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EVALUATION OF EXPERIMENTAL FACTORS THAT INFLUENCE THE APPLICATION AND DISCRIMINATION CAPABILITY OF THE PRODUCT CONSISTENCY TEST (U)

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ABSTRACT

Liquid high-level nuclear waste will be immobilized at the Savannah River Site (SRS) by vitrification in borosilicate glass. The glass will be fabricated in the Defense Waste Processing Facility (DWPF) where the glass will be poured into stainless steel canisters for eventual disposal in a geologic repository. In order to comply with the Waste Acceptance Preliminary Specifications (WAPS) the durability of the glass needs to be measured during production in order to demonstrate its long term stability and radionuclide release properties.

A durability test, designated the Product Consistency Test (PCT), was developed for DWPF glass in order to meet the WAPS requirements. The response of the PCT procedure was based on extensive testing with glasses of widely differing compositions. The PCT was determined to be very reproducible, yield reliable results rapidly, and be easily performed in shielded cell facilities with radioactive samples.

In the study attached,[1] Battelle Pacific Northwest Laboratory independently verified the optimum PCT test parameters for glasses of very similar composition. During this study the following four parameters were investigated: the test duration, necessity for leachate filtration, necessity for sample washing, and the optimum ratio of the sample surface area (SA) to leachant volume (V).

The Battelle study indicated the following:

- All four factors influenced the solution pH and the elemental releases from the glasses
- The four factors did not influence the precision of the pH measurements but did affect the precision of the elemental releases
- The SA/V ratio and the leachate filtration were found to have the largest effect on the precision of the elemental releases
- Larger SA/V ratios (more glass to less solution) and leachate filtration improved discrimination of the test
- Washing of the fines may dampen the discrimination ability of the test but improves the precision of the replicates

[1]Battelle Pacific Northwest Laboratory Subcontract AX-828297

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INTRODUCTION

A durability test, designated the Product Consistency Test (PCT), has been developed for glasses fabricated in the Defense Waste Processing Facility (DWPF)[1] in order to meet the requirements of the Waste Acceptance Preliminary Specifications (WAPS) 1.3 and 1.4.[2] Specification 1.3 requires the DWPF to demonstrate control of the radionuclide release properties of the final waste form. Although changes in phase composition due to devitrification do not greatly alter the rate of release of material from the glass[3] of the type which will be produced in DWPF, the WAPS Specification 1.4 requires that the release properties of devitrified glass be similar to those determined in Specification 1.3. Since the PCT is a production control test, the DWPF is also responsible for relating the results of the PCT to repository site-specific release tests, or, alternatively, for performing the repository site-specific release tests.

The Product Consistency Test (PCT) has been developed, in part, to satisfy the WAPS requirements by providing a test which is (1) sensitive to glass composition and homogeneity, and (2) has the potential to be related to repository site-specific release tests. The test was designed to provide confirmation of the consistency of DWPF glass while considering the following:

- o sensitivity of the test to glass composition and homogeneity
- o time necessary to demonstrate product quality
- o ease of sample preparation for radioactive glass
- o ease of test procedure for remote operation
- o precision of the test results
- o acceptance by waste form developers and repository projects

During PCT development, sample size was limited to 100-200 mesh (149-74μm) crushed glass because leaching of finer mesh sizes can cause overestimation of saturation concentrations, e.g. if finer powders are used, mass balance calculations need to be used to determine the maximum saturation concentration expected from a given particle size.[4] Fine particles also contribute larger errors to the estimation of the sample surface area than coarser sized samples. Moreover, use of a coarser mesh crushed glass simplifies sample preparation for radioactive service.

One test temperature, 90°C, was chosen for the PCT. This temperature is representative of the anticipated temperature in a repository because of the heat of decay of the radionuclides in DWPF waste glass.

A single leachant, ASTM Type I water, was specified so that the test would be dominated by elemental species leached from the glass.

The V_{soln}/m_{solid} ratio for the PCT was chosen as 10 mL/g and test durations of 1, 3, 7, 14, and 28 days were evaluated. Seven days was chosen as the minimum test duration which optimized test precision but did not sacrifice discrimination.[1]

Leachate filtration to <0.45 μm was determined to improve the precision of the PCT. Filtering is advantageous because it removes colloidal species which would otherwise dissolve during the leachate acidification step and erroneously be measured as soluble elemental species. Filtering the leachate also removes the potential for fine glass particulates becoming entrained in the leachate acidification.[5] Such a dissolved particulate of glass would give an erroneously high soluble leachate concentration or contribute excessive radioactivity to the leachate.

PCT sample preparation specifies that the sieved glass should be washed in ASTM Type I water and absolute ethyl alcohol to remove electrostatically adhering fine particles. Comparisons of B.E.T. specific surface area measurements of alcohol washed and unwashed crushed basalt demonstrated that there was less than a 5% difference in the total surface area.[5] Other studies[6-9] have demonstrated that the <1μm fine particles only affect the initial non-linear kinetics of dissolution, e.g. the first 24 hour period. Thereafter, the fines are consumed with no further effect on the bulk dissolution. However, the amount of fines adhering to a glass sample is an uncontrollable quantity and, hence, sample washing was included in the PCT. Later experimental studies verified that sample washing improved the precision and the accuracy of the PCT.

An SRL internal round robin[1] and a seven laboratory external round robin were completed[10] in order to determine the precision and accuracy of the PCT. Confirmatory testing on radioactive samples was also performed.[11] These studies indicated that the PCT was very reproducible, yielded reliable results rapidly, and could be easily performed in shielded cell facilities with radioactive samples.

CONCLUSIONS

The Battelle Pacific Northwest Laboratory study confirmed that 7 day PCT test duration was more precise than 3 day test duration and that leachate filtration improved the test precision and discrimination. The study indicated that SA/V ratios of 5 or 10 ml/g, discriminate between similar glasses better than values of 100 ml/g. This confirmed the usage of the 10mL/g value specified in the PCT. The Battelle study also confirmed that sample washing to remove fines improved precision but may affect discrimination slightly.

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ATTACHMENT A

EVALUATION OF EXPERIMENTAL FACTORS THAT INFLUENCE THE APPLICATION AND DISCRIMINATION CAPABILITY OF THE PRODUCT CONSISTENCY TEST

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SUMMARY

An experiment was performed to investigate the effects (on pH and elemental releases of Al, Fe, K, Na, Si, B, Li, and Mn) of modifications to the test conditions of the Product Consistency Test (PCT). The experiment was replicated three times, where each replicate involved leach testing two glasses with each of 24 different sets of PCT conditions. The 24 sets of test conditions consist of all combinations of the levels of four factors:

Factor	Levels
Test duration	3 and 7 days
A/V	5, 10, and 100 ml/g
Cleaning	with and without
Filtering	0.45 μm and without

The two glasses used in the testing were provided by Savannah River Laboratory (SRL) and are denoted SRL-202-G and SRL-202-P. The two glasses are similar in composition, with SRL-202-P designed to be less durable than SRL-202-G.

The experimental data were statistically analyzed to assess the individual and interaction effects of the four factors on: (1) the pH and elemental release results, (2) the precision of the pH and elemental release results, and (3) the ability to discriminate between SRL-202-G and SRL-202-P based on pH and elemental releases. The following conclusions were reached in each of these three areas:

1. All four factors significantly affect (either individually or in interactions) pH and the elemental releases, with a few exceptions.

2. None of the four factors significantly affect (either individually or in interactions) the precision of pH results, while all four factors significantly affect elemental release results. A/V and filtering have the most important effects on the precision of elemental releases.

3. Only a few factors were found to have significant individual or interaction effects on the ability to discriminate, and the findings were not always consistent from one element to the next. This may be due to the small number of replicates and the effects of the factors in "separating" SRL-202-G and SRL-202-P cancelling the effects of the factors on precision. However, there was evidence (although not always statistically significant) that A/V and filtering affect the ability to discriminate. The ability to discriminate was better for tests with larger A/V values and filtered leachates. There was also an indication that "not cleaning" the crushed glass samples prior to leaching may improve the ability to discriminate for some elements.

The following sets of PCT conditions were determined to provide the "best" ability to discriminate among all the sets, given that they discriminated between SRL-202-G and SRL-202-P for eight or all nine of the responses (i.e., pH and the eight elemental releases):

Test Duration (days)	A/V (ml/g)	Cleaning (y or n)	Filtering (y or n)
3	5	n	y
3	5	y	y
7	5	n	y
7	5	y	y
7	10	n	y

It was also concluded that long-term (replicate-to-replicate) variation in PCT results is significantly larger than the short-term (within-replicate) variation for pH and several elemental releases. For the sets of test conditions providing the best ability to discriminate, precisions (including short-term and long-term variation) quantified in terms of percent relative standard deviations were in the 0.5-1.5% range for pH, in the 5-10% range for elemental releases having better precisions, and in the 12-30% range for elemental releases having poorer precisions.

1.0 INTRODUCTION

It is desirable to have a means of monitoring possible changes in waste glass durability during production so that the product remains within acceptable limits. A leach test called the Product Consistency Test (PCT) was developed by Savannah River Laboratory (SRL) as such a production test for the Defense Waste Processing Facility (DWPF). This report examines some of the experimental factors that may be used in the PCT which could influence test precision and its ability to function as intended.

1.1 BACKGROUND

The PCT is a powder leach test that was developed to provide a simple and rapid procedure for monitoring possible variations of composition and homogeneity of DWPF glass during production. To function as intended, the PCT must be able to distinguish between glasses that may have different durabilities based on repository acceptance testing of 28 days duration, yet be able to make this distinction within a few days while the glasses are being produced. Preferably the test duration should not be longer than 5 to 7 days, but less than three days would be even more desirable. There must be a high degree of precision associated with the test so that leaching effects associated with small differences in composition and homogeneity can be distinguished because these properties are the major contributors to glass durability. The test must also be relatively easy to apply in a hot cell environment.

The Waste Acceptance Preliminary Specification 1.3 (WAPS) requires that the DWPF demonstrate control of radionuclide release properties of the final waste form during production. In addition, it is the responsibility of the DWPF to show that the results of the production control test can be related to site-specific release tests or else these tests must be conducted separately. Site-specific tests are usually conducted for a month or more, are rock-water dominated rather than waste form dominated, and include tests at elevated pressure. Consequently they are not easily adapted to a production test. Furthermore, the current Nevada Nuclear Waste Storage Investigations (NNWSI) Project waste acceptance criteria requires an elemental mass loss not to exceed 1 g/m^2-d in a 28 day MCC-1 test. Since 28 days is too long for a reasonable production test, the PCT must be demonstrated as a good predictor of the acceptance criteria. For a given pH, the normalized release rate relationship between crushed glass and glass monoliths has been shown to be a continuous function of surface area/volume times time [(A/V) x t] (Pederson et al., 1983). In view of these considerations, Jantzen and Bibler (1987) adapted a crushed glass type of leach test as the basis for the PCT.

