CONF-901116--6

UCRL-JC--103653

DE91 005263

by 207
DEC 24 1990

The Effect of Sample Preparation
Methods on Glass Performance

M. S. Oh
V. M. Oversby

This Paper was Presented at the
XIV Symposium on the Scientific
Basis for Nuclear Waste Management
Materials Research Society
Boston, MA
November 26-29, 1990
October 24, 1990

Lawrence
Livermore
National
Laboratory

This is a preprint of a paper intended for publication in a journal or proceedings. Since changes may be made before publication, this preprint is made available with the understanding that it will not be cited or reproduced without the permission of the author.

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

DISCLAIMER

This document was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor the University of California nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial products, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or the University of California. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or the University of California, and shall not be used for advertising or product endorsement purposes.

THE EFFECT OF SAMPLE PREPARATION METHODS ON GLASS PERFORMANCE

M. S. OH and V. M. OVERSBY, Chemistry and Materials Science Department,
Lawrence Livermore National Laboratory, Livermore, CA 94550

ABSTRACT

A series of experiments was conducted using SRL 165 synthetic waste glass to investigate the effects of surface preparation and leaching solution composition on the alteration of the glass. Samples of glass with as-cast surfaces produced smooth reaction layers and some evidence for precipitation of secondary phases from solution. Secondary phases were more abundant in samples reacted in deionized water than for those reacted in a silicate solution. Samples with saw-cut surfaces showed a large reduction in surface roughness after 7 days of reaction in either solution. Reaction in silicate solution for up to 91 days produced no further change in surface morphology, while reaction in DIW produced a spongy surface that formed the substrate for further surface layer development. The differences in the surface morphology of the samples may create microclimates that control the details of development of alteration layers on the glass; however, the concentrations of elements in leaching solutions show differences of 50% or less between samples prepared with different surface conditions for tests of a few months duration.

INTRODUCTION

The composition of aqueous solutions used in waste glass testing has been shown to have a strong influence on the leaching behavior of the glasses in short term tests. Data have also suggested that sample preparation methods, because of the resulting variability in glass surface area, influence the results of short term tests; however, this phenomenon has not been studied in a controlled manner. It is also possible that both surface preparation methods and chemical composition of leaching solutions could affect the nature of the alteration layers that develop on the glasses. If different assemblages of metastable reaction products form on the glass surfaces in leachants of different chemical composition, the influence of the solution composition might be long-lasting.

We conducted a controlled experiment to investigate the effects of sample preparation method and leaching solution composition on

1. The release rate and total release of glass constituents to solution;
2. The morphology of the surface layer development on the altered glasses;
3. The chemical and phase composition of the alteration layers.

This report will focus on the first two topics. Results of surface layer characterization will be reported in a separate communication.

EXPERIMENTAL PROCEDURES

The glass used in these studies was SRL 165 Black Frit with uranium added. This glass was selected because of its relevance to the DWPF and to allow us to compare our results to those of previous work by J. K. Bates and co-workers [1, 2 and references contained therein]. Two sample preparation methods were used. One set of samples was core-drilled from a slab of material and then sliced into wafers 0.24 cm thick using procedures similar to those used in the MCC-1 test protocol. Samples were cut using a low

MASTER
DISTRIBUTION OF THIS DOCUMENT IS UNLIM
pr

concentration Buehler diamond cut-off wheel, a speed setting of 5, and a counter balance weight of 75 gm with the counter weight extended one inch from minimum. The cut samples were not polished and were expected to have a surface finish approximating a 240 grit polish [3]. The second sample set was prepared by casting the glass in Pt-5% Au molds to provide samples with completely "as-cast" surfaces. The molds have slanted sides to facilitate removal of the casting. The cast samples had a top diameter of about 1 cm, a bottom diameter of 0.6 cm, and an average thickness of 0.2 cm. As the cast glass cools the glass adheres to the mold and contracts in the center of the casting, resulting in a concave top surface with an edge of thin glass. All glass specimens used were prepared at ANL under the supervision of J. K. Bates to ensure that the surface finish would be comparable to that of samples used in previous work by Bates and co-workers.

Two leaching solutions were used. The first was prepared using a soluble silica standard solution prepared with sodium bicarbonate and nitric acid additions to give a leach solution containing 60 ppm Si, 174 ppm Na, 200 ppm bicarbonate, and 250 ppm nitrate. This solution would contain less than the silica concentration found in previous long-term testing of SRL 165 glass alteration [4] and is a poorly-poised buffer solution with an initial pH of 8.8. Testing in this solution should produce behavior that is representative of long-term alteration of the glass at near-steady-state conditions. The second leaching solution was high-purity deionized water. This solution will produce an initial rapid dissolution rate of the glass that may have an effect on the composition and thickness of the alteration layer developed on the glass surface and may produce a layer that is different from the layer produced using leaching solutions representative of those present under steady-state conditions.

Several glass samples of each preparation type were examined by SEM to document the condition of the surfaces; representative examples are shown in Fig. 1. The saw-cut surfaces were extremely rough and showed a wavy pattern with wave lengths of 5 to 20 micrometers. Small particles could be seen on the cut surface with sizes ranging from a few micrometers down to the limit of resolution of the SEM photo. The cast samples were very smooth, with the only blemishes noted being small particles that appeared to be lying on and in the surface and gas bubbles, which generally were seen only on the bottom surfaces of the samples.

Fig. 1 Scanning electron microscope photos of unreacted glass samples; a) saw-cut surface; b) top of cast surface.

Samples of each type of glass were tested in parallel at 90°C for total test durations of 1, 2, 7, 14, 28, 60, and 91 days. Duplicate samples of each glass surface preparation type were tested in each leaching solution and triplicate samples of each type were tested in the silicate solution for the three longest test periods. The geometric surface area of the glass samples was calculated for each specimen tested. For cast samples the surface area was generally between 1.8 and 1.9 sq. cm, while for the cut samples the surface area was usually very close to 2.15 sq. cm. The surface area estimates made no attempt to include the irregularities in surface finish revealed in the SEM photos.

The experiments were conducted in Teflon leaching containers with the glass samples set on a Teflon support. Details of the cleaning procedures used and the experimental procedure are available [5]. At the end of the reaction period leaching solutions were characterized for pH, anions by ion chromatography, and cations by a combination of ICP-ES, ICP-MS, and AA. Two analysts using different instruments and/or analytical methods made determinations of Al, B, Ca, Fe, Li, Mg, Mn, Na, and Si. Because of the low solution concentrations, only ICP-MS could be used for determination of Ni, Ce, Sr, and U. One reaction vessel was carried through the procedures with leaching solution but no glass sample for each exposure time to serve as a procedural blank. Analysis of the blank solutions showed no indication of contamination for any of the elements of interest in this study [5]. One or more of the glass samples from each duplicate or triplicate set was characterized by SEM at the end of the reaction period to monitor the changes in surface appearance of the specimen. Samples were stored for use in future layer characterization studies, with storage under controlled humidity conditions for samples not used for SEM work or under ambient conditions in closed plastic vials for samples that had been carbon coated and exposed to vacuum conditions in the SEM.

