

**Preliminary corrosion studies of glass
ceramic code 9617 and a sealing frit
for nuclear waste canisters**

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PRELIMINARY CORROSION STUDIES OF GLASS-
CERAMIC CODE 9617 and a SEALING FRIT
FOR NUCLEAR WASTE CANNISTERS

J. D. Sundquist
Corning Glass Works
Corning, New York

March 14, 1978

for

Karnbranselsakerhet Order No. 166
Stockholm, Sweden

PRELIMINARY CORROSION STUDIES OF GLASS-
CERAMIC CODE 9617 AND A SEALING FRIT
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J. D. Sundquist

ABSTRACT

At the request of ASEA-ATOM, Sweden, a study was initiated to evaluate glass-ceramic Code 9617 and a sealing frit (glass Code 186AYU) to determine the suitability of these materials for use in the construction of a cannister to contain and permanently isolate spent nuclear fuel rods. The study was aborted after one quarter of the work was completed. Nevertheless results obtained define test conditions for future work and provide preliminary estimates of corrosion rates.

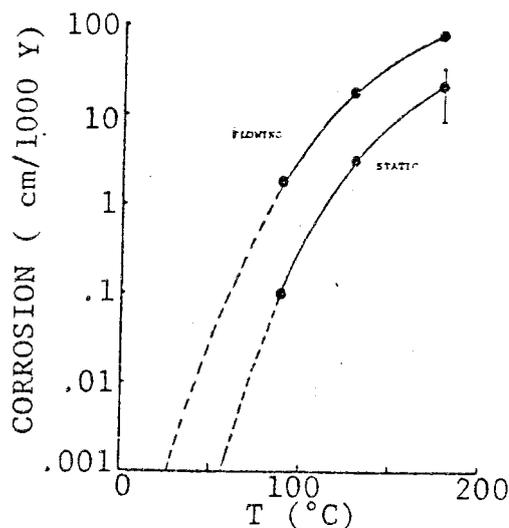
The granite repository storage conditions included ground water in contact with bentonite clay plus sand with temperature cooling from 100 to 40 in 500 years. The water exchange rate is 1 ml per cm² of cannister surface per year. Tests were conducted at 60, 90, 130, and 180°C. Most of this preliminary work was done with distilled water with a few tests in water containing bentonite and sand or salt.

It is recommended that future tests be conducted under conditions which embrace the actual storage temperatures, thus allowing prediction of long term rates from an extrapolation of time rather than from the accelerated effect of increased temperature which our results indicate may alter the reaction mechanism. Other test conditions should also simulate