The PCT developed by Jantzen and Bibler (1987) is based on a modified version of the Materials Charaterization Center (MCC) MCC-3 test. Other tests that use crushed glass, including ASTM D33987-81 and a Corning Glass Works test, were also reviewed for possible use. The modifications to the MCC-3 test protocol included shorter time periods, one sieve size fraction, and uncleaned glass samples, but conceptually was very similar to the original MCC-3 protocol. The feasibility of the PCT was demonstrated by a round robin test conducted internally by SRL in 1987 using glasses representing a wide range of compositions and durability (Jantzen and Bibler, 1987). These results did not provide a lab-to-lab comparison or permit discrimination between glasses of similar composition. Subsequently a second PCT round robin was conducted by the MCC (Jones et al., 1989) which involved seven laboratories and four glasses, two of which were supplied by SRL. The second round robin provided information on interlab comparison and the ability to discriminate between glasses of similar composition.

1.2 PURPOSE AND SCOPE

The purpose of the present work (designated Phase 1) was to examine other factors that may affect the discrimination ability of the PCT protocol. These factors including sample preparation, leachate filtration, sample surface area to leachant volume ratio, and test duration. Phase 1 is intended as a screening effort. Sample cleaning and leachate filtration, for example, would require additional hot cell procedures that may or may not be warranted by the amount of increase in discrimination resolution.

The purpose of the second phase, designated Phase 2, is to focus on methods of improving test precision and to show how PCT results for a given glass predict the 28 day MCC-1 test results while maintaining satisfactory discrimination ability. Identifying and eliminating factors that might mask results or cause analytical interference should improve precision. It is also expected that an acceptable functional relationship that shows how PCT results for a given glass predict the 28 day MCC-1 test results will be determined. Phase 2 will also investigate the use of short-term methods such as pH-stat measurements as a discrimination tool, and explore the use of geochemical codes to evaluate reaction paths in both PCT and MCC-1 tests to help demonstrate convergence of results. The results of Phase 1 testing are reported here. Phase 2 testing will be initiated later in FY 1989.

2.0 EXPERIMENTAL FACTORS AND POSSIBLE EFFECTS

Phase 1 of the PCT experimental factor evaluation was designed as a screening experiment to identify and prioritize factors that might improve or detract from the intended function of the test. These factors, the levels (ie. values) at which they were investigated, and their possible effects on PCT sensitivity are discussed below.

2.1 EXPERIMENTAL FACTORS AND LEVELS INVESTIGATED

The selection of experimental factors for investigation was based partly on previous waste glass leach testing experience and partly on an interest in those factors that would simplify the test if they could be eliminated. Only two glass compositions were used. These glasses were supplied by SRL and are designated SRL-202-G and SRL-202-P. All testing was conducted at 90°C in deionized water using one size fraction (-100+200 mesh). The factors evaluated were test duration, sample surface-area-to-volume ratio (A/V, in terms of leachate volume per gram of crushed sample, ml/g), cleaning the glasss after crushing, and filtering the leachates through 0.45 um filters. The list of factors and their levels are shown in Table 2.1.

2.2 POSSIBLE EFFECTS OF THE EXPERIMENTAL FACTORS

Fine grained particles in the micron to submicron size range are formed during the sample preparation process and become electrostatically attached to sample surfaces. Because of their small size, these particles contribute excess surface free energy and rapidly dissolve during the initial stages of

TABLE 2.1 Experimental Factors and Their Levels.

Factor	Levels
Test duration	3 and 7 days
A/V	5, 10, and 100 ml/g
Filtering	0.45 μm and without
Cleaning	with and without

glass leaching. Usually they are completely dissolved within the first 24 hours. There are several ways in which the fines might affect test results. If unequal amounts and distributions of fines on different glasses exist, differences in leachate compositions will occur that are related to fines differences rather than to compositional differences among glasses. Rapid dissolution of fines might result in an abrupt pH change that enhances the leaching of the bulk glass so that it appears less durable. If a non-homogeneous glass is tested, there is a potential that the composition of fines may be different from that of the bulk glass. In this case, test results will be biased in favor of material contributed by the fines. In order to evaluate the effects of the presence of fines, half of the -100+200 mesh fraction of each glass was cleaned while the other half was used in the as-crushed condition without cleaning.

As indicated above, Phase 1 testing was conducted for two test durations, 3 and 7 days, with A/V ratios of 5, 10, and 100 ml/g. Decreasing the volume to mass ratio (equivalent to increasing the specimen surface area to leachant volume ratio) may enhance the ability of the test to discriminate at short time periods. Also the use of a variable A/V x time function will permit a correlation with the MCC-1 repository acceptance test. The A/V ratio for the MCC-1 test is 0.1 cm^{-1} so a 28 day test will have an A/V x time function of 2.8 cm^{-1}-d. The -100+200 mesh size fraction used in the PCT test has a nominal specific surface area of about 180 cm^2/g so an A/V ratio of 100 ml/g would yield an A/V x time function of 1.8 cm^{-1}-d while the next highest ratio will be 18.0 cm^{-1}-d. The usefulness of this relationship as a possible predictor of MCC-1 test results will be investigated in the Phase 2 study, but some initial observations of the effects of time and A/V ratios are made in this report.

There are several types of particulates that can occur in leachates so the effects of filtering may be important. Suspended particulates and colloids may occur in leachates, particularly those from tests at high A/V ratios, so that more scatter (that is, less precision) in leachate analytical results will be expected from unfiltered samples than from filtered samples. Also, solid glass samples may be inadvertantly included during the pipetting process of obtaining the leachate sample after a test. Lower A/V ratio tests may not require filtration if leachate concentrations are low, and if this type of test is still able to discriminate satisfactorily between glasses, the PCT protocol could be simplified.

Filtering may have an effect on the precision of some elements, but not others. The concentrations of eight elements, Al, Fe, Mn, K, Na, Li, Si, and B, plus pH were determined in the leachates. Presumably those elements that remain in true solution and do not readily form precipitates or colloid particles would be the best choice to use for discriminating between glasses. At high pH values that are typical of waste glass leachates, Al, Fe, and Mn can form precipitates with low solublity while B, Li, and Na are usually undersaturated. K analyses are considered less reliable than Na, but K should behave in a similar manner to Na. The solubility of Si is often controlled by amorphous silica saturation which is pH dependent. Under the test conditions used in this report Si may be present in colloidal forms or particulates at the higher A/V ratios. If precipitation might interfere with PCT discrimination abilty, low A/V ratios should help reduce this type of interference.

3.0 EXPERIMENTAL METHOD AND DESIGN

A brief description of the glass samples used, and the modification to the PCT method as required to evaluate the selected experimental factors are presented here. This section also includes a description of the experimental design.

3.1 EXPERIMENTAL METHOD

Two glass samples, about 500 g each, labeled SRL-202-G and SRL-202-P were received from SRL as frit with particles varying from a few mm to about 2 cm in size. These glasses represent a second batch prepared by Corning Glass Works. They are similar to, but not the same composition as, the two SRL-provided glasses that were previously used in the PCT round robin (Jones, 1989). The nominal compositions of the glasses used in this study are given in Table 3.1. These are compositions were provided to SRL by Corning. The MCC did not analyze these glasses.

A portion of each of these glasses was crushed and sieved so that a sufficient amount of material in the -100+200 mesh size range was obtained to complete the test matrix in triplicate. The glass was crushed using a tungsten carbide disk mill until the entire crushed material passed through a 100 mesh sieve. The material retained on the 200 mesh sieve was then used in the tests. Stainless steel sieves were used throughout. About half of the -100+200 mesh material for each glass was cleaned in the following manner. Approximately 50 g samples were placed in a beaker along with deionized water equal to about 3 volumes of the glass. The beaker and contents were ultrasonically cleaned for about 30 seconds then the liquid was decanted. This procedure was repeated 2 or 3 times until the liquid appeared clear. After decanting the last water, an amount of fresh absolute ethanol was added to the glass and the ethanol-glass mixture was ultrasonically cleaned for about a minute. The cleaned glass was air dried for 24 hours. Individual 50 g aliquots of cleaned and dried samples of the same glass were then combined and used as starting materials for the test matrix.

TABLE 3.1. Nominal Composition of SRL-202-G and SRL-202-P Glasses (wt. percent)

Oxide	SRL-202-G	SRL-202-P
Al_2O_3	5.15	4.78
B_2O_3	7.81	9.72
BaO	0.16	0.22
CaO	1.38	1.27
Cr_2O_3	0.11	0.10
CuO	0.43	0.74
Fe_2O_3	13.62	12.60
K_2O	3.78	6.76
Li_2O	4.31	3.91
MgO	1.34	1.22
MnO	2.23	2.06
Na_2O	9.41	10.22
NiO	0.82	0.77
SiO_2	48.65	44.19
TiO_2	0.70	1.28
	100.00	100.00

Note that the crushing, sieving and cleaning of the two glasses was completed prior to the initiation of leach testing. Because these operations were performed only once prior to leach testing and not separately for each of the three replicates, the PCT experimental results do not contain information on the variation due to these operations. This makes it easier to statistically assess the effect of varying the factors studied in the experiment, but does not provide for quantifying the contributions of these operations to PCT imprecision. It is expected that the crushing, sieving, and cleaning steps of the PCT procedure are major contributors to PCT variation and uncertainty.

Each of the two glasses were tested using the PCT protocol described by Jantzen and Bibler, 1987, except for modifications related to the factors under investigation. The modifications included two test durations, 3 and 7 days, the use of cleaned glass, and the use of some unfiltered leachate samples. The tests were conducted at 90°C in deionized water using Teflon PFA leach containers. After the test an aliquot of leachate was pipetted into a small polyethylene beaker for pH determination then the remaining leachate was extracted for analysis. If the sample was to be filtered, the filter used was a sterile 0.45 um syinge filter. Only cations were analyzed so all leachate specimens were stabilized by adding an amount of concentrated HNO_3 equal to 1% of the specimen volume. There was no attempt to randomize either leach containers or analytical samples. The tests were conducted in triplicate at three separate times over a two-and-a-half month period.

3.2 EXPERIMENTAL DESIGN

The approach that was used to develop a test matrix for this study involved identifying and prioritizing specific objectives, and then statistically designing a test matrix that would provide adequate data to address these particular objectives without restrictive or arbitrary assumptions. This approach yielded the test matrix presented in Table 3.2. This test matrix represents only a small fraction of the possible sets of test conditions that would have been studied, so a logical experimental strategy would be to: (1) run the test matrix presented in Table 3.2 (2) statistically analyze and interpret the results, and then (3) use the results for guidance in determining whether additional tests are needed, and if so, what additional test conditions should be included.

The test matrix shown in Table 3.2 investigates the effects of four factors having 2, 3, 2 and 2 levels (see Table 2.1), so that a total of 2x3x2x2 = 24 sets of test conditions are investigated. The test matrix was performed on the SRL-202-G and SRL-202-P glasses, and it will be convenient to think of "glass" as another factor having two levels (hence, there are 2x3x2x2x2 = 48 glass and test condition combinations). The 24 sets of conditions applied to the two glasses were replicated three times over a time period of two-and-a-half months.

The PCT experiment can be classified as a 2x3x2x2x2 factorial experiment carried out in randomized complete blocks (Montgomery, 1976). The three

TABLE 3.2. Test Matrix for the PCT Modification Evaluation with SRL-202-G and SRL-202-P Glasses.