RESULTS AND DISCUSSION

The alteration of glass in aqueous solutions involves the development of hydrated layers on the glass surface. The key to the mechanism of glass alteration lies in the understanding of how these layers develop and whether the layer itself can control or contribute to control of further glass degradation. Unfortunately, most of the tools available to study the glass alteration layers in the detail required if we are to elucidate the reaction mechanism require that the sample be subjected to coating with a conductive medium and then be placed in a vacuum for analysis. One might argue that the conductive coating will not interfere with the layer itself; however, it is difficult to imagine that placing a glass specimen with a hydrated layer into a vacuum system will not cause changes in the layer. Interpretations of the SEM photos of reacted glass surfaces must include allowance for this artifact of the experimental method.

Cast samples reacted in silicate leaching solution showed very little change in surface appearance for even the longest reaction times. Close examination of the SEM photos reveals that the small particles on the original unreacted surfaces are still present after short reaction times. They appear to have been imbedded in the original cast surface and are now imbedded in a forming reaction layer. After 14 days of reaction, larger particles that appear to have been precipitated from the solution phase are seen on the reacted glass surface. There is no significant difference in surface appearance for the cast samples reacted in silicate solution for 14 days and for periods up to 91 days. For cast samples reacted in deionized water, the abundance and size of the apparently precipitated particles at a reaction time of 14 days are much larger than for silicate solution; in

addition, the layer has a slightly "spongy" appearance. The SEM photo of the cast sample reacted in DIW for 28 days shows a cracked surface layer that is interpreted to be the result of vacuum dehydration of the hydrated alteration layer. Precipitated phases are now rare on the surface, but close examination shows that some precipitates have been covered by the smooth alteration layer. Exposure of 91 days in DIW has produced a layer a few micrometers thick that cracks under our experimental conditions, producing small flakes on the smooth surface. This suggests the reaction layer formed in DIW is not adhering to the glass surface. A few particles that appear to be separate precipitated phases are also seen. Representative SEM photos are reproduced in Fig. 2.

The surface appearance of saw-cut samples reacted in silicate solution showed a dramatic reduction in surface roughness occurring over the first 7 days of reaction and then a remarkably similar surface appearance for all reaction times up to and including 91 days. Only minor amounts of particulate material that might be precipitated phases could be seen on the sample surface for any of the reaction times. The saw-cut samples exposed to deionized water showed a progression of surface development. After 7 days there was even less residual surface roughness than in the silicate-leached samples (Fig. 3). After 14 days the DIW reacted surface had a "spongy" appearance; with longer reaction times this surface became more coherent,

2a

2b

2c

2d

Fig. 2 SEM photos showing surface appearance of cast glass samples after reaction in a) silicate solution for 91 days; b) DIW for 14 days; c) DIW for 28 days; d) DIW for 91 days.

until with 91 days of reaction the surface appearance is similar to that for 7 days (Fig. 3). The similar appearance of the 7 and 91 day reacted surfaces is fortuitous, since the 7 day surface reflects residual roughness from the unaltered glass, while the 91 day surface represents topology of forming the alteration layer from the 14 day "sponge".

Full tables of analytical data for solution samples taken at the end of the reaction period are given in reference 5. Data from procedural blanks showed that the silicate solution maintained stable concentrations of Na, Si, and nitrate throughout the reaction period. Bicarbonate concentrations decreased slowly from the initial value of about 200 ppm to a value of 128 ppm at 60 and 91 days. Values of pH averaged about 9.1 to 9.2 for the silicate blanks. The DIW blanks had final pH values of 5.2 to 4.3 and showed bicarbonate concentrations of about 50 ppm for the longer reaction times, which accounts for the low pH. The variation in bicarbonate for both sets of blanks is interpreted to be the result of CO_2 equilibration of the solutions with the atmosphere of the oven through the teflon container walls. Data for runs with glass samples showed the same behavior for bicarbonate concentrations.

Concentration of Si increased from the initial value of 60 ppm in silicate solution to 80 ppm after 91 days of reaction for both cast and

3a

3b

3c

3d

Fig. 3 SEM photos showing surface appearance of saw-cut glass samples after reaction in a) silicate solution for 7 days; b) DIW for 7 days; c) DIW for 14 days; d) DIW for 91 days.

saw-cut samples. Concentration of Si in DIW leachates after 91 days of reaction was 30 ppm for cast samples and 47 ppm for saw-cut samples. Glass samples reacted in silicate water produced solutions with pH that increased slowly to a maximum value of 9.8 at 60 days and then decreased to 9.2 at 91 days for both surface preparation types. Saw-cut samples reacted in DIW produced pH values of 9.6 after 2 days of reaction for all reaction times up to 60 days; at 91 days pH had decreased to 8.6. Cast samples showed a slower increase in pH, reaching a maximum of 9.3 after 7 days reaction in DIW and decreasing to 9.0 at 91 days.

Elements with high solubilities, such as boron, lithium, and uranium, showed the expected pattern of solution concentrations for samples leached in silicate solution. Fig. 4 shows solution concentration data for Li and B for saw-cut and cast samples reacted in silicate solution. The total release to solution from saw-cut samples is greater than that for cast samples for all reaction times. Solution data for less soluble elements showed higher concentrations from reaction of saw-cut samples for short exposure times, but after 28 days, the highest concentrations of Fe and Sr were observed for cast samples.

Plotting data as solution concentrations may mask differences due to small differences in sample weight and solution volume. A more meaningful approach is to present the data as loss of each element per gram of glass. Selected analytical data normalized to loss per gram of glass are given in Table 1. The data shown are the average value for duplicate or triplicate glass samples for each reaction time. Where multiple analytical results were available for a given element, these results were first averaged to give a single value for the glass sample and then the average of the multiplet samples was done. Data were then converted into loss of the element per gram of glass. The original solution volume was approximately 21 ml in all cases; thus, the geometric surface area to leaching solution volume for these experiments was approximately 0.1 cm^2 . The saw-cut samples appear in SEM photos to have at least a factor of two "real" surface area in comparison to the geometric calculated area.

Fig. 4 Boron and lithium concentrations in silicate liquid for saw-cut and as-cast glass samples.

TABLE 1. LOSS TO SOLUTION DURING LEACHING, MICROGRAMS PER GRAM OF GLASS

SAMPLE	ELEMENT	LEACHING TIME, DAYS						
		1	2	7	14	28	60	91
CAST-Si	Al	1.2	10	24	38	62	67	83
CUT-Si	Al	4	12	25	37	42	51	65
CAST-DIW	Al	<3	<3	41	85	107	116	156
CUT-DIW	Al	11	59	82	111	127	128	136
CAST-Si	B	3.0	10.3	20.1	36	57	71	85
CUT-Si	B	5.5	14	25	41	45	60	75
CAST-DIW	B	1.4	5.3	50	88	123	147	199
CUT-DIW	B	19.6	60	95	133	156	172	195
CAST-Si	Fe	2.6	4.2	11.9	39	90	119	131
CUT-Si	Fe	1.7	3.6	11	31	42	69	83
CAST-DIW	Fe	<.6	<.6	2.5	3.6	6.1	12.3	22
CUT-DIW	Fe	3.3	4.6	8.6	16.8	25	43	32
CAST-Si	Li	4.2	11.9	22	44	69	76	95
CUT-Si	Li	7.1	16.6	29	51	54	66	84
CAST-DIW	Li	2.1	6.4	53	107	140	157	250
CUT-DIW	Li	21	64	99	160	173	179	194
CAST-Si	Sr	0.18	0.29	0.5	0.7	1.1	1.4	1.6
CUT-Si	Sr	0.20	0.30	0.14	0.50	0.62	0.86	0.96
CAST-DIW	Sr	0.08	0.24	1.2	1.7	1.2	1.0	1.5
CUT-DIW	Sr	0.6	1.05	0.97	0.82	0.77	0.79	0.72
CAST-Si	U	1.4	4.4	9.2	16.6	24.4	23.7	35.6
CUT-Si	U	2.0	6.1	11.3	16.4	19.4	18.8	30
CAST-DIW	U	0.03	0.2	3.6	7.7	9.8	11.3	28.3
CUT-DIW	U	0.97	4.4	9.4	14.8	20.4	21.3	31.8
CAST-DIW	Na	121	171	274	454	461	598	937
CUT-DIW	Na	182	406	460	572	547	695	782
CAST-DIW	Si	24	73	555	981	1216	1629	2053
CUT-DIW	Si	233	668	959	1319	1480	1828	1861

