized water must be used.
- Tests are to be conducted for 7-day test durations. The times of day a test vessel is placed in the oven and removed from the oven must agree within 3.4 hours.

- The use of unsensitized Type 304L stainless steel vessels is preferred, but tests may be conducted with TFE-Teflon vessels if steel vessels are not available. The vessel type used in the tests must be reported. Vessels must be cleaned according to the PCT procedure.
- Duplicate blank tests with demineralized water are to be conducted simultaneously with the tests with glass. The vessels used in the blank tests must have been cleaned in the same batch as the vessels used in the tests with glass.
- The oven temperature must remain within 2°C of the desired test temperature throughout the test.
- The total mass of the test vessel must not change during the test by more than 5 % of the mass of demineralized water that was initially added to the vessel.
- Solutions generated in the tests with glass and the blanks tests are to be filtered through 0.45-µm pore size filters, acidified with concentrated nitric acid, and then analyzed for B, Na, and Si. Estimated detection limits must be reported.
- A model PCT data sheet is provided in Fig. X2 of the ASTM procedure. Other data sheets containing the test information can be used.
- While a particular analytical method is not required for solution analysis, the method(s) used must be reported.

Tests conducted by different scientists and solutions analyzed with different instruments will be treated as separate determinations. To provide the largest possible data base for the PCT, we encourage you to conduct separate determinations, if possible. No restrictions are placed on the method used to dissolve the glass for chemical analysis, except that the method used to dissolve and analyze the glass must be reported with the analytical results. The target composition of the glass is provided in Table 1 to facilitate selection of the method and analysis.

The concentrations measured at different laboratories will be analyzed to measure the reproducibility of the test method and measurement of the glass composition. These results will be documented in a report to the TFA and may be used by DOE in future privatization contracts. We also plan to present and publish the results at the American Ceramic Society (ACerS) meeting in Indianapolis, IN, on April 25-28, 1999. As has been done in previous round-robins, participating laboratories will be recognized in all reports, but the results will not be linked to the laboratories. Please include desired wording to be included in acknowledgements of laboratories and contributors with reported results.

Test results must be reported to ANL before March 31, 1999 to allow compilation and review of the results for presentation at the ACerS meeting. Participants will be provided with the compiled test results to verify correct transcription prior to communication or presentation of those results. If you are able to participate, and if you know of other scientists who may be interested in participating, please contact me by phone, fax, or e-mail:

Bill Ebert
Argonne National Laboratory
Phone: 630/252-6103
Fax: 630/252-4771
e-mail: ebert@cmt.anl.gov

References:

ASTM C 1285

Standard Test Methods for Determining Chemical Durability of Nuclear Waste Glasses: The Product Consistency Test (PCT) Standard C 1285-97, Annual Book of ASTM Standards, Vol.

12.01. American Society for Testing and Materials, West Conshohocken, PA. The procedure was originally published as C 1285-94. The first revision was published as C 1285-97 March 1998.

EBERT-1998

W.L. Ebert, S.F. Wolf, and D.M. Strachan, "Evaluation of a Standard Test Method and Material for Low-Activity Waste Product Acceptance," Proceedings of XVIII International Glass Conference, July 5-10, 1998, San Francisco, CA. Eds. M. K. Choudhary, N. T. Huff, and C. H. Drummond III. Available on CD-ROM from The American Ceramic Society, Westerville, OH.

WOLF-1998

A Data Base and a Standard Material for Use in Acceptance Testing of Low-Activity Waste Products, S. F. Wolf, W. L. Ebert, J. S. Luo, and D. M. Strachan, Argonne National Laboratory report ANL-98/9 (1998).

Table 1. Formulation of Low-Activity Reference Glass,
in oxide mass %.

Oxide	Formulated mass %	Oxide	Formulated mass %
Al ₂ O ₃	10	Li ₂ O	0.1
B ₂ O ₃	8	MgO	0.1
BaO	0.005	MnO	0.1
CaO	0.5	MoO ₃	0.1
CdO	0.2	Na ₂ O	20
Cl	0.8	NiO	0.1
Cr ₂ O ₃	0.2	P ₂ O ₅	0.5
F	1	PbO	0.1
Fe ₂ O ₃	1	SO ₃	0.2
HgO	0.002	SiO ₂	54
I	0.002	SnO ₂	0.1
K ₂ O	1.5	TiO ₂	0.1
La ₂ O ₃	0.009	ZrO ₂	1

Second Letter to Participants

Dear Colleague,

Thank you for participating in the round robin testing program that we are conducting under the auspices of the DOE Tanks Focus Area (TFA) for Immobilization. Enclosed you will find about 17 g of crushed glass referred to as "TFA Reference Glass LRM" that has been sieved to isolate the -100 +200 mesh fraction and washed to remove fines. Aliquots of glass taken from the same preparation batch have been provided to all participants. No further sample preparation is necessary. This amount will be sufficient to run two sets of triplicate tests at each temperature and to provide glass for composition analysis.

The glass is a borosilicate glass that contains about 20 mass % Na_2O and small amounts of other components that are either present in DOE waste streams or may be added to ILAW products. The glass also contains small amount of the RCRA metals Ba, Cd, Cr, and Pb. We have shown the glass itself to be non-hazardous per the TCLP. A paper describing the glass composition and the results of tests conducted with the prototype glass is enclosed for your information. Additional detail of the testing done with the prototype glass is available [WOLF-1998]. The glass used in the round robin was made to closely match the composition of the prototype.