Run ID	Duration (Days)	A/V (ml/g)	Filtering (y/n)	Cleaning (y/n)
1	3	5	y	y
2	3	5	y	n
3	3	5	n	y
4	3	5	n	n
5	3	10	y	y
6	3	10	y	n
7	3	10	n	y
8	3	10	n	n
9	3	100	y	y
10	3	100	y	n
11	3	100	n	y
12	3	100	n	n
13	7	5	y	y
14	7	5	y	n
15	7	5	n	y
16	7	5	n	n
17	7	10	y	y
18	7	10	y	n
19	7	10	n	y
20	7	10	n	n
21	7	100	y	y
22	7	100	y	n
23	7	100	n	y
24	7	100	n	n

replicates are referred to in statistical jargon as "blocks", and they are "complete" because the whole test matrix was performed on both glasses for each replicate. Ideally, "randomized" means that the tests within a block are set-up, run, and leachates analyzed in a random order. The tests within each block were not set-up in a random order but the consequences of this are expected to be small (the set-up steps are not likely to introduce confounding effects). Of more concern is that the leachates within each block required over a day to analyze, and calibrations and other within-day and day-to-day changes in the ICP lab can occur in this time frame. Without randomizing the order of leachate analyses, such changes can be confounded with the effects of the factors being investigated.

4.0 STATISTICAL ANALYSES OF THE DATA

The statistical investigations of the data from the PCT experiment focused on the following areas:

- summarizing the means, standard deviations (SDs) and percent relative standard deviations (%RSDs) of pH and each elemental release over the three replicates for each combination of glass and PCT conditions
- assessing how pH and the elemental releases were affected by varying the glass tested and the PCT conditions
- assessing how the precision of pH and the elemental releases were affected by varying the glass tested and the PCT conditions
- assessing how the ability to discriminate between SRL-202-G and SRL-202-P was affected by varying the PCT conditions.

The first bullet involves calculating some summary statistics, whereas the last three bullets involve making inferences about which of the factors investigated in the experiment have significant effects on the PCT elemental releases, precision, and ability to discriminate between two glasses.

4.1 MEANS, STANDARD DEVIATIONS, AND %RSDs

The raw data (pH values and elemental release values) from the PCT experiment are given in Tables A.1 through A.6 of Appendix A. Using these raw data, the means, SDs, and %RSDs for pH and the elemental releases of Al, Fe, K, Na, Si, B, Li, and Mn were computed and are listed in Table 4.1. These summary statistics are listed for both the SRL-202-G and SRL-202-P glasses for each of the 24 PCT conditions investigated.

In order to interpret the SDs and %RSDs listed in Table 4.1, it is important that the sources of variation contributing to the results be discussed. The SDs and %RSDs were computed from the triplicate results, which were obtained at three separate times over the space of two-and-a-half months. Because of the two-and-a-half month time span, it is clear that both short-term within-lab and long-term within-lab sources of variation are included in the results. However, not all of the potential sources of variation that affect PCT results were allowed to vary over the three replicate experiments.

TABLE 4.1. Means, Standard Deviations, and %RSDs from the PCT Experiment on Glasses SRL-G and SRL-P

Time (Days)	V/m (ml/gm)[1]	Cleaned (y or n)	Filtered (y or n)	Glass	Stat.	pH	Elemental Releases (mg/l) Al	Fe	K	Na	Si	B	Li	Mn
3	5	n	n	G	Mean	10.66	10.83	24.90	29.80	104.17	128.33	33.93	32.43	3.23
					SD	0.021	3.91	15.66	4.66	18.92	4.51	6.30	6.38	2.15
					%RSD	0.20	36.13	62.89	15.64	18.17	3.51	18.57	19.67	66.36
3	5	n	n	P	Mean	11.07	11.77	28.83	76.77	172.00	162.33	68.43	41.83	4.53
					SD	0.133	5.06	16.30	15.79	35.16	36.67	15.59	8.84	2.55
					%RSD	1.20	43.04	56.53	20.57	20.44	22.59	22.78	21.12	56.25
3	5	n	y	G	Mean	10.69	6.13	7.63	30.77	100.03	134.00	31.97	30.57	0.92
					SD	0.146	0.38	0.25	4.52	11.75	6.00	2.63	3.30	0.09
					%RSD	1.36	6.17	3.30	14.70	11.74	4.48	8.23	10.80	9.90
3	5	n	y	P	Mean	11.18	7.50	11.47	68.13	159.67	145.67	63.40	38.30	1.89
					SD	0.070	0.36	1.01	5.95	10.60	6.43	4.91	2.56	0.24
					%RSD	0.63	4.81	8.78	8.74	6.64	4.41	7.75	6.69	12.95
3	5	y	n	G	Mean	10.37	5.77	5.73	21.20	65.47	97.87	20.67	19.73	0.71
					SD	0.179	0.38	1.02	1.93	2.50	5.42	2.80	1.07	0.12
					%RSD	1.73	6.57	17.82	9.10	3.82	5.54	13.55	5.42	16.13
3	5	y	n	P	Mean	11.02	7.77	9.20	59.20	128.67	129.00	60.20	31.13	1.60
					SD	0.082	1.21	1.90	3.27	11.59	15.52	15.91	2.58	0.36
					%RSD	0.74	15.52	20.65	5.53	9.01	12.03	26.43	8.29	22.53
3	5	y	y	G	Mean	10.44	6.57	6.47	15.13	76.70	114.33	23.67	23.07	0.83
					SD	0.150	0.47	1.01	13.22	11.68	8.74	2.00	2.34	0.26
					%RSD	1.44	7.20	15.57	87.33	15.23	7.64	8.45	10.12	31.09
3	5	y	y	P	Mean	10.90	7.47	9.70	58.67	136.67	126.33	54.50	33.03	1.79
					SD	0.169	0.49	1.21	6.35	22.94	11.85	12.14	6.50	0.37
					%RSD	1.55	6.61	12.50	10.83	16.79	9.38	22.28	19.69	20.57

TABLE 4.1. Means, Standard Deviations, and %RSDs from the PCT Experiment on Glasses SRL-G and SRL-P (cont)

Time (Days)	V/m (ml/gm)	Cleaned (y or n)	Filtered (y or n)	Glass	Stat.	pH	Elemental Releases (mg/l) Al	Fe	K	Na	Si	B	Li	Mn
3	10	n	n	G	Mean	10.53	9.53	17.67	29.50	63.23	100.90	21.10	19.30	2.23
					SD	0.104	4.66	17.35	22.10	12.79	6.37	3.06	3.12	2.39
					%RSD	0.99	48.89	98.20	74.90	20.23	6.31	14.52	16.19	106.92
3	10	n	n	P	Mean	10.95	9.23	14.77	47.80	96.27	117.67	37.67	24.07	2.13
					SD	0.062	1.36	6.60	1.06	3.26	8.33	2.31	0.51	0.85
					%RSD	0.57	14.74	44.72	2.21	3.38	7.08	6.14	2.13	39.87
3	10	n	y	G	Mean	10.50	6.20	4.10	19.77	60.40	101.03	19.43	34.00	0.41
					SD	0.056	0.17	0.53	1.08	4.89	5.95	1.78	25.69	0.07
					%RSD	0.53	2.79	12.91	5.46	8.09	5.89	9.15	75.56	17.27
3	10	n	y	P	Mean	10.95	6.90	5.23	44.03	93.00	104.67	36.47	23.10	0.51
					SD	0.060	0.35	0.51	2.63	7.55	3.51	1.97	1.05	0.45
					%RSD	0.55	5.02	9.81	5.97	8.12	3.36	5.39	4.56	87.84
3	10	y	n	G	Mean	10.32	5.87	3.52	19.80	46.63	87.50	15.30	14.17	0.38
					SD	0.090	0.12	0.20	11.49	2.54	1.95	1.22	0.40	0.07
					%RSD	0.87	1.97	5.75	58.05	5.45	2.22	7.95	2.85	18.98
3	10	y	n	P	Mean	10.80	6.53	5.13	35.67	74.43	90.63	28.87	18.43	0.77
					SD	0.080	0.64	1.82	4.51	8.16	9.66	3.03	2.47	0.27
					%RSD	0.74	9.84	35.41	12.64	10.96	10.66	10.49	13.39	34.87
3	10	y	y	G	Mean	10.32	6.27	3.67	17.57	50.83	92.90	16.10	15.53	0.40
					SD	0.092	0.38	0.76	2.68	4.25	4.99	1.93	1.52	0.13
					%RSD	0.89	6.04	20.65	15.24	8.37	5.37	12.00	9.77	33.07
3	10	y	y	P	Mean	10.87	6.73	3.90	35.67	73.07	91.43	28.67	18.33	0.57
					SD	0.044	0.40	0.85	4.16	6.13	5.10	1.84	1.33	0.12
					%RSD	0.40	6.00	21.91	11.67	8.39	5.57	6.44	7.26	20.38

TABLE 4.1. Means, Standard Deviations, and %RSDs from the PCT Experiment on Glasses SRL-G and SRL-P (contd)

Time (Days)	V/m (ml/gm)	Cleaned (y or n)	Filtered (y or n)	Glass	Stat.	pH	Elemental Releases (mg/l) Al	Fe	K	Na	Si	B	Li	Mn
3	100	n	n	G	Mean	9.85	4.93	5.07	15.37	21.23	53.90	6.93	6.04	0.67
					SD	0.157	1.36	3.93	10.18	3.15	9.34	1.19	1.01	0.51
					%RSD	1.60	27.60	77.59	66.24	14.82	17.34	17.21	16.66	76.38
3	100	n	n	P	Mean	10.14	5.40	4.90	18.80	25.80	56.73	9.93	6.43	0.72
					SD	0.031	0.44	2.90	1.31	1.31	2.00	0.25	0.06	0.44
					%RSD	0.30	8.07	59.18	6.98	5.08	3.53	2.53	0.90	60.20
3	100	n	y	G	Mean	9.81	4.10	0.59	12.70	19.90	50.17	6.57	5.57	0.06
					SD	0.168	0.62	0.36	8.05	1.87	5.29	0.78	0.51	0.04
					%RSD	1.71	15.23	60.33	63.42	9.41	10.55	11.83	9.22	60.09
3	100	n	y	P	Mean	10.12	4.70	0.58	17.97	25.43	52.20	9.61	6.17	0.32
					SD	0.100	0.26	0.23	1.05	1.91	2.31	0.46	0.06	0.42
					%RSD	0.99	5.63	39.77	5.85	7.53	4.42	4.74	0.94	130.24
3	100	y	n	G	Mean	9.93	5.78	6.67	14.67	22.47	55.50	7.60	6.50	0.86
					SD	0.055	2.10	6.70	6.66	5.49	6.22	1.91	1.65	0.90
					%RSD	0.55	36.25	100.47	45.40	24.43	11.21	25.10	25.33	104.75
3	100	y	n	P	Mean	10.13	4.70	1.30	17.60	23.77	51.57	9.10	5.87	0.16
					SD	0.050	0.10	0.20	1.64	1.37	0.75	0.26	0.21	0.06
					%RSD	0.50	2.13	15.38	9.30	5.74	1.46	2.91	3.55	33.72
3	100	y	y	G	Mean	9.76	3.87	1.00	11.90	18.67	46.37	5.93	4.97	0.12
					SD	0.220	0.93	0.82	7.11	2.06	9.17	1.24	1.01	0.09
					%RSD	2.26	24.03	81.85	59.73	11.01	19.79	20.94	20.37	77.78
3	100	y	y	P	Mean	10.18	4.83	0.65	17.40	23.77	51.27	9.21	5.87	0.07
					SD	0.012	0.15	0.22	1.51	1.37	1.12	0.27	0.12	0.05
					%RSD	0.11	3.16	33.53	8.68	5.74	2.17	2.96	1.97	73.99