For the shortest reaction time of 1 day, loss per gram of glass in silicate leaching solutions showed greater values for all elements except Fe in tests using saw-cut samples. Differences ranged from a factor of about 2 for Al, B, and Li, 1.5 for U, and 1.1 or less for Sr. As the reaction time increased, the differences in normalized loss to solution for the two sample surface types decreased and the amount of loss in tests using cast samples became higher than that for tests with saw-cut samples for all elements. This effect is illustrated in Fig. 5 for the boron and lithium data. The times at which the cross-over in normalized loss occurred were Fe < 1 day, Sr < 7 days, Al < 14 days, and B, Li, and U < 28 days. After 91 days of reaction, the largest differences for the two sample types were for Fe and Sr, with cast samples having about 1.5 times the normalized release to solution as compared to saw-cut samples. Concentrations of elements in the leaching solutions showed a somewhat simpler pattern, with the saw-cut samples having higher solution concentrations than the cast samples for all reaction times for Al, B, Li, and U, and similar to slightly lower concentrations for Fe and Sr.

Fig. 5 Boron and lithium loss per gram of glass in silicate liquid for saw-cut and as-cast glass samples.

An explanation for the differences in solution chemistry and normalized loss per gram of glass for the six elements for which data are given in Table 1 may be found in the surface topology of the specimens. At long reaction times the normalized loss of elements with high predicted solubility [B, Li, U] is only about 10% higher for the cast samples than the saw-cut samples. The normalized loss per gram of glass for these elements shows a nearly perfect linear dependence on time for the reaction times of 28, 60, and 91 days, as shown in Fig. 6, and the difference between saw-cut and cast samples remains constant. The largest difference in normalized loss occurs for Fe, which has the lowest expected solubility of the six elements under consideration. The surface roughness on the saw-cut specimens provides "pits" and "crevices" in which chemical microclimates can rapidly develop. These microclimates can enhance the precipitation of secondary phases, which if rapidly precipitated may incorporate some of the more soluble elements.

The initial dissolution of the glasses in silicate solutions may be dominated by surface regions with high surface energy, which would be expected to cause an initial rapid dissolution. In the case of the cast samples, this high energy surface occurred at the rim of the sample and release would be more likely to remain in solution than in the case of the saw-cut samples where the high energy regions occurred uniformly over the specimen and precipitation could occur in the pit and crevice microclimates. Release to solution from this high energy region probably occurs during the first few days of reaction, based on the post-reaction SEM data for saw-cut samples, which show similar surface morphology for all reaction times greater than or equal to 7 days in silicate solution.

Comparison of leaching solution data for the two sample preparation types in DIW showed a similar pattern of greater release from the saw-cut samples at short leaching times, with a cross-over in relative release occurring at longer exposure times. In the case of DIW, however, the cross-over occurred at much longer leaching times (except for Sr). For U and Fe release in DIW, the cross-over point did not occur even at 91 days of exposure. Total releases for the two sample types in DIW at 91 day exposure

Fig. 6 Boron loss per gram of glass in silicate liquid for saw-cut and as-cast samples.

Fig. 7 Iron release to solution in DIW and silicate liquid for saw-cut and as-cast glass samples.

times agreed to within 20 relative percent for Al, B, Li, U, Na, and Si. Concentration in the solution phase was higher for saw-cut samples for all of the elements in Table 1 except Sr. In the case of DIW reaction, both sample types will experience an initial rapid dissolution due to the high degree of undersaturation of the solution with respect to Si, causing a large increase in pH. This can result in precipitation at the sample surface of phases with

low solubility, such as hydrous oxides, in the forming reaction layer and formation of local chemical microclimates that dominate further reactions. Fig. 7 shows iron release as a function of time for both leaching solutions and sample preparation types. The greater release of iron from the samples reacted in silicate solution suggests that locally high pH caused by rapid glass dissolution in DIW controls Fe solubility for those samples, while in silicate liquid the pH is less severely altered by glass dissolution (even in local regions in saw-cut samples) and greater release of iron to solution can occur. Further evidence for the effects of rapid glass dissolution in DIW can be found by examining the alteration layers formed, which show a complicated, multilayer structure for those developed in DIW and a simple single layer for those developed in silicate liquids [6].

CONCLUSIONS

Visual observation by SEM of reaction layers developed on cast and saw-cut glass surfaces showed that the layers formed in silicate leachants were qualitatively different from those formed in DIW, with the layers developed in DIW showing a spongy nature early in their development. Loss of elements to silicate leaching solution when normalized to a per-gram-of-glass basis was greater from saw-cut samples at short reaction times, but greater from cast samples at long reaction times. This is interpreted to be due to trapping of some liquid in the pits and crevices of the saw-cut samples as well as precipitation due to chemical microclimates in those regions. Solution concentrations for Al, B, Li, U, and Sr are remarkably similar for saw-cut and as-cast samples in both leaching media. This similarity, when contrasted with the clear differences in layer development seen in the SEM data, suggests that testing of glass using only data for release to solution may lead to erroneous conclusions about glass durability.

ACKNOWLEDGEMENTS

We thank the many people who helped us in this work by performing analyses on the samples studied - J. Deering (IC and C), T. Duewer and A. Goins (ICP-ES), C. Otto (ICP-MS), C. Strickland (AA), and J. Yoshiyama (SEM). Work done under the auspices of the U. S. Department of Energy by the Lawrence Livermore National Laboratory under Contract No. W-7405-ENG-48.

REFERENCES

1. W. L. Ebert and J. K. Bates, The reaction of synthetic nuclear waste glass in steam and hydrothermal solution, in Mat. Res. Soc. Symp. Proc. Vol. 176, V. M. Oversby and P. W. Brown (ed.), pp. 339-346 (1990).
2. J. K. Bates, T. J. Gerding, and A. B. Woodland, Parametric effects of glass reaction under unsaturated conditions, in Mat. Res. Soc. Symp. Proc. Vol. 176, V. M. Oversby and P. W. Brown (ed.), 347-354 (1990).
3. J. K. Bates, Letter to M. S. Oh, January 9, 1989.
4. N. E. Bibler, G. G. Wicks, and V. M. Oversby, Leaching Savannah River Plant Nuclear Waste Glass in a Saturated Tuff Environment, Lawrence Livermore National Laboratory Report UCRL-91258, November 1984.
5. M. S. Oh and V. M. Oversby, The Effect of Sample Preparation Methods on Glass Performance, Lawrence Livermore National Laboratory Report UCRL-JC-103653, November 1990.
6. D. L. Phinney and V. M. Oversby, work in progress.

Appendix A

Experimental Procedures for Nuclear Waste Glasses Degradation Project

I. Cleaning Procedures

Ia. New Teflon Leaching Containers

This procedure is a modification of PCT1.