The procedure for PCT Method A is to be followed for tests at both 40 and 90°C, except that tests with a standard reference glass are not required. A copy of the ASTM PCT procedure is provided. Key aspects of the testing are summarized below:

- Three replicate tests are to be conducted at both 40 and 90°C with between 1 to 1.5 g of crushed glass and a mass of demineralized water (ASTM Type I water) that is exactly 10 times the mass of glass used. For example, if 1.15 g of glass is used, then 11.50 ± 0.02 g of demineralized water must be used.
- All tests are to be conducted for 7-day test durations. The times of day a test vessel is placed in the oven and removed from the oven must agree within 3.4 hours.
- The use of unsensitized Type 304L stainless steel vessels is preferred, but tests may be conducted with TFE-Teflon vessels if steel vessels are not available. The vessel type used in the tests must be reported.
- Duplicate blank tests with demineralized water are to be conducted simultaneously with the tests with glass at each temperature. The vessels used in the blank tests must have been cleaned in the same batch as the vessels used in the tests with glass.
- The oven temperature must remain within 2°C of the desired test temperature throughout the test.
- The total mass of the test vessel must not change during the test by more than 5 % of the mass of demineralized water that was initially added to the vessel.
- Solutions generated in the tests with glass and the blanks tests are to be filtered through 0.45- μm pore size filters, acidified with concentrated nitric acid, and then analyzed for Al, B, Na, and Si. Analysis for other components is optional. Estimated detection limits must be reported.
- A model PCT data sheet is provided in Fig. X2 of the ASTM procedure. Other data sheets containing the test information can be used.
- While a particular analytical method is not required for solution analysis, the method(s) used must be reported. (Refer to the concentrations measured in tests with the prototype glass in the enclosed paper for expected range of concentrations for selection of method.)

No restrictions are placed on the method used to dissolve the glass for chemical analysis, except that the method used to dissolve and analyze the glass must be reported with the analytical results. The target composition of the glass is provided in Table 1 to facilitate selection of the method and analysis.

The concentrations measured at different laboratories will be analyzed to measure the reproducibility of the test method and measurement of the glass composition. These results will be documented in a report to the TFA and may be used by DOE in future privatization contracts. We also plan to present and publish the results at the American Ceramic Society (ACerS) meeting in Indianapolis, IN, on April 25-28, 1999. As has been done in previous round robins, participating laboratories will be recognized in all reports, but the results will not be linked to the laboratories. Please include desired wording to be included in acknowledgements of laboratories and contributors with reported results.

Test results must be reported to ANL before April 16, 1999 to allow compilation and review of the results for presentation at the ACerS meeting. Participatants will be provided with the compiled test results to verify correct transcription prior to communication or presentation of those results.

Thank you for your participation in this round-robin program. Please contact me with any questions or comments.

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Argonne National Laboratory
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References:

ASTM C 1285

Standard Test Methods for Determining Chemical Durability of Nuclear Waste Glasses: The Product Consistency Test (PCT) Standard C 1285-97, Annual Book of ASTM Standards, Vol. 12.01, American Society for Testing and Materials, West Conshohocken, PA. The procedure was originally published as C 1285-94. The first revision has published as C 1285-97 March 1998, but copies of this revision are not yet available.

EBERT-1998

W.L. Ebert, S.F. Wolf, and D.M. Strachan, "Evaluation of a Standard Test Method and Material for Low-Activity Waste Product Acceptance," Proceedings of XVIII International Glass Conference, July 5-10, 1998, San Francisco, CA. Eds. M. K. Choudhary, N. T. Huff, and C. H. Drummond III. Available on CD-ROM from The American Ceramic Society, Westerville, OH.

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in oxide mass %.

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CaO	0.5	MoO ₃	0.1
CdO	0.2	Na ₂ O	20
Cl	0.8	NiO	0.1
Cr ₂ O ₃	0.2	P ₂ O ₅	0.5
F	1	PbO	0.1
Fe ₂ O ₃	1	SO ₃	0.2
HgO	0.002	SiO ₂	54
I	0.002	SnO ₂	0.1
K ₂ O	1.5	TiO ₂	0.1
La ₂ O ₃	0.009	ZrO ₂	1

Third Letter to Participants

Dear Colleague,

I hope all is well with the round robin tests and analyses you are running. In response to questions from some participants, I have listed below the information expected to be in the data report:

For the PCTs:

Mass glass and mass demineralized water used in each test

Vessel type (material and approximate volume)

Mass loss incurred during test

Measured pH (at room temperature)

Concentrations of Al, B, Na, Si (and other components that were quantified) in solutions from tests with glass. Concentrations to be adjusted for any dilutions made but without background subtraction.

Concentrations of Al, B, Na, Si (and other components that were quantified) in solutions from blank tests

Analytical technique(s) used to analyze solutions (with estimated detection limits)

Any observations or comments on execution of test or analysis

For the glass analysis:

Method used to dissolve glass

Mass of glass dissolved and volume solution

Analytical technique(s) used to analyze solutions

Dilution-corrected concentrations of all components that were quantified (with estimated detection limits)

Any observations or comments on analysis

In addition to these data, any calculations you have done are most welcome (e.g., calculation of glass composition on an oxide basis, normalized concentrations, etc.). I will provide you opportunity to review with my work-up of the data before any presentation. Thank you for your help in this effort.

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