TABLE 4.1. Means, Standard Deviations, and %RSDs from the PCT Experiment on Glasses SRL-G and SRL-P (cont)

Time (Days)	V/m (ml/gm)	Cleaned (y or n)	Filtered (y or n)	Glass	Stat.	pH	Elemental Releases (mg/l) Al	Fe	K	Na	Si	B	Li	Mn
7	5	n	n	G	Mean	10.86	8.17	17.20	30.47	118.77	149.33	40.37	35.67	2.03
					SD	0.006	1.55	5.96	4.11	19.56	4.93	3.72	2.84	0.86
					%RSD	0.05	18.98	34.67	13.47	16.47	3.30	9.21	7.96	42.56
7	5	n	n	P	Mean	11.28	10.20	24.93	107.67	222.33	183.33	106.67	52.23	4.10
					SD	0.070	3.08	10.28	21.22	41.79	25.74	19.60	11.07	1.93
					%RSD	0.62	30.20	41.23	19.71	18.80	14.04	18.38	21.20	47.04
7	5	n	y	G	Mean	10.76	6.40	11.07	24.87	113.63	152.00	37.90	34.93	1.25
					SD	0.085	0.61	2.18	3.44	18.40	10.58	2.34	3.45	0.25
					%RSD	0.79	9.50	19.68	13.85	16.19	6.96	6.18	9.87	20.00
7	5	n	y	P	Mean	11.23	8.57	18.63	101.23	207.67	162.00	82.23	47.67	3.13
					SD	0.130	0.40	1.10	19.01	12.50	9.00	6.73	3.41	0.35
					%RSD	1.16	4.72	5.89	18.78	6.02	5.56	8.18	7.16	11.21
7	5	y	n	G	Mean	10.57	15.93	41.73	20.33	110.90	125.33	37.20	33.53	5.43
					SD	0.215	15.83	56.52	8.33	43.84	21.03	12.44	12.07	7.77
					%RSD	2.03	99.36	135.44	40.95	39.53	16.78	33.45	35.99	142.92
7	5	y	n	P	Mean	11.12	7.80	14.57	84.90	173.33	158.00	72.87	41.87	2.53
					SD	0.032	0.46	1.55	15.04	9.50	5.20	3.40	2.60	0.21
					%RSD	0.29	5.88	10.64	17.72	5.48	3.29	4.67	6.22	8.22
7	5	y	y	G	Mean	10.62	6.60	10.20	12.60	88.90	131.33	28.93	27.07	1.16
					SD	0.187	0.53	2.07	11.29	9.97	5.03	0.31	1.66	0.25
					%RSD	1.76	8.02	20.26	89.61	11.21	3.83	1.06	6.14	21.58
7	5	y	y	P	Mean	11.16	9.57	19.30	74.97	175.33	151.67	74.07	43.20	3.45
					SD	0.157	0.21	1.14	14.74	34.53	8.39	11.37	6.49	0.39
					%RSD	1.41	2.18	5.88	19.66	19.69	5.53	15.36	15.01	11.32

TABLE 4.1. Means, Standard Deviations, and %RSDs from the PCT Experiment on Glasses SRL-G and SRL-P (contd)

Time (Days)	V/m (ml/gm)	Cleaned (y or n)	Filtered (y or n)	Glass	Stat.	pH	Elemental Releases (mg/l) Al	Fe	K	Na	Si	B	Li	Mn
7	10	n	n	G	Mean	10.63	6.27	6.80	22.70	68.03	111.67	22.93	20.53	0.70
					SD	0.057	0.57	2.00	2.86	5.95	3.51	1.51	1.20	0.26
					%RSD	0.54	9.07	29.37	12.59	8.75	3.14	6.60	5.85	37.16
7	10	n	n	P	Mean	11.09	7.03	9.27	80.33	117.97	124.33	49.37	29.80	1.34
					SD	0.065	0.35	1.26	22.55	19.73	7.37	2.32	1.06	0.22
					%RSD	0.59	4.99	13.58	28.07	16.72	5.93	4.71	3.55	16.58
7	10	n	y	G	Mean	10.65	6.03	5.40	21.97	71.23	117.00	25.10	21.23	0.47
					SD	0.042	0.25	0.26	3.05	7.54	6.93	0.36	1.10	0.06
					%RSD	0.39	4.17	4.90	13.89	10.59	5.92	1.44	5.19	12.94
7	10	n	y	P	Mean	11.05	6.67	8.13	66.13	128.67	124.00	49.73	30.10	1.18
					SD	0.030	0.12	0.40	12.33	8.50	3.61	3.41	2.85	0.03
					%RSD	0.27	1.73	4.97	18.65	6.61	2.91	6.85	9.47	2.94
7	10	y	n	G	Mean	10.51	19.77	51.53	42.13	90.90	102.40	30.33	26.93	6.63
					SD	0.036	22.57	76.68	37.15	52.41	5.05	18.60	16.08	10.20
					%RSD	0.34	114.18	148.79	88.16	57.66	4.93	61.31	59.71	153.89
7	10	y	n	P	Mean	10.96	9.33	15.87	60.83	104.67	131.67	44.43	27.57	2.33
					SD	0.085	4.48	15.70	21.87	25.54	33.50	5.70	3.84	2.40
					%RSD	0.78	48.02	98.97	35.94	24.40	25.44	12.84	13.94	102.71
7	10	y	y	G	Mean	10.55	6.70	4.80	18.53	60.90	112.00	20.17	17.97	0.38
					SD	0.049	1.01	0.87	1.75	6.41	8.00	2.60	1.06	0.08
					%RSD	0.47	15.15	18.04	9.43	10.53	7.14	12.91	5.90	19.92
7	10	y	y	P	Mean	10.98	6.90	6.97	59.53	108.10	115.33	43.10	26.17	0.90
					SD	0.065	0.40	0.31	13.42	12.64	9.02	2.54	1.76	0.21
					%RSD	0.59	5.80	4.39	22.54	11.69	7.82	5.88	6.71	22.70

TABLE 4.1. Means, Standard Deviations, and %RSDs from the PCT Experiment on Glasses SRL-G and SRL-P (contd)

Time (Days)	V/m (ml/gm)	Cleaned (y or n)	Filtered (y or n)	Glass	Stat.	pH	Elemental Releases (mg/l) Al	Fe	K	Na	Si	B	Li	Mn
7	100	n	n	G	Mean	9.91	4.97	3.70	10.93	23.23	62.80	8.40	6.90	0.43
					SD	0.125	0.21	0.26	1.10	1.33	1.31	0.17	0.10	0.06
					%RSD	1.26	4.19	7.15	10.07	5.72	2.09	2.06	1.45	13.32
7	100	n	n	P	Mean	10.08	5.50	4.83	19.60	29.70	60.90	12.20	7.83	0.70
					SD	0.102	0.20	0.59	2.95	4.11	2.82	0.70	0.35	0.10
					%RSD	1.01	3.64	12.12	15.03	13.83	4.62	5.74	4.48	14.24
7	100	n	y	G	Mean	9.91	4.87	1.30	8.63	24.77	64.47	8.80	7.23	0.09
					SD	0.174	0.32	0.20	7.75	0.40	2.16	0.10	0.29	0.01
					%RSD	1.76	6.61	15.38	89.80	1.63	3.35	1.14	3.99	6.19
7	100	n	y	P	Mean	10.07	5.03	2.08	20.60	28.43	58.63	12.07	7.70	0.31
					SD	0.046	0.35	1.50	3.14	3.88	2.51	0.70	0.40	0.25
					%RSD	0.46	6.98	71.85	15.26	13.65	4.28	5.82	5.19	81.80
7	100	y	n	G	Mean	9.70	4.83	4.90	7.03	21.20	59.33	7.57	6.30	0.56
					SD	0.249	0.35	0.30	6.11	0.53	2.52	0.38	0.46	0.10
					%RSD	2.57	7.27	6.12	86.84	2.50	4.24	5.00	7.27	18.56
7	100	y	n	P	Mean	10.01	4.77	1.55	18.03	26.73	55.33	10.93	7.03	0.22
					SD	0.050	0.12	0.61	1.70	3.10	0.58	0.45	0.25	0.08
					%RSD	0.50	2.42	39.19	9.45	11.60	1.04	4.12	3.58	35.25
7	100	y	y	G	Mean	9.96	4.83	1.13	9.70	23.87	61.30	8.37	6.67	0.08
					SD	0.104	0.29	0.06	0.61	1.03	2.04	0.25	0.23	0.02
					%RSD	1.05	5.97	5.09	6.27	4.30	3.33	3.01	3.46	21.65
7	100	y	y	P	Mean	10.04	5.00	0.97	13.07	27.23	58.13	11.60	7.27	0.13
					SD	0.090	0.61	0.06	11.57	4.29	6.18	1.22	0.58	0.01
					%RSD	0.89	12.17	5.97	88.53	15.74	10.64	10.49	7.95	4.56

The crushing, sieving, and cleaning of the SRL-202-G and SRL-202-P glasses was all performed at the same time, prior to the three replicates of the PCT experiment. Because these activities were performed only once and not separately before each of the three replicates of the experiment, the data do not contain any information on the variation in PCT results due to the crushing, sieving, or cleaning steps of the procedure. The crushing and sieving of glass is thought to be a major source of variation in the PCT procedure, so that the SDs and %RSDs in Table 4.1 may be substantially less than what they would have been had this source of variation been included in the experimental design.

Table 4.2 lists the minimum, median, and maximum %RSDs for pH and each element over the 24 test conditions investigated. It should be kept in mind that the %RSDs are based on samples of size three, indicating that they are fairly uncertain. Still, it is clear that: (1) the precision of the PCT varies both with respect to test conditions and the element of interest, and (2) the short-term and long-term precision of the PCT can be very large (even with some of the major sources of variation "missing") depending on the test conditions.

4.2 EFFECTS OF THE EXPERIMENTAL FACTORS ON pH AND ELEMENTAL RELEASES

The analysis of variance (ANOVA) technique was used to investigate whether pH and elemental releases depend on the levels of the following factors:

Factor	Notation	Levels
Replicates	R	1, 2, 3
Glass	G	SRL-202-G, SRL-202-P
Test Duration	T	3, 7 days
A/V	S	5, 10, 100 ml/g
Cleaning	C	yes, no
Filtering	F	yes, no

ANOVA is a statistical technique for determining whether varying the levels of factors affects one cr more responses of interest. Factors may affect the responses individually and/or via interactions with other factors.