1. Bake empty vessels in an oven at 200°C for one week.
2. Soak for 1 hour in 6 M HNO_3 + 0.2 M HF. Use a one-gallon polyethylene (PE) container.
3. Rinse with 3 container volumes of distilled water.
4. Soak in 6 M HNO_3 for 4 hours at 50°C. For steps 4-6, use 9x6x5 in PE box with lid and a water bath.
5. Soak for 30 minutes in >60°C in distilled water.
6. Soak for at least 8 hours in fresh distilled water at 80°C.
7. Boil for 30 minutes in fresh distilled water. Use a Pyrex beaker and a hot plate.
8. Measure pH of distilled water and make sure it is between 5 and 7. Rinse with a minimum of 3 container volumes of distilled water until the pH of the two consecutive rinse solutions is within 0.5 pH unit of the original distilled water.
9. Measure pH of deionized high purity water (DI water) and make sure it is between 5 and 7. Fill the container about 95% full with DI water. Close the lid and leave in a 90°C oven for at least 16 hours.
10. Measure the pH of an aliquot of the water. If the pH is not in the range 5.0 - 7.0, repeat steps 7 through 10.
11. If the 5.0 - 7.0 pH range cannot be achieved by 3 repetitions of steps 7 through 10, then repeat the cleaning and testing procedure, starting at step 4.
12. Put a support to each container and mark the containers with a unique identifier, and the date when cleaning was completed.
13. Monitor the oven temperature continuously every four hours if the data logger is connected. If not, monitor the temperature daily.

Ib. Polyethylene/Polypropylene containers

1. Wash with tap water. Use soap if oily and then rinse very thoroughly with tap water.
2. Rinse with distilled water twice.
3. Add distilled water to about 95% full and close the lid. Leave in the oven at 60°C for 2 days.
4. Empty the container and add fresh distilled water. Repeat step 3 two more times.
5. Invert the container and let it drip dry on a clean lab paper or Kimwipes.
6. Mark it 'Clean'.

Ic. Glass Disk

1. Put a disk in 20 ml clean PE bottle and add 15 cc of DI water. Clean it ultrasonically for 15 minutes.
2. Add fresh DI water and repeat step 1.
3. Put a water-cleaned disk into 15 cc fresh ethanol in a 20 ml PE bottle and clean it ultrasonically for 15 minutes.
4. Add fresh ethanol and repeat step 2 two more times.
5. After the final ethanol cleaning, place disk into clean and identified PE vial. Use a clean Teflon support in each vial. Put the vial into a 90°C oven for about two to four hours until dry.
6. Place disk into clean and identified PE vial. Use a clean Teflon support in each vial.

Id. Used Teflon Leaching Containers

This is a modification of PCT1.

1. Rinse the vessel, lid and the support with distilled water.
2. Submerge in 1% HNO_3 at 90°C for 1 hour.
3. Rinse thoroughly with distilled water.
4. Boil in distilled water for 1 hour. Rinse with distilled water 3 times.
5. Measure pH of DI water. Make sure the pH is in the range of 5.0 - 7.0. Fill the container 95% full of DI water. Close the lid and leave in a 90°C oven for at least 24 hours.
6. Measure the pH of an aliquot of the water.
7. If the pH is not in the range of 5.0 - 7.0, repeat steps 4 through 6.
8. If the 5.0 - 7.0 pH range cannot be achieved after 3 repetitions of steps 4 through 6, then repeat steps 2 through 6.
9. Mark the container with the date when cleaning was completed.

II. pH Measurements

This procedure is for Orion combination electrode model 8135, which has a flat bottom and design for small samples. The pH meter was Orion model SA 520. For maintenance of the electrode and meter, follow the instruction in manual.

1. Calibrate with buffers 7 and 4 or 7 and 10. Use auto-calibration.
2. Rinse the electrode with DI water and wipe the vertical surface with Kimwipes.
3. Instead of drying the flat bottom surface with Kimwipes, dip the flat surface of the electrode to 1 cc of the sample solution.
4. Put the electrode to 2 cc of the sample solution for the measurement.

III. Leaching Solution Preparation

Prepare fresh batch of solutions for each series of experiments.

A. Solution A - 500 cc of 60 ppm Si, 200 ppm HCO_3^- , 250 ppm HNO_3 .

1. Prepare 50 cc of 1200 ppm Si solution.

Weigh a 125 cc wide mouth polyethylene bottle. Add 6 cc of 10 mg Si/ml solution and weigh bottle + Si solution. Add 44 cc of DI water. Weigh bottle + Si solution + water and calculate the exact concentration of Si:

$$\text{Si conc} = C_{\text{Si}} (\text{ppm}) = 10,000 \times \text{Si solution} / (\text{Si solution} + \text{Water}).$$

2. Prepare 100 cc of 2000 ppm HCO_3^- solution.

Weigh a 125 cc wide mouth polyethylene bottle. Add 0.275 g of NaHCO_3 and weigh bottle + NaHCO_3 . Add 99.7 g of DI water. Weigh bottle + NaHCO_3 + water and calculate the exact concentration of HCO_3^- .

$$\text{HCO}_3^- \text{ conc} = C_{\text{bic}} (\text{ppm}) = 10^6 \times \text{wt NaHCO}_3 \times 0.7262 / (\text{Water} + \text{wt NaHCO}_3)$$

3. Prepare 200 g of 5000 ppm HNO_3

Use a 250 cc narrow mouth polyethylene bottle. Weigh the bottle and add 100 g DI water. Weigh bottle + water. Add 1 cc of concentrated HNO_3 solution (70.6%, density = 1.423 g/cm^3) and weigh. Then add 98.6 g water so that the total weight in the bottle is 200 g.

$$\text{HNO}_3 \text{ conc} = C_{\text{N}} (\text{ppm}) = 10^6 \times \text{wt HNO}_3 \times 0.706 / (\text{water} + \text{HNO}_3).$$

4. Weigh a 1000 cc wide mouth polyethylene bottle. Add 400 cc of DI water and weigh (W_w).

5. Add 25 cc of 1200 ppm Si solution and weigh (W_{Si}).

6. Add 50 cc of 2000 ppm HCO_3^- solution and weigh (W_{bic}).

7. Add 25 cc of 5000 ppm solution HNO_3 and weigh (W_{N}).

8. Calculate the exact concentration of each component.

$$C_{\text{Si},f} (\text{ppm}) = C_{\text{Si}} \times W_{\text{Si}} / (W_w + W_{\text{Si}} + W_{\text{bic}} + W_{\text{N}})$$

$$C_{\text{bic},f} (\text{ppm}) = C_{\text{bic}} \times W_{\text{bic}} / (W_w + W_{\text{Si}} + W_{\text{bic}} + W_{\text{N}})$$

$$C_{\text{N},f} (\text{ppm}) = C_{\text{N}} \times W_{\text{N}} / (W_w + W_{\text{Si}} + W_{\text{bic}} + W_{\text{N}})$$

$$C_{\text{Na},f} (\text{ppm}) = C_{\text{Si},f} \times 1.6429 + C_{\text{bic},f} \times 0.377$$

9. For 600 cc of total solution, use 30 cc of 1200 ppm Si solution, 60 cc of 2000 ppm HCO_3^- solution, 30 cc of 5000 ppm solution HNO_3 , and 480 cc DI water.

10. Measure the pH of the solution.

11. Allow to equilibrate for at least 4 hours. Remeasure pH.

IV. Leaching Procedures

1. Mark the leaching container (the vessel and lid). Make sure markings are dry and weigh the vessel and the lid separately.
2. Add the Teflon basket to the vessel and weigh.
3. Weigh the glass frit. Add the frit and weigh.
4. Add 21 cc of leaching solution or DI water (use Pipetmen P-5000, 5 cc capacity, four times and P-1000, 1 cc capacity, once) to the leaching assembly and weigh. Make sure the lid is closed tightly.

Repeat 1-4 for all containers.

5. Put leaching containers to a preheated oven and mark the time. Put a pan of water in the oven to reduce evaporation loss.
6. Monitor the oven temperature every 4 hours, if the data-logger is connected, and daily if the data-logger is not available. Add water to the water pan as necessary.

After Leaching

7. Take the containers out of the oven and mark the time.
8. Let the solution cool down at least for an hour and weigh.
9. Take solution samples as outlined in steps 13-19. When the solution level is below the support, lift out the frit and let it dry in air for an hour. Use Teflon coated tweezers.
10. Puncture a small hole into the lid of the vial which stored the frit before leaching. Transfer the frit to the labeled vial.
11. Submit samples labeled "a" to SEM lab. After SEM analysis, frits will be submitted for SIMS.
12. Add a water to a desiccator instead of desiccant for moisture control. Store the vial in the desiccator until analysis. Keep the hole in the desiccator open. Samples with "b" are for NRP and samples with "c" are for X-ray Tomography.

For steps 13-19, use Pipetmen P-1000 (1 cc capacity) for transferring solution samples into PE centrifuge tubes (12 or 7 ml capacity) with lid which are used for submitting solution samples, except carbonates, and pH measurement.

13. Take 3 cc out for pH measurement. Put 1 cc into one tube and 2 cc to the second tube.
14. Take 3 cc for ICP-ES measurement.
15. Take 2 cc for AA measurement.
16. Take 3 cc for IC measurement.
17. Take 2 cc for ICP-MS measurement.
18. Take 3 cc for the second ICP-ES measurement.
19. Take 2 cc for carbonate (HCO_3^- plus $\text{CO}_3^{=}$) measurement. Use non-porous glass vials for submitting samples.

20. Keep the rest in a PE centrifuge tube.
21. Make pH measurements.
22. Submit solution samples for analysis.

APPENDIX B

Solution Analysis Data

Note: CS = Cored and sliced sample
CA = Cast sample
Si = Silicate leachate
W = Deionized water leachate

Samples labeled CSSi3A, etc. were reacted for 2 days rather than the originally planned 3 days, and those labelled SCSi56A, etc. were reacted for 60 days rather than the originally planned 56 days.

CORED AND SLICED SAMPLES, SILICATE WATER SOLUTION CONC., PPM

	CSSi1A	CSSi1B	CSSi3A	CSSi3B	CSSi7A	CSSi7B	CSSi14A	CSSi14B	CSSi28A	CSSi28B	CSSi28C	CSSi56A	CSSi56B	CSSi56C	CSSi91A	CSSi91B	CSSi91C
Al-T	0.11	0.12	0.30	0.31	0.51	0.56	0.80	1.09	0.96	1.09	1.01	1.54	1.23	1.36	1.60	1.63	1.56 Al-T
Al-A	0.06	0.06	0.23	0.23	0.54	0.59	0.61	0.83	0.93	1.06		1.24	0.91	1.05			Al-A
B-T	0.13	0.13	0.32	0.32	0.54	0.57	0.83	1.07	1.02	1.18	1.02	1.74	1.29	1.39	1.97	1.83	1.74 B-T
B-A	0.12	0.12	0.30	0.31	0.54	0.61	0.82	1.00	0.95	1.17		1.59	1.18	1.31			B-A
Ca-T	0.06	0.07	0.10	0.09	0.11	0.31	0.09	0.13	0.09	0.09	0.08	0.34	0.12	0.16	0.18	0.22	0.11 Ca-T
Ca-A	0.07	0.02	0.07	0.05	0.03	0.02	<.004	<.004	0.03	0.03		0.13	0.08	0.14			Ca-A
Fe-T	0.04	0.04	0.08	0.08	0.24	0.25	0.60	0.82	0.54	1.12	0.96	1.90	1.35	1.72	2.33	2.07	1.77 Fe-T
Fe-A	0.04	0.03	0.08	0.08	0.24	0.26	0.61	0.80	1.08	1.22		1.87	1.28	1.68			Fe-A
K-A	<3	<3	<3	<3	<3	<3	<3	<3	<3	<3		<3	<3	<3			K-A
Li-AA	0.17	0.17	0.39	0.39	0.60	0.65			1.19	1.42	1.28	2.04	1.58	1.75	2.21	2.04	1.99 Li-AA
Li-A	0.15	0.15	0.33	0.37	0.63	0.70	1.08	1.24	1.16	1.39		1.53	1.16	1.29			Li-A
Mg-A	<5	<5	<5	<5	0.63	0.47	0.56	0.56	0.52	0.52		<5	<5	<5			Mg-A
Mg-T	0.03	0.03	0.06	0.06	0.07	0.09	0.12	0.25	0.14	0.14	0.12	0.20	0.18	0.19	0.23	0.22	0.19 Mg-T
Mn-A	<.08	<.08	<.08	<.08	<.08	<.08	0.09	0.10	0.16	0.16	127	209	134	185	247	224	204 Mn-C, ppb
Mn-C, ppb					37	41	90	95	156	149							Mo-A
Mo-A	<.1	<.1	<.1	<.1	<.1	<.1	<.1	<.1	<.1	<.1		<.1	<.1	<.1			Na-A
Na-A	182	184	177	190	188	191	188	191	190	194	155	208	191	198	200	191	235 Na-AA
Na-AA									146	154		274	245	276			Ni-A
Ni-A	<.1	<.1	<.1	<.1	<.1	<.1	0.11	0.11	<.1	<.1	19	<.1	<.1	<.1	21	36	21 Ni-C, ppb
Ni-C, ppb					12	12	19	11	28	22		23	19	21			Si-A
Si-A	62.4	62.6	61.8	64.8	63.8	65.2	66.0	69.0	60.7	63.8		79.9	71.0	74.3	82.8	82.9	80.5 Si-T
Si-T	60.2	59.8	61.4	61.6	63.9	59.8	65.7	70.5	70.4	73.3	71.1	80.1	73.2	74.5	58.1	59.2	59.6 Si-AA
Si-AA	54.5	53.8	54.5	55.2	57.1	53.8			55.7	56.8	55.4	57.9	52.2	53.2	<.2	<.2	<.2 Ce, ppb
Ce, ppb	0.05	0.05	0.17	0.13	<.04	0.14	<.04	<.05	0.6	0.5	0.4	0.7	0.3	0.5			22 Sr, ppb
Sr, ppb	4.8	4.3	6.8	6.5	4.0	2.4	12	11	15	15	14	22	19	20	24	25	726 U, ppb
U, ppb	48	44	130	143	225	282	331	418	424	514	440	521	400	422	786	709	24.9 C
C	38.2	37.9	36.4	37.0	32.3	30.5	35.7	31.7	43.1	29.1	30.4	28.6	25.9	26.0	22.4	26.7	127 HCO3
HCO3	194	193	185	188	164	155	181	161	219	148	155	145	132	132	114	136	247 Nitrate
Nitrate	214	239	238	238	232	236	231	238	245	252	249	263	275	266	225	383	9.24 pH
pH	9.11	9.03	9.21	9.18	9.37	9.47	9.42	9.67	9.53	9.71	9.64	9.98	9.76	9.82	9.17	9.22	0.4673 glass in
glass in	0.4694	0.467	0.4652	0.4588	0.4654	0.4597	0.4751	0.4569	0.4664	0.5087	0.4686	0.4538	0.4694	0.4673	0.4778	0.462	0.4656 glass out
glass out	0.4699	0.4669	0.4647	0.4584	0.4652	0.4594	0.4748	0.4561	0.466	0.5082	0.4639	0.453	0.4691	0.4671	0.4815	0.4611	-0.0007 wt. chg.
wt. chg.	0.0005	-0.0001	-0.0005	-0.0004	-0.0002	-0.0003	-0.0003	-0.0008	-0.0004	-0.0005	-0.0047	-0.0008	-0.0003	-0.0002	0.0037	-0.0009	20.6966 soln. in
soln. in	20.7311	20.7107	20.9482	20.8145	20.7943	20.8514	20.782	20.8524	21.0385	21.0215	21.0143	21.3157	21.0239	20.8828	20.8589	20.7702	20.6966 soln. in
soln. out	20.6665	20.6526	20.8403	20.714	20.6066	20.5957	20.5081	20.266	20.5235	20.2385	20.4349	19.1709	19.914	19.4286	18.8108	19.1868	19.0986 soln. out
soln loss	0.0646	0.0581	0.1079	0.1005	0.1877	0.2557	0.2739	0.5864	0.515	0.783	0.5794	2.1448	1.1099	1.4542	2.0481	1.5834	1.598 soln loss