TABLE 4.2. Minimum, Median, and Maximum %RSDs Over the 24 Test Conditions Investigated in the PCT Experiment

%RSD	pH	Al	Fe	K	Na	Si	B	Li	Mn
Min	0.05	1.73	3.30	2.21	1.62	1.04	1.06	0.90	2.94
Median	0.76	6.79	19.97	15.25	10.77	5.53	8.06	7.27	26.89
Max	2.57	114.18	148.79	89.80	57.66	25.44	61.31	75.56	153.89

All main effects (i.e. effects of individual factors) and two-factor interactions were investigated, as well as the three- and four-factor interactions of the G, T, S, C, and F factors. When a k-factor interaction exists, the effect that one of the factors has on pH or a given elemental release depends on the levels of the other k-1 factors involved in the interaction. The main effect of a factor is denoted by its letter notation (e.g., T denotes the effect of changing the duration of the test), while interactions are denoted by joining their letter notations with "*" (e.g., C*F denotes the two-factor interaction between the cleaning and filtering factors).

An ANOVA was performed for pH and each of the eight elemental releases using the GLM procedure in SAS (1985). The results are summarized in Table 4.3. Factors that show up in one or more significant effects (main or interaction) for a given element or pH are said to have a statistically significant effect on the elemental release or pH (henceforth, pH and the eight elemental releases are referred to as "responses").

TABLE 4.3. Statistically Significant[a] Factor Effects Found in the ANOVAs Performed on the pH and Elemental Release Data from the PCT Experiment

Effect	pH	Al	Fe	K	Na	Si	B	Li	Mn
R	x			x	x	x	x	o	
G	x			x	x	x	x	x	
T	x		o	x	x	x	x	x	o
S	x	x	x	x	x	x	x	x	x
C	x			x	x	x	x	x	
F		x		x			x		x
R*G									
R*T					x	x			
R*S	o	o	o	x	x		x	x	o
R*C									
R*F									
G*T				x	x		x	x	
G*S	x			x	x	x	x	x	
G*C	o			x	x		x		
G*F						x			
T*S	x			x	x	x	x	x	
T*C		x	x					x	x
T*F									
S*C	x			x	x	x	x	x	
S*F							o		
C*F									
G*T*S				x	o		x		
G*T*C							o		
G*T*F									
G*S*C									
G*S*F						x			
G*C*F								o	
T*S*C									
T*S*F									
T*C*F	x	x	x		o				x
S*C*F									
G*T*S*C									
G*T*S*F									
G*T*C*F							x		
G*S*C*F									
T*S*C*F									

(a) x = effects significant at the 95% level ($\alpha = 0.05$)
o = effects significant at the 90% level ($\alpha = 0.10$).

Several observations can be made from Table 4.3:

- R has a significant main effect for pH and several elemental releases. This means that the long-term variation in PCT results (regardless of the test conditions) is significantly larger than the short-term variation within a replicate.

- R*S being significant for most of the responses indicates that the differences in results due to the three volume-to-mass (equivalently, surface area-to-volume) ratios varies from replicate to replicate.

- With one exception (which could be a spurious result due to chance), no four-factor interactions were significant.

- The T*C*F three-factor interaction was significant for several of the responses. This indicates that the difference in response between the two levels of any one of the three factors depends on the levels of the other two factors. For example, Figure 3.1 illustrates how filtering effects Na concentration depending on the test duration and whether the crushed glass was cleaned.

- All six of the factors R, G, T, S, C, and F have statistically significant (at the 95% level) effects (either main or interaction) on pH, K, Si, and B. This conclusion is based on the results from all data, and does not preclude the absence of effects of some factors for certain test conditions, glasses, or replicates.

- All of the factors except F (filtering) have a statistically significant effect (either main or interaction) on Na and Li concentrations. This conclusion is based on the results from all 24 test conditions, and does not preclude a filtering effect on Na and Li for a few test conditions.

- G is involved in several significant interactions with the test condition variables (T, S, C, and F) for K, Na, Si, B and Li. This indicates that differences between the elemental releases of these elements from SRL-G and SRL-P depend on the levels of the test condition variables. This indicates that the ability to discriminate between the glasses may depend on the test conditions, although the precision of each set of test conditions also influences this.

- There is no statistically significant effect of G (i.e., no differences in results between SRL-G and SRL-P) for Al, Fe, and Mn. This conclusion is based on the results from all 24 test conditions and does not preclude a "glass effect" on Al, Fe, and Mn concentrations for a few test conditions.

As mentioned in some of the observations above, the conclusions are a sort of "consensus" based on all of the data. Focusing on subsets of the data could lead to different conclusions.

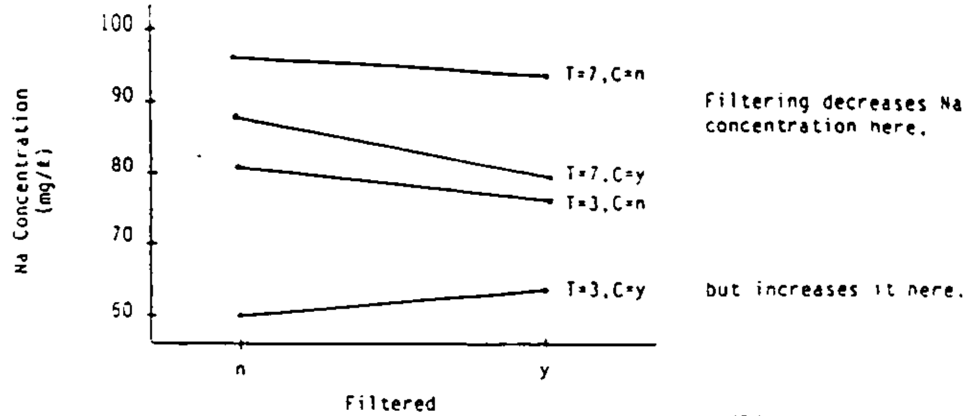

FIGURE 4.1. Illustration of the T*C*F Interaction Effect on Na Concentration. The effect of filtering depends on the test duration and whether or not the crushed glass was cleaned.

4.3 EFFECTS OF THE EXPERIMENTAL FACTORS ON PRECISION

The ANOVA technique was also used to investigate whether the precision of the pH and elemental release results depends on the levels of the factors G, T, S, C, and F. Specifically, ANOVA was performed on the natural logarithms of the variances[(a)] computed from the three replicate values for each test condition and glass combination. Because the replicate data were required to compute the variances (short-term plus long-term for the sources of variation previously discussed), it was not possible to investigate the effect of replicates (R) or of the four-factor interactions in this ANOVA. The GLM procedure of SAS (1985) was used to perform the ANOVA.

The results of the ANOVA to investigate the factors affecting the precision of the PCT are given in Table 4.4. The following observations are made:

- None of the factors have a statistically significant effect on the precision of the pH results.
- All of the factors (G, T, S, C, and F) affect the precision of the Al, Fe, Si, Li, and Mn results.

(a) It is common statistical practice to analyze the logarithms of the variances because they tend to more nearly satisfy the normality and constant uncertainty assumptions required for ANOVA.

TABLE 4.4. Statistically Significant[a] Factor Effects Found in the ANOVAs Performed on Log(Variances) of the pH and Elemental Release Data from the PCT Experiment

Effect	pH	Al	Fe	K	Na	Si	B	Li	Mn
G		o					x		
T				o					
S		x	x	x	x	x	x	x	x
C									
F		x	x				x		x
G*T				x			x	x	
G*S						x	o	o	
G*C									o
G*F							o		o
T*S			x						x
T*C		x	x						x
T*F									
S*C									
S*F		x							
C*F									
G*T*S		o	x		x	x	x	x	
G*T*C								x	
G*T*F									
G*S*C						o			
G*S*F						o			
G*C*F									
T*S*C			x						o
T*S*F									
T*C*F		x	x						x
S*C*F								o	

(a) x = effects significant at the 95% level ($\alpha = 0.05$)
o = effects significant at the 90% level ($\alpha = 0.10$).

- All of the factors except C (cleaning) and F (filtering) affect the precision for K and Na. All of the factors except for C affect the precision of the B results.
- Factor S has a significant main effect on the precision for all eight elements. The precision increases (i.e., the variance decreases) as the volume/mass ratio increases.
- Factor F has a significant main effect for four elements (Al, Fe, B, and Mn). The precision is higher (i.e., the variance is smaller) when the leachate is filtered. Although F is not indicated as having a significant main effect for K, Na, Si, and Li, the trend is also there for improved precision with filtering.

The above observations are based on ANOVA of the variances for both glasses and for all 24 sets of test conditions. The observations might differ if attention were focused on subsets of the data.

4.4 EFFECTS OF THE EXPERIMENTAL FACTORS ON THE ABILITY TO DISCRIMINATE

The ANOVA technique was also used to investigate whether the ability to discriminate between two glasses based on pH and elemental releases depends on the levels of the factors T, S, C, and F. Specifically, ANOVA was performed on t-ratios comparing SRL-202-P and SRL-202-G using the replicate results. T-ratios for pH and the eight elements were computed according to the formula:

$$t = \frac{(d_1 + d_2 + d_3)/3}{sd(d_1, d_2, d_3)/sqrt(3)} \tag{4.1}$$

where d_i = the difference in pH or elemental release values between the SRL-202-G and SRL-202-P glasses for the data from replicate i. The value for SRL-202-G is subtracted from the value for SRL-202-P to form d_i.

The numerator of (4.1) is merely the average of the three d_i values, while the denominator is the standard deviation of the three d_i values divided by the square root of three. The denominator of (4.1) is the standard deviation of the numerator, and hence (4.1) fits the definitional form for a "t-ratio".

The t-ratios for pH and the eight elemental releases for each of the 24 sets of test conditions are given in Table 4.5. Note that for a sample of size three, a t-ratio must be larger than 2.92 to discriminate between SRL-202-P and SRL-202-G with 95% confidence[a]. For pH, Na, B, and (to a lesser extent) K, the t-ratios are larger than 2.92 for most of the 24 sets

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(a) The 2.92 critical value corresponds to the 95th percentile of the t-distribution with two degrees of freedom. Since SRL-P is a less durable glass than SRL-G, it is assumed that only positive t-ratios indicate discrimination between the two glasses. Thus, the t-ratio critical value of 2.92 corresponds to a one-sided t-test.