SOLUTION CONC., PPM

CAST SAMPLES, SILICATE WATER

	CASi1a	CASi1b	CASi3a	CASi3b	CASi7a	CASi7b	CASi14a	CASi14b	CASi28a	CASi28b	CASi28c	CASi56a	CASi56b	CASi56c	CASi91a	CASi91b	CASi91c
Al-T	<.05	<.05	0.17	0.21	0.39	0.36	0.72	0.70	1.15	0.97	0.95	1.03	1.09	1.35	1.25	1.46	1.52 Al-T
Al-A	0.03	0.01	0.12	0.12	0.38	0.37	0.48	0.43	1.08	0.89	0.95	0.86	0.92	1.13			Al-A
B-T	<.07	<.07	0.15	0.16	0.33	0.29	0.61	0.55	0.99	0.85	0.76	1.11	1.10	1.32	1.35	1.46	1.51 B-T
B-A	0.04	0.05	0.17	0.16	0.32	0.32	0.54	0.49	1.02	0.85	1.04	0.98	1.02	1.22			B-A
Ca-T	0.04	0.04	0.06	0.05	0.08	0.28	0.10	0.08	0.13	0.09	0.27	0.14	0.08	0.15	0.35	0.22	0.19 Ca-T
Ca-A	<.04	<.04	0.05	0.02	<.02	<.04	0.06	<.004	0.06	0.06	0.04	0.16	0.09	0.13			Ca-A
Fe-T	0.04	0.04	0.06	0.07	0.20	0.17	0.62	0.61	1.68	1.39	1.22	1.72	1.70	2.25	2.07	2.43	2.21 Fe-T
Fe-A	0.04	0.04	0.06	0.07	0.19	0.19	0.57	0.58	1.87	1.55	1.05	1.66	1.69	2.22			Fe-A
K-A	<.3	<.3	<.3	<.3	<.3	<.3	<.3	<.3	<.3	<.3	<.3	<.3	<.3	<.3			K-A
Li-AA	0.07	0.07	0.17	0.19	0.37	0.29			1.23	1.04	0.95	1.26	1.32	1.57	1.50	1.65	1.70 Li-AA
Li-A	0.05	0.066	0.20	0.13	0.35	0.38	0.70	0.64	1.20	1.02	1.25	0.93	0.99	1.17			Li-A
Mg-A	<.5	<.5	<.5	<.5	0.43	0.44	0.54	0.53	0.53	0.52	0.50	<.5	<.5	<.5			Mg-A
Mg-T	0.02	0.02	0.04	0.03	0.06	0.07	0.10	0.09	0.15	0.14	0.14	0.18	0.18	0.20	0.22	0.23	0.21 Mg-T
Mn-A	<.08	<.08	<.08	<.08	<.08	<.08	0.08	0.08	0.26	0.22	0.14	0.24	0.23	0.30	298	295	Mn-A
Mn-C, ppb					112	32	93	93	234	238	246	231	191	258			92 Mn-C, ppb
Mo-A	<.1	<.1	<.1	<.1	<.1	<.1	<.1	<.1	<.1	<.1	<.1	<.1	<.1	<.1			Mo-A
Na-A	178	194	190	190	183	190	186	184	197	195	190	193	198	199	190	211	Na-A
Na-AA									152	164	165	226	294	262			233 Na-AA
Ni-A	<.1	<.1	<.1	<.1	<.1	<.1	0.11	0.11	<.1	<.1	<.1	<.1	<.1	<.1	40	41	Ni-A
Ni-C, ppb					43	15	26	28	30	49	70	32	27	32			50 Ni-C, ppb
Si-A	60.9	65.1	64.8	64.2	62.0	63.1	63.2	62.7	63.8	62.1	61.5	70.2	72.4	75.1			Si-A
Si-T	60.5	61.9	59.3	60.0	63.0	59.8	65.5	65.5	74.4	73.3	71.1	72.3	72.2	76.3	77.7	79.5	79.5 Si-T
Si-AA	54.3	56.9	53.4	53.6	55.9	51.7			57.9	56.3	54.4	51.1	49.3	59.5	55	56	56 Si-AA
Ce, ppb	0.12	0.10	0.12	0.12	<.04	<.04	<.07	<.07	2.0	2.0	2.4	1.1	1.4	2.5	<.2	<.2	<.2 Ce, ppb
Sr, ppb	2.7	2.9	4.6	4.3	11	4.9	10	11	19	17	19	23	21	23	27	26	26 Sr, ppb
U, ppb	22	21	67	71	140	151	256	246	450	382	350	340	352	430	564	616	636 U, ppb
C	37.0	37.5	38.7	35.3	32.5	31.4	30.2	29.7	26.5	29.6	29.1	23.8	24.5	25.3	25.2	27.2	25.9 C
HCO3	188	191	197	179	165	160	154	151	135	150	148	121	125	129	128	138	137 HCO3
Nitrate	235	245	234	240	234	237	233	233	258	254	248	263	260	272	327	375	262 Nitrate
pH	9.09	9.11	9.12	9.27	9.30	9.36	9.49	9.47	9.90	9.73	9.62	9.76	9.77	9.92	9.23	9.26	9.18 pH
glass in	0.3206	0.3071	0.3101	0.3366	0.3164	0.3329	0.3133	0.3055	0.3319	0.3330	0.2987	0.3011	0.3087	0.3141	0.3184	0.3375	0.3045 glass in
glass out	0.3206	0.3074	0.3100	0.3361	0.3161	0.3446	0.3128	0.3051	0.3311	0.3327	0.2982	0.3003	0.3318	0.3163	0.3224	0.3511	0.3120 glass out
wt. chg.	0.0000	0.0003	-0.0001	-0.0005	-0.0003	0.0117	-0.0005	-0.0004	-0.0008	-0.0003	-0.0005	-0.0008	0.0231	0.0022	0.0040	0.0136	0.0075 wt. chg.
soln. in	20.7852	20.7208	20.8872	20.7464	20.7884	20.8614	20.7687	20.7844	21.0823	20.7097	20.9432	20.2988	20.7562	21.0578	20.5183	20.5475	20.1371 soln. in
soln. out	20.7038	20.6541	20.7959	20.6024	20.6224	20.6650	20.4157	20.4400	19.8083	19.7547	20.3128	19.4315	19.1317	19.9392	18.9213	18.9855	18.4846 soln. out
soln loss	0.0814	0.0667	0.0913	0.1440	0.1660	0.1964	0.3530	0.3444	1.2740	0.9550	0.6304	0.8673	1.6245	1.1186	1.5970	1.5610	1.6525 soln loss

CORED AND SLICED SAMPLES, DIW

SOLUTION CONC., PPM

	CSW1a	CSW1b	CSW3a	CSW3b	CSW7a	CSW7b	CSW14a	CSW14b	CSW28a	CSW28B	CSW56a	CSW56B	CSW91a	CSW91B
Al-T	0.15	0.64	1.44	1.37	2.09	1.75	2.59	2.77	2.90	2.98	3.55	3.25	3.49	3.45 Al-T
Al-A	<.008	0.09	1.29	1.29		1.94	2.34	2.59		2.86	3.02	2.76		Al-A
B-T	0.29	0.59	1.41	1.42	2.32	2.12	3.01	3.23	3.52	3.62	4.51	4.28	4.96	5.04 B-T
B-A	0.30	0.59	1.33	1.33		2.26	2.89	3.20		3.56	4.18	3.97		B-A
Ca-T	0.13	0.21	0.22	0.26	0.34	0.17	0.18	0.20	0.14	0.13	0.16	0.10	0.07	0.06 Ca-T
Ca-A	0.08	0.17	0.33	0.34		0.06	<.004	<.004		0.09	0.13	0.09		Ca-A
Fe-T	0.02	0.1	0.11	0.09	0.25	0.15	0.35	0.42	0.51	0.54	1.32	0.73	0.91	0.73 Fe-T
Fe-A	<.01	0.09	0.12	0.10		0.21	0.36	0.43		0.61	1.39	0.82		Fe-A
K-A	<.3	<.3	<.3	<.3		<.3	<.3	<.3		<.3	<.3	<.3		K-A
Li-AA	0.33	0.66	1.48	1.45	2.36	2.00			3.78	3.96	5.00	4.74	4.88	5.02 Li-AA
Li-A	0.31	0.61	1.45	1.45		2.48	3.54	3.92		4.07	4.06	3.82		Li-A
Mg-A	<.5	0.5	0.54	0.54		0.44	<.5	0.52		<.5	<.5	<.5		Mg-A
Mg-T	0.07	0.12	0.16	0.17	0.06	0.04	0.04	0.05	0.08	0.05	0.23	0.10	0.12	0.09 Mg-T
Mn-A	<.08	<.08	<.08	<.08	53	<.08	<.08	<.08	6	0.08	0.16	0.12	174	Mn-A
Mn-C, ppb						32	66	69	6	6	159	111		137 Mn-C, ppb
Mo-A	<.1	<.1	<.1	<.1		<.1	<.1	<.1		<.1	<.1	<.1		Mo-A
Na-A	4.5	3.7	11.5	7.3		10.5	12.7	13.9	20.7	12.7	17.6	16.6		Na-A
Na-AA										24.0	146	150	20	20 Na-AA
Ni-A	<.1	<.1	<.1	<.1		<.1	<.1	<.1		<.1	<.1	<.1		Ni-A
Ni-C, ppb					10	7	13	19	11	17	20	18	23	23 Ni-C, ppb
Si-A	4.1	6.9	17.6	16.1		24.2	31.0	33.8		31.7	46.3	43.4		Si-A
Si-T	3.3	6.7	14.0	13.7	22.7	19.5	28.1	29.8	35.5	36.7	46.6	43.6	47.4	47.8 Si-T
Si-AA	2.6	5.6	13.8	13.7	22.5	19.2			27	24	31.7	30.9	32	34 Si-AA
Ce, ppb	0.19	0.25	0.40	0.23	0.15	0.71	1.3	1.6	4.1	5.3	5.7	4.8	2.4	2.1 Ce, ppb
Sr, ppb	11	16	23	25	27	21	20	18	17	18	22	17	20	17 Sr, ppb
U, ppb	15	29	105	96	234	227	328	359	450	472	549	500	790	835 U, ppb
C	<.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5	3.6	9.0	2.8	8.2	2.9	3.7 C
HC03									18.3	45.8	14.2	41.7	14.7	18.8 HC03
pH	7.25	8.86	9.63	9.61	9.55	9.69	9.65	9.69	9.47	9.60	9.51	9.47	8.67	8.60 pH
F											2.0	1.8		F
glass in	0.4653	0.4654	0.4978	0.4595	0.4840	0.4694	0.4707	0.4737	0.4447	0.4689	0.4665	0.4673	0.4681	0.4634 glass in
glass out	0.4653	0.4651	0.4969	0.4587	0.4827	0.4682	0.4686	0.4718	0.4425	0.4664	0.4650	0.4714	0.4722	0.4680 glass out
wt. chg.	0	-0.0003	-0.0009	-0.0008	-0.0013	-0.0012	-0.0021	-0.0019	-0.0022	-0.0025	-0.0015	0.0041	0.0041	0.0046 wt. chg.
soln. in	20.8970	20.5520	20.9162	20.9557	20.7053	20.7154	20.7079	20.8388	20.7700	19.6811	20.6540	20.7717	20.6685	20.6343 soln. in
soln. out	20.8414	20.4877	20.8002	20.8526	18.4555	20.5461	20.1851	20.4276	20.1384	20.1967	18.7201	19.2542	18.1038	18.3069 soln. out
soln loss	0.0556	0.0643	0.1160	0.1031	2.2498	0.1693	0.5228	0.4112	0.6316	-0.5156	1.9339	1.5175	2.5647	2.3274 soln loss