TABLE 4.5. T-Ratios[a] for Discriminating Between SRL-G and SRL-P for Each of 24 Sets of PCT Conditions Investigated in the PCT Experiment

I	S	C	F	pH	Al	Fe	K	Na	Si	B	Li	Mn	# t's > 2.92	Comments
3	5	n	n	4.69	0.24	0.30	4.21	2.40	1.47	2.96	1.20	0.64	3	
3	5	n	y	11.28	41.00	6.00	17.36	68.06	4.85	23.78	17.24	5.67	9	Smallest is 4.85.
3	5	y	n	5.49	2.25	2.14	13.16	12.02	2.95	4.12	13.05	3.25	7	
3	5	y	y	3.27	2.56	5.11	6.15	9.19	4.54	5.15	4.04	6.99	8	Good.
3	10	n	n	12.89	-0.14	-0.42	1.51	3.81	2.26	5.36	2.28	-0.10	3	
3	10	n	y	15.59	2.65	4.15	11.93	5.70	0.74	11.13	-0.71	0.45	5	
3	10	y	n	5.64	1.52	1.66	3.22	8.35	0.56	8.10	2.89	3.38	5	
3	10	y	y	13.61	1.24	0.67	12.36	17.11	-0.90	28.75	10.58	2.77	5	
3	100	n	n	3.92	0.76	-0.19	0.60	3.93	0.63	4.61	0.69	0.57	3	
3	100	n	y	2.38	1.96	-0.12	1.13	166.00	0.69	7.26	1.96	1.11	1	
3	100	y	n	20.00	-0.86	-1.42	0.73	0.33	-0.98	1.20	-0.61	-1.39	1	
3	100	y	y	3.21	2.01	-0.60	1.62	10.34	0.89	4.16	1.38	-0.64	3	
7	5	n	n	10.39	2.05	2.93	6.15	5.74	2.08	5.10	3.30	3.32	7	
7	5	n	y	6.78	6.60	5.46	5.90	13.35	4.80	16.67	8.31	6.10	9	Smallest is 4.80.
7	5	y	n	4.86	-0.89	-0.83	4.88	3.12	2.20	6.65	1.47	-0.65	4	
7	5	y	y	12.77	7.90	5.01	4.34	5.69	8.71	6.97	5.57	8.64	9	Smallest is 4.34.
7	10	n	n	39.84	1.46	1.33	4.44	5.15	2.17	18.21	11.45	2.59	5	
7	10	n	y	23.80	3.80	8.60	8.08	101.35	3.36	11.37	8.75	14.39	9	Smallest is 3.36, seven are > 8.08.
7	10	y	n	14.26	-1.00	-1.01	2.11	0.80	1.42	1.88	0.09	-0.95	1	
7	10	y	y	9.38	0.42	5.64	5.37	10.67	0.64	36.21	20.29	4.67	7	
7	100	n	n	4.23	16.00	2.88	4.91	3.95	-2.11	11.82	3.59	4.15	7	
7	100	n	y	1.58	1.15	0.91	1.98	1.81	-13.23	9.39	3.88	1.47	2	
7	100	y	n	2.67	-0.46	-6.47	2.44	2.64	-3.46	9.07	2.08	-3.51	1	
7	100	y	y	6.93	0.90	-2.50	0.53	1.68	-1.29	5.71	3.00	5.29	4	

(a) Positive t-ratios indicate that the mean pH or elemental release for SRL-P was larger than the corresponding value for SRL-G. T-ratios larger than 2.92 indicate a statistically significant difference.

of test conditions. This indicates that these variables are a good basis for discriminating between SRL-202-G and SRL-202-P regardless of the PCT conditions used. The elements Al, Fe, Si, Li, and Mn have nonsignificant (smaller than 2.292) t-ratios for several of the 24 test conditions, and some of the t-ratios are even negative. However, there are some of the 24 test conditions for which even these elements have significant t-ratios.

The results of the ANOVA to investigate the effects of the factors T, S, C, and F on the t-ratios are given in Table 4.6. Only main effects and two-factor interactions were investigated. A few scattered effects were found to be significant, but there doesn't appear to be much commonality from one response (pH or one of the eight elements) to the next. This may be because the t-ratios are only based on a sample of size three, leading to considerable variation and uncertainty in the values. This variation may have made it difficult for the ANOVA to declare significance. It is important to remember that failure to declare an effect significant doesn't mean it isn't, there just may not have been enough evidence to declare it significant.

The results in Table 4.6 do indicate that there are significant main effects of S and F for several responses. The means of the t-ratios for each level of each factor are given in Table 4.7 as an aid to understanding the main effects of the factors. For example, the ability to discriminate appears to be better (for most responses) when S = 5 or 10 rather than 100. Similarly, filtering the leachate appears to increase the ability to discriminate. There doesn't appear to be much effect of cleaning on the ability to discriminate except for pH, Al, Fe, and Na where "not cleaning" increases the ability to discriminate. Finally, the test duration does not seem to have a consistent effect across the responses.

4.5 THE "BEST" SETS OF TEST CONDITIONS FOR THE PCT

Based on the preceding statistical analyses, it is of interest to select the "best" sets of test conditions for the PCT among the 24 studied in the experiment. Criteria for comparing the 24 sets of test conditions include:

TABLE 4.6. Statistically Significant[(a)] Factor Effects Found in the ANOVAs Performed on t-Ratios of the pH and Elemental Release Data from the PCT Experiment

Effect	pH	Al	Fe	K	Na	Si	B	Li	Mn
T									
S	x		x	x		x			
C			x						
F			x		o		o		x
T*S						o			
T*C			o						
T*F									
S*C	o								
S*F									
C*F									

(a) x = effects significant at the 95% level ($\alpha = 0.05$)
o = effects significant at the 90% level ($\alpha = 0.10$).

TABLE 4.7. Means of t-ratios for Each Level of Each Factor

Factor	Level	pH	Al	Fe	K	Na	Si	B	Li	Mn
T	3	8.5	4.6	1.4	6.2	25.6	1.5	8.9	4.5	1.9
	7	11.4	3.2	1.8	4.3	13.0	0.4	11.6	6.0	3.8
S	5	7.4	7.7	3.3	7.7	14.9	4.0	8.9	6.8	4.2
	10	16.9	1.2	2.6	6.1	19.1	1.3	15.1	7.0	3.4
	100	5.6	2.7	-0.9	1.7	23.8	-2.4	6.6	2.0	0.9
C	n	11.4	6.5	2.6	5.7	31.8	0.6	10.6	5.2	3.4
	y	8.5	1.3	0.6	4.7	6.8	1.3	9.8	5.3	2.3
F	n	10.7	1.7	0.1	4.0	4.3	0.8	6.6	3.5	0.9
	y	9.2	6.0	3.2	6.4	34.2	1.2	13.9	7.0	4.7

- good separation of pH and elemental release results for different but similar glasses
- good precision for pH and elemental releases
- good ability to discriminate between different but similar glasses

The third criterion is actually a combination of the first two.

The results of the ANOVA on the t-ratios (Section 4.4) indicated that volume-to-mass (surface area-to-volume) ratio, filtering, and to some extent cleaning affect the ability of the PCT to discriminate between the SRL-202-G and SRL-202-P glasses. These conclusions are in agreement with the tabulations and comments in the last columns of Table 4.5. The tabulation column in Table 4.5 shows for each of the 24 sets of test conditions the number out of nine responses (pH and the eight elemental releases) having significant (greater than 2.92) t-ratios. The rationale for this tabulation is that, presumably, test conditions that provide discrimination ability for most or all of the responses are the "best" based on the discrimination criterion. Four sets of test conditions had all nine t-ratios significant and one had eight significant:

"Best Discrimination" Sets of PCT Conditions

T	S	C	F	# t-ratios Significant
3	5	n	y	9
3	5	y	y	8
7	5	n	y	9
7	5	y	y	9
7	10	n	y	9

These results are in agreement with those from the ANOVA of t-ratios in that test conditions with smaller volume-to-mass ratios (5 and 10), filtering, and (possibly) no cleaning provide better ability to discriminate between SRL-202-G and SRL-202-P.

It can be verified from Table 4.1 that the five "best discrimination" sets of conditions shown above have some of the better precisions among the 24 sets of conditions investigated. This should be expected, since t-ratios incorporate precision information. However, it can also be seen from Table 4.1 that other sets of conditions have comparable or even better precisions (e.g., many of the sets of conditions with volume/mass ratios = 100).

Because the separation between PCT results from SRL-202-G and SRL-202-P is not as good for these other sets of conditions, they are not "best" from a discrimination standpoint.

Table 4.8 summarizes the %RSDs for the five "best discrimination" sets of conditions shown above. This gives an idea of the short- plus long-term precision that can be expected from these test conditions given the sources of variation included. Including the sources of variation due to glass crushing, sieving, and cleaning (which were not included in the PCT experiment) would decrease the precision (increase the %RSDs).

TABLE 4.8. %RSDs Pooled Over SRL-202-G and SRL-202-P for the Five "Best Discrimination" Sets of PCT Conditions

T	S	C	F	pH	Al	Fe	K	Na	Si	B	Li	Mn
3	5	n	y	1.06	5.53	6.63	12.09	9.54	4.44	7.99	8.98	11.53
3	5	y	y	1.50	6.91	14.12	10.83*	16.03	8.55	16.85	15.65	26.36
7	5	n	y	0.99	7.50	14.52	16.50	12.21	6.30	7.25	8.62	16.21
7	5	y	y	1.59	5.88	14.92	19.66*	16.02	4.76	10.89	11.70	17.23
7	10	n	y	0.34	3.19	4.94	16.44	8.83	4.66	4.95	7.64	9.38

* The value for SRL-202-P is listed, because the value for SRL-202-G was considerably larger.

5.0 CONCLUSIONS

The main goal of this work was to assess how the ability of the PCT to discriminate between two glasses depends on the levels of several test factors. The ability to discriminate for a given set of test conditions depends on the precision of the test and the "separation" (i.e., difference) in results (pH or elemental releases) for the two glasses for the given set of conditions. The sets of test conditions that provide the best ability to discriminate are those that have good precision for the amount of separation produced. Neither good precision nor good separation alone are sufficient to provide good discrimination. Both are important and tradeoffs between the two may be required to achieve good discrimination.

The following conclusions are made relative to separation and precision of the results and the ability of the PCT to discriminate.

- The long-term (replicate-to-replicate) variation in PCT results is significantly larger than the short-term (within-replicate) variation for pH and several elemental releases.
- The effect of A/V (on pH and most of the elemental releases) varied from replicate to replicate. This indicates that any effort to model PCT responses as a function of A/V should be based on replicate tests conducted at different times.
- In most cases, the pH and elemental releases depend on the glass tested, the test duration, A/V, cleaning, and filtering (i.e., these factors had significant individual or interaction effects for most responses). The exceptions to this are that filtering does not affect Na and Li, and there are no significant differences between the two glasses for Al, Fe, and Mn.
- The test condition factors (test duration, A/V, cleaning, and filtering) displayed significant interactions with "glass" for several responses, indicating that the test factors affect the separation in results between the two glasses.
- The precision of the pH results was not dependent on the glass tested nor the test condition factors.
- The precision of all elemental releases was affected by the glass tested and the test condition factors with the following exceptions: (1) cleaning did not affect the precision of B, and (2) cleaning and filtering did not affect the precision of K and Na.

- A/V and filtering had the most significant effects on PCT precision. The precision increases for all eight elemental releases when leachates are not filtered and as A/V decreases.

Note that the above conclusions are based on the results from all 24 sets of test conditions performed on both the SRL-202-G and SRL-203-P glasses and hold for the majority of the data. The conclusions regarding significance or nonsignificance of factor effects may be different for specific subsets of the data. For example, there are a few sets of test conditions for which there is a significant difference between SRL-202-G and SRL-202-P for Al, Fe, and Mn (which is contrary to the third bullet above).

The following conclusions are made regarding the effects of the test condition factors on ability of the PCT to discriminate between SRL-202-G and SRL-202-P.

- The effects of the test condition factors (test duration, A/V, cleaning, and filtering) on the ability to discriminate between SRL-202-G and SRL-202-P was investigated using t-ratios computed over the three replicates of the experiment. Only scattered individual and interaction effects of the factors were found to be statistically significant, but this may be due to the small number of replicates.

- It did appear that A/V and filtering have some effect on the ability to discriminate. The ability to discriminate appears to be better for larger A/V values (smaller volume-to-mass ratios) and for tests in which the leachate is filtered. "Not cleaning" test samples may have some effect on ability to discriminate for pH, Al, Fe, and Na.

- The following five sets of test conditions were identified as providing the best ability to discriminate because they discriminate between SRL-202-G and SRL-202-P for eight or all nine of the responses (pH and the eight elemental releases):

Test Duration (days)	A/V in units of V/m (ml/gm)	Cleaned (y or n)	0.45 μm Filtered (y or n)
3	5	n	y
3	5	y	y
7	5	n	y
7	5	y	y
7	10	n	y

These sets of conditions have some of the better precisions among the sets of conditions considered, but some test conditions with volume/mass ratios = 100 ml/gm had as good or better precisions. Although the precision for such sets of test conditions was good, the separation of results between SRL-202-G and SRL-202-P was not good, and hence the ability to discriminate was not as good.

Note that these conclusions are specific to discrimination between SRL-202-G and SRL-202-P, and may or may not hold for other similar glasses. The general effect of high A/V conditions and longer leach times is to increase the total amount of solids dissolved. The dissolution rate usually increases with A/V also. For the same test conditions, a less durable glass would be expected to dissolve faster and yield a higher total solute concentration than a more durable glass. Usually higher total concentrations exhibit greater analytical precision relative to dilute solutions. At high pH conditions, however, elements such as Al, Fe, and Mn tend to form relatively insoluble precipitates or colloids which could control leachate concentrations. Such an effect may have occurred with the two glasses used in this study and could be examined further using geochemical codes. Another problem associated with high A/V systems is that some of the original glass particles can be included with the leachate during the leachate extraction process at the end of a run where the leachate is pipetted into a sample vial. If not filtered, these particles could confuse the results. The use of lower A/V conditions and a focus on alkali elements and B, which tend to stay in solution, may avoid some of these types of problems. The effects of very low A/V conditions can be investigated using pH-stat methods. Whether or not the results of this study apply to other glasses that may exhibit some of these problems to different degrees is still uncertain and is part of the objective of Phase II of this work.

6.0 REFERENCES

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7.0 APPENDIX A

ELEMENTAL RELEASE DATA FOR THE THREE REPLICATES

TABLE A.1. SRL-202-G, Replicate 1, DIW, 90C

Sample Number	Time Days	SA/V ml/gm	Cleaned Y/N	Filtered Y/N	pH	Al	Fe	K	Na	Si	B	Li	Mn
001-gc	3	5	y	y	10.28	6.2	5.4	24.4	68.1	107.0	21.7	21.0	0.59
002-g	3	5	n	y	10.79	6.4	7.4	31.3	90.1	134.0	29.6	27.3	0.82
003-gc	3	5	y	n	10.22	5.5	5.3	22.6	65.4	92.2	19.1	19.5	0.71
004-g	3	5	n	n	10.64	9.2	17.4	28.4	92.5	128.0	30.6	28.8	2.20
005-gc	3	10	y	y	10.40	6.0	2.8	14.7	46.5	87.2	14.0	13.9	0.25
006-g	3	10	n	y	10.51	6.0	3.5	19.3	58.2	95.1	17.5	63.6	0.34
007-gc	3	10	y	n	10.32	6.0	3.4	14.4	43.7	86.8	14.7	14.1	0.30
008-g	3	10	n	n	10.56	7.2	7.7	17.5	56.1	95.7	18.9	17.7	0.80
009-gc	3	100	y	y	9.54	2.8	0.1	6.7	16.3	35.8	4.5	3.8	0.02
010-g	3	100	n	y	9.66	3.4	0.2	8.1	17.8	44.2	5.7	5.0	0.02
011-gc	3	100	y	n	9.90	8.2	14.4	13.0	28.8	62.6	9.8	8.4	1.90
012-g	3	100	n	n	9.71	3.4	0.6	8.1	17.6	43.4	5.6	4.9	0.10
013-gc	7	5	y	y	10.76	6.4	8.5	21.8	77.4	132.0	29.0	25.3	0.90
014-g	7	5	n	y	10.77	6.1	9.3	27.6	92.9	144.0	35.2	31.2	1.00
015-gc	7	5	y	n	10.67	6.2	9.0	23.0	75.7	124.0	27.4	25.4	1.00
016-g	7	5	n	n	10.87	6.6	11.8	29.4	96.3	146.0	36.1	32.6	1.30
017-gc	7	10	y	y	10.61	5.8	3.8	16.6	53.7	104.0	17.5	17.0	0.30
018-g	7	10	n	y	10.64	5.8	5.5	18.9	62.7	109.0	25.4	20.1	0.50
019-gc	7	10	y	n	10.55	5.7	4.2	19.4	54.7	98.2	17.8	16.0	0.30
020-g	7	10	n	n	10.61	6.1	6.3	21.1	62.1	108.0	21.2	19.3	0.60
021-gc	7	100	y	y	10.01	5.0	1.2	10.1	23.6	62.9	8.4	6.8	0.07
022-g	7	100	n	y	9.97	5.1	1.5	10.9	24.3	65.4	8.8	7.4	0.10
023-gc	7	100	y	n	9.89	5.2	4.9	10.1	21.8	62.0	8.0	6.8	0.50
024-g	7	100	n	n	9.90	4.8	3.6	9.8	21.7	61.4	8.2	7.0	0.40

TABLE A.2. SRL-202-P, Replicate 1, DIW, 90C

Sample Number	Time Days	SA/V ml/gm	Cleaned Y/N	Filtered Y/N	pH	Al	Fe	K	Na	Si	B	Li	Mn
001-pc	3	5	y	y	11.02	7.8	9.5	55.0	121.0	120.0	46.80	28.6	1.70
002-p	3	5	n	y	11.23	7.8	12.4	64.4	150.0	141.0	59.20	35.9	2.10
003-pc	3	5	y	n	11.09	7.9	8.7	55.6	127.0	128.0	77.60	30.4	1.50
004-p	3	5	n	n	11.10	6.5	11.6	67.3	158.0	135.0	61.40	37.5	2.00
005-pc	3	10	y	y	10.92	6.5	3.1	31.0	66.2	87.0	27.10	16.8	0.50
006-p	3	10	n	y	11.01	7.1	4.8	43.1	86.0	105.0	35.70	22.1	0.00
007-pc	3	10	y	n	10.88	5.8	3.1	31.0	65.3	79.7	25.40	15.7	0.46
008-p	3	10	n	n	11.00	9.7	14.5	47.4	94.8	127.0	38.90	24.2	2.10
009-pc	3	100	y	y	10.19	4.7	0.8	17.2	22.3	51.7	9.34	6.0	0.10
010-p	3	100	n	y	10.08	4.6	0.4	16.9	23.3	52.0	9.53	6.2	0.05
011-pc	3	100	y	n	10.12	4.6	1.5	15.8	22.3	50.7	8.80	5.7	0.20
012-p	3	100	n	n	10.11	5.1	2.0	17.4	24.4	55.2	9.90	6.4	0.30
013-pc	7	5	y	y	11.21	9.4	19.8	71.9	140.0	156.0	64.40	38.1	3.25
014-p	7	5	n	y	11.10	8.1	19.5	84.7	195.0	153.0	74.80	44.1	3.50
015-pc	7	5	y	n	11.16	8.2	16.1	73.7	164.0	161.0	69.40	39.2	2.70
016-p	7	5	n	n	11.29	7.9	17.2	84.0	192.0	170.0	125.00	43.9	2.70
017-pc	7	10	y	y	10.98	6.9	6.7	52.6	97.3	116.0	41.30	24.5	0.90
018-p	7	10	n	y	11.02	6.6	8.5	56.4	119.0	120.0	46.20	27.3	1.20
019-pc	7	10	y	n	11.06	7.0	6.8	46.5	80.0	117.0	41.60	25.2	0.90
020-p	7	10	n	n	11.09	7.0	9.1	102.0	96.9	127.0	48.50	29.0	1.20
021-pc	7	100	y	y	10.10	5.4	0.9	17.2	23.7	61.4	12.20	7.6	0.12
022-p	7	100	n	y	10.02	5.0	1.4	17.8	24.3	58.9	12.00	7.7	0.20
023-pc	7	100	y	n	10.06	4.9	1.8	17.1	23.2	56.0	10.90	7.0	0.20
024-p	7	100	n	n	10.01	5.3	5.5	17.8	25.1	57.7	11.50	7.5	0.80

TABLE A.3. SRL-202-G, Replicate 2, DIW, 90C

Sample Number	Time Days	SA/V ml/gm	Cleaned Y/N	Filtered Y/N	pH	Al	Fe	K	Na	Si	B	Li	Mn
101-gc	3	5	y	y	10.45	7.1	7.4	21	90.0	124.0	25.7	25.60	1.10
102-g	3	5	n	y	10.52	6.3	7.9	35	113.0	140.0	34.8	33.90	1.00
103-gc	3	5	y	n	10.33	5.6	5.0	22	68.0	98.4	23.9	20.90	0.60
104-g	3	5	n	n	10.68	8.0	14.4	26	94.0	124.0	30.0	28.70	1.80
105-gc	3	10	y	y	10.22	6.1	4.0	18	55.0	96.5	17.8	16.90	0.50
106-g	3	10	n	y	10.44	6.3	4.3	19	57.0	101.0	19.8	17.50	0.40
107-gc	3	10	y	n	10.23	5.8	3.4	12	48.2	89.7	16.7	14.60	0.40
108-g	3	10	n	n	10.41	6.5	7.6	16	55.6	99.0	19.8	17.30	0.91
109-gc	3	100	y	y	9.98	4.5	1.2	9	19.7	52.3	6.7	5.60	0.13
110-g	3	100	n	y	9.77	4.6	0.9	8	21.4	54.3	7.2	6.00	0.09
111-gc	3	100	y	n	9.89	4.7	2.9	9	19.1	52.9	6.6	52.90	0.36
112-g	3	100	n	n	9.82	6.0	8.0	11	23.1	61.3	7.9	6.81	1.09
113-gc	7	5	y	y	10.41	6.2	9.6	0	94.3	126.0	29.2	28.60	1.19
114-g	7	5	n	y	10.67	6.0	10.4	26	120.0	148.0	39.1	35.60	1.25
115-gc	7	5	y	n	10.32	34.2	107.0	27	160.0	105.0	51.2	47.40	14.40
116-g	7	5	n	n	10.86	9.7	23.6	35	132.0	147.0	42.9	38.20	2.98
117-gc	7	10	y	y	10.52	6.5	5.3	20	63.0	112.0	20.3	17.80	0.45
118-g	7	10	n	y	10.62	6.0	5.6	22	77.0	121.0	24.7	22.30	0.51
119-gc	7	10	y	n	10.50	7.8	10.4	22	67.0	108.0	21.5	19.40	1.19
120-g	7	10	n	n	10.58	6.9	9.0	21	74.0	115.0	24.0	21.70	0.99
121-gc	7	100	y	y	9.84	4.5	1.1	10	23.0	59.0	8.1	6.40	0.07
122-g	7	100	n	y	9.71	4.5	1.1	0	25.0	62.0	8.7	6.90	0.09
123-gc	7	100	y	n	9.42	4.5	5.2	0	20.8	57.0	7.3	5.90	0.68
124-g	7	100	n	n	9.79	4.9	4.0	11	24.0	63.0	8.5	6.80	0.50