SOLUTION CONC., PPM

CAST SAMPLES, DIW

CAW1a	CAW1b	CAW3a	CAW3b	CAW7a	CAW7b	CAW14a	CAW14b	CAW28a	CAW28b	CAW56a	CAW56b	CAW91a	CAW91b
Al-T	<.05	<.05	<.05	0.91	0.62	1.39	1.39	1.80	1.78	2.20	2.33	2.36	2.29 Al-T
Al-A	<.008	<.008	<.008	0.69	0.42	1.16	1.16	1.61	1.61	1.97	1.78	3.04	Al-A
B-T	<.07	0.81	0.07	0.85	0.67	1.27	1.34	1.99	2.00	2.58	2.87	2.91 B-T	B-A
B-A	0.023	0.09	0.08	0.91	0.74	1.34	0.26	1.95	1.91	2.64	2.40	0.11	0.10 Ca-T
Ca-T	0.01	0.04	0.04	0.24	0.15	0.25	0.05	0.22	0.21	0.07	0.06	0.32 Fe-T	Ca-A
Ca-A	<.004	<.004	<.004	0.09	0.11	<.004	0.05	0.04	0.04	0.07	0.06	0.32 Fe-T	Fe-A
Fe-T	<.02	<.02	<.02	0.03	0.03	0.05	0.06	0.07	0.11	0.20	0.25	0.20	K-A
Fe-A	<.01	<.01	<.01	0.05	0.05	0.06	0.06	0.08	0.13	0.23	0.20	3.2 Li-AA	Li-A
K-A	<.3	<.3	<.3	<.3	<.3	<.3	<.3	<.3	<.3	<.3	<.3	0.07 Mg-T	Mg-A
Li-AA	0.03	0.03	0.09	0.94	0.67	1.65	18	2.28	2.23	2.95	3.33	0.08	121 Mn-C, ppb
Li-A	0.035	0.031	0.09	1.0	0.77	1.65	0.16	2.24	2.20	2.58	2.34	0.08	Mo-A
Mg-A	<.5	<.5	<.5	0.54	0.54	0.58	0.18	<.5	<.5	<.5	<.5	0.08	Na-A
Mg-T	0.01	0.01	0.02	0.17	0.05	0.18	0.16	0.05	0.06	0.04	0.04	0.08	12 Na-AA
Mn-A	<.08	<.08	<.08	<.08	<.08	<.08	0.18	<.08	<.08	<.08	<.08	0.08	Ni-A
Mn-C, ppb				22	25	16	18	48	49	40	48	0.08	28 Ni-C, ppb
Mo-A	<.1	<.1	<.1	<.1	<.1	<.1	7	<.1	<.1	<.1	<.1	0.08	Si-A
Na-A	2.2	1.4	1.9	4.8	3.9	7.0	7	7.5	7.2	11.1	10.2	31.3	30.0 Si-T
Na-AA								19	14	396	200	31.3	20 Si-AA
Ni-A	<.1	<.1	<.1	<.1	<.1	<.1	7	<.1	<.1	<.1	<.1	31.3	4.5 Ce, ppb
Ni-C, ppb				16	16	13	7	<.1	<.1	9	10	31.3	19 Sr, ppb
Si-A	0.60	0.32	1.6	10.2	8.2	15.5	14.2	17.5	17.0	30.2	27.3	31.3	362 U, ppb
Si-T	0.26	0.27	0.95	9.4	7.5	14.1	14.2	21.8	21.2	27.8	30.9	31.3	0.9 C
Si-AA	<.2	<.2	<.2	8.5	6.5	14.1	14.2	16	15.6	19.1	21.7	31.3	5 HC03
Ce, ppb	<.04	0.05	<.06	0.21	0.38	0.51	0.67	2.4	2.3	4.9	5.6	31.3	8.98 pH
Sr, ppb	1.2	1.2	3.7	20	19	26	24	20	19	18	18	31.3	F
U, ppb	0.4	0.4	2.3	80	36	116	113	156	155	184	220	31.3	0.2359 glass in
C	<.5	<.5	<.5	2	<.5	<.5	<.5	9.0	8.8	9.1	6.2	31.3	0.2495 glass out
HC03								46	45	46	32	31.3	0.0136 wt. chg.
pH	6.41	5.24	7.23	9.35	9.26	9.41	9.34	9.04	9.17	9.36	8.92	31.3	20.6013 soln. in
F										1.6	2.4	31.3	18.4144 soln. out
glass in	0.3087	0.3095	0.3157	0.3263	0.3215	0.3171	0.3054	0.3273	0.3111	0.3037	0.3252	0.3371	2.1869 soln loss
glass out	0.3089	0.3102	0.3155	0.3256	0.3209	0.3159	0.3043	0.3258	0.3096	0.3022	0.3235	0.3575	
wt. chg.	0.0002	0.0007	-0.0002	-0.0002	-0.0006	-0.0012	-0.0011	-0.0015	-0.0015	-0.0015	-0.0017	0.0204	
soln. in	20.9338	20.8677	20.8967	20.9229	20.6635	20.8928	21.9197	20.7566	21.7011	20.7753	20.6117	20.6254	
soln. out	20.8641	20.7833	20.8044	20.8170	20.3068	20.5815	21.4655	20.1219	19.9216	17.7230	17.4990	18.9976	
soln loss	0.0697	0.0844	0.0923	0.1059	0.1973	0.3113	0.4542	0.6347	1.7795	3.0523	3.1127	1.6278	

SOLUTION CONC., PPM

BLANKS

BSi1	BSi13	BSi3	BSi7	BSi14	BSi28	BSi56	BSi91	BW91
Al-T	<.05	<.05	<.05	<.05	<.05	<.05	0.47	0.40 Al-T
Al-A	<.008	<.008	0.06	<.008	0.05	<.07	<.07	Al-A
B-T	<.07	<.07	<.07	<.07	<.07	<.07	<.07	B-T
B-A	<.005	<.005	<.005	0.006	<.005	<.01	0.01	B-A
Ca-T	0.02	0.02	0.01	<.01	0.01	<.01	<.01	Ca-T
Ca-A	<.004	0.05	<.004	<.004	0.02	<.02	<.02	Ca-A
Fe-T	<.02	<.02	<.02	<.02	<.02	<.02	<.02	Fe-T
Fe-A	<.01	<.01	<.01	<.01	<.01	<.01	<.01	Fe-A
K-A	<.03	<.03	<.03	<.03	<.03	<.03	<.03	K-A
Li-AA	<.05	<.05	<.05	0.02	0.008	ND	ND	Li-AA
Li-A	<.005	<.005	0.01	0.02	0.008	ND	ND	Li-A
Mg-A	<.05	<.05	0.42	<.05	<.05	<.01	<.01	Mg-A
Mg-T	0.01	<.01	0.01	<.01	<.01	<.01	<.01	Mg-T
Mn-A	<.08	<.08	<.08	<.08	<.08	1.9	<.7	Mn-A
Mn-C, ppb			12	4	6	2.1	<.7	Mn-C, ppb
Mo-A	<.1	<.1	<.1	<.1	<.1	ND	ND	Mo-A
Na-A	184	173	186	186	188	278	252	Na-A
Na-A	<.1	<.1	<.1	<.1	<.1	ND	ND	Na-A
Ni-A	<.1	<.1	<.1	<.1	<.1	ND	ND	Ni-A
Ni-C, ppb			16	6	8	ND	ND	Ni-C, ppb
Si-A	61.6	57.6	58.9	58.8	53.4	63.9	64.9	Si-A
Si-T	59.2	59.2	59.4	60.1	62.1	43.7	44	Si-T
Si-AA	55.8	51.7	52.8	<.07	48.2	ND	ND	Si-AA
Ce, ppb	<.04	0.11	<.04	<.04	<.1	<.1	<.2	Ce, ppb
Sr, ppb	0.95	0.89	<.2	<.3	<.2	0.6	0.6	Sr, ppb
U, ppb	<.5	0.14	<.5	<.5	<.5	<.5	<.5	U, ppb
C	37.1	37.6	32.6	33.5	42.4	25.2	25.1	C
HC03	189	191	166	170	216	128	128	HC03
Nitrate	239	243	214	233	249	266	189	Nitrate
pH	9.09	5.16	5.05	9.39	8.99	9.81	9.07	pH
F						1.9	1.9	F
soln. in	20.8223	20.9105	20.6305	20.9764	20.9764	20.6305	20.9594	soln. in
soln. out	20.7345	20.8438	20.5332	20.8609	20.8609	20.5332	20.8438	soln. out
soln loss	0.0878	0.0667	0.0973	0.1155	0.1155	0.0667	0.0878	soln loss

- END -

DATE FILMED

01 / 29 / 91