TABLE A.4. SRL-202-P, Replicate 2, DIW, 90C

Sample Number	Time Days	SA/V ml/gm	Cleaned Y/N	Filtered Y/N	pH	Al	Fe	K	Na	Si	B	Li	Mn
101-pc	3	5	y	y	10.71	7.7	11.00	66	163	140.0	68.5	40.5	2.20
102-p	3	5	n	y	11.10	7.6	11.60	75	171	153.0	68.8	41.0	1.94
103-pc	3	5	y	n	10.93	8.9	11.30	60	141	145.0	56.6	34.0	2.00
104-p	3	5	n	n	10.92	16.6	44.00	95	212	204.0	86.3	52.0	7.10
105-pc	3	10	y	y	10.85	7.2	4.80	39	78	97.0	30.7	19.2	0.70
106-p	3	10	n	y	10.89	7.1	5.80	47	101	108.0	38.7	24.2	0.84
107-pc	3	10	y	n	10.80	6.8	5.70	36	77	94.2	30.2	19.1	0.92
108-p	3	10	n	n	10.88	7.7	8.30	47	100	111.0	39.1	24.5	1.30
109-pc	3	100	y	y	10.17	4.8	0.75	16	24	52.1	9.4	5.8	0.09
110-p	3	100	n	y	10.23	5.0	0.84	19	27	54.6	10.1	6.2	0.11
111-pc	3	100	y	n	10.08	4.8	1.30	19	24	52.0	9.3	5.8	0.19
112-p	3	100	n	n	10.13	5.9	7.80	20	26	59.0	10.2	6.4	1.17
113-pc	7	5	y	y	10.98	9.8	20.10	91	209	142.0	86.6	50.5	3.90
114-p	7	5	n	y	11.22	8.8	17.40	97	220	162.0	87.9	50.9	2.80
115-pc	7	5	y	n	11.10	7.9	14.60	79	183	161.0	76.2	44.4	2.60
116-p	7	5	n	n	11.21	13.7	36.60	114	270	213.0	109.0	64.8	6.30
117-pc	7	10	y	y	10.92	6.5	6.90	51	105	106.0	42.0	26.0	1.11
118-p	7	10	n	y	11.05	6.8	7.70	62	135	127.0	53.0	33.0	1.14
119-pc	7	10	y	n	10.90	6.5	6.80	50	103	108.0	40.7	25.5	1.00
120-p	7	10	n	n	11.02	6.7	8.10	57	121	116.0	47.6	29.4	1.23
121-pc	7	100	y	y	9.94	4.3	1.00	22	26	51.0	10.2	6.6	0.13
122-p	7	100	n	y	10.08	4.7	1.05	24	29	56.0	11.4	7.3	0.13
123-pc	7	100	y	n	9.96	4.7	0.86	20	29	55.0	11.4	7.3	0.15
124-p	7	100	n	n	10.04	5.5	4.60	23	33	62.0	12.9	8.2	0.71

TABLE A.5. SRL-202-G, Replicate 3, DIW, 90C

Sample Number	Time Days	SA/V ml/gm	Cleaned Y/N	Filtered Y/N	pH	Al	Fe	K	Na	Si	B	Li	Mn
201-gc	3	5	y	y	10.58	6.40	6.60	0	72.0	112	23.6	22.6	0.79
202-g	3	5	n	y	10.75	5.70	7.60	26	97.0	128	31.5	30.5	0.93
203-gc	3	5	y	n	10.57	6.20	6.90	19	63.0	103	19.0	18.8	0.83
204-g	3	5	n	n	10.65	15.30	42.90	35	126.0	133	41.2	39.8	5.70
205-gc	3	10	y	y	10.34	6.70	4.20	20	51.0	95	16.5	15.8	0.45
206-g	3	10	n	y	10.55	6.30	4.50	21	66.0	107	21.0	20.9	0.48
207-gc	3	10	y	n	10.41	5.80	3.75	33	48.0	86	14.5	13.8	0.44
208-g	3	10	n	n	10.61	14.90	37.70	55	78.0	108	24.6	22.9	4.99
209-gc	3	100	y	y	9.75	4.30	1.70	20	20.0	51	6.6	5.5	0.20
210-g	3	100	n	y	9.99	4.30	0.68	22	20.5	52	6.8	5.7	0.07
211-gc	3	100	y	n	9.99	4.45	2.70	22	19.5	51	6.4	5.6	0.32
212-g	3	100	n	n	10.02	5.40	6.60	27	23.0	57	7.3	6.4	0.82
213-gc	7	5	y	y	10.70	7.20	12.50	16	95.0	136	28.6	27.3	1.40
214-g	7	5	n	y	10.84	7.10	13.50	21	128.0	164	39.4	38.0	1.50
215-gc	7	5	y	n	10.71	7.40	9.20	11	97.0	147	33.0	27.8	0.90
216-g	7	5	n	n	10.86	8.20	16.20	27	128.0	155	42.1	36.2	1.80
217-gc	7	10	y	y	10.53	7.80	5.30	19	66.0	120	22.7	19.1	0.40
218-g	7	10	n	y	10.70	6.30	5.10	25	74.0	121	25.2	21.3	0.40
219-gc	7	10	y	n	10.48	45.80	140.00	85	151.0	101	51.7	45.4	18.40
220-g	7	10	n	n	10.69	5.80	5.10	26	68.0	112	23.6	20.6	0.50
221-gc	7	100	y	y	10.03	5.00	1.10	9	25.0	62	8.6	6.8	0.10
222-g	7	100	n	y	10.04	5.00	1.30	15	25.0	66	8.9	7.4	0.09
223-gc	7	100	y	n	9.80	4.80	4.60	11	21.0	59	7.4	6.2	0.50
224-g	7	100	n	n	10.04	5.20	3.50	12	24.0	64	8.5	6.9	0.40

TABLE A.6. SRL-202-P, Replicate 3, DIW, 90C

Sample Number	Time Days	SA/V ml/gm	Cleaned Y/N	Filtered Y/N	pH	Al	Fe	K	Na	Si	B	Li	Mn
201-pc	3	5	y	y	10.98	6.9	8.6	55	126	19.0	48.2	30.0	1.48
202-p	3	5	n	y	11.21	7.1	10.4	65	158	143.0	62.2	38.0	1.62
203-pc	3	5	y	n	11.04	6.5	7.6	62	118	114.0	46.4	29.0	1.30
204-p	3	5	n	n	11.18	12.2	30.9	68	146	148.0	57.6	36.0	4.50
205-pc	3	10	y	y	10.84	6.5	3.8	37	75	90.3	28.2	19.0	0.50
206-p	3	10	n	y	10.95	6.5	5.1	42	92	101.0	35.0	23.0	0.69
207-pc	3	10	y	n	10.72	7.0	6.6	40	81	98.0	31.0	20.5	0.93
208-p	3	10	n	n	10.97	10.3	21.5	49	94	115.0	35.0	23.5	3.00
209-pc	3	100	y	y	10.19	5.0	0.4	19	25	50.0	8.9	5.8	0.01
210-p	3	100	n	y	10.04	4.5	0.5	18	26	50.0	9.2	6.1	0.80
211-pc	3	100	y	n	10.18	4.7	1.1	18	25	52.0	9.2	6.1	0.10
212-p	3	100	n	n	10.17	5.2	4.9	19	27	56.0	9.7	6.5	0.70
213-pc	7	5	y	y	11.28	9.5	18.0	62	177	157.0	71.2	41.0	3.20
214-p	7	5	n	y	11.36	8.8	19.0	122	208	171.0	84.0	48.0	3.10
215-pc	7	5	y	n	11.11	7.3	13.0	102	173	152.0	73.0	42.0	2.30
216-p	7	5	n	n	11.35	9.0	21.0	125	205	167.0	86.0	48.0	3.30
217-pc	7	10	y	y	11.05	7.3	7.3	75	122	124.0	46.0	28.0	0.70
218-p	7	10	n	y	11.08	6.6	8.2	80	132	125.0	50.0	30.0	1.20
219-pc	7	10	y	n	10.93	14.5	34.0	86	131	170.0	51.0	32.0	5.10
220-p	7	10	n	n	11.15	7.4	10.6	82	136	130.0	52.0	31.0	1.60
221-pc	7	100	y	y	10.09	5.3	1.0	0	32	62.0	12.4	7.6	0.13
222-p	7	100	n	y	10.11	5.4	3.8	20	32	61.0	12.8	8.1	0.60
223-pc	7	100	y	n	10.02	4.7	2.0	17	29	55.0	10.5	6.8	0.30
224-p	7	100	n	n	10.20	5.7	4.4	18	31	63.0	12.2	7.8	0.60

8.0 APPENDIX 8

SUPPLEMENTARY 5 DAY AND 18A DATA FOR SRL-202-G AND SRL-202-P GLASS

TABLE B.1. Supplemental Table, SRL-202-G and SRL-202-P, Deionized Water, 90C

Sample Number	Time Days	SA/V ml/gm	Cleaned Y/N	Filtered Y/N	pH	Al	Fe	K	Na	Si	B	Li	Mn
sr1025gc18	7	5	y	18A	10.40	2.8	0.0	12	65	80	20.5	19.4	0.0
sr1026g18	7	5	n	18A	10.88	3.3	0.2	21	95	121	30.5	27.0	0.0
sr1025pc18	7	5	y	18A	11.28	2.8	0.0	39	112	94	42.0	23.0	0.0
sr1026p18	7	5	n	18A	11.37	3.2	0.0	61	147	132	63.0	34.0	0.0
sr1027gc	5	10	y	y	10.37	4.9	3.2	18	46	80	13.5	13.2	0.3
sr1028g	5	10	n	y	10.60	5.6	4.7	0	69	107	21.0	19.9	0.5
sr1027pc	5	10	y	y	10.98	6.8	4.5	42	92	105	35.0	21.0	0.6
sr1028p	5	10	n	y	11.09	6.8	6.3	50	118	122	46.0	27.0	0.9

DISTRIBUTION

1 - 3	W. F. Perrin, DOE-SR
4 - 50	SRL File, 773-A
51-150	DOE OSTI-TIC (for distribution under UC-701)

Distribution

1-3. W. F. Perrin, DOE-SR, 703-A
4. D. L. McIntosh, SRS, 773-A
5. E. W. Holtzscheiter, SRS, 773-A
6. J. R. Knight, SRS, 773-A
7. J. T. Carter, SRS, 704-1T
8. C. T. Randall,SRS, 704-1T
9. L. F. Landon, SRS, 704-1T
10. M. J. Plodinec, SRS, 773-A
11. N. E. Bibler, SRS, 773-A
12. G. G. Wicks, SRS, 773-A
13. A. A. Ramsey, SRS, 704-S
14. M. K. Andrews, SRS, 773-62A
15. B. C. Ha, SRS, 773-A
16. K. E. Mottell, SRS, 773-43A
17. R. F. Schumacher, SRS, 773-42A
18. D. C. Beam, SRS, 773-A
19. D. F. Bickford, SRS, 773-A
20. S. L. Marra, SRS, 704-S
21. J. R. Harbour, SRS, 773-43A
22. J. F. Sproull, SRS, 704-S
23. L. O. Westphal, HWVP at SRS, 704-S
24. C. M. Jantzen, SRS, 773-A
25-50 SRL Records, 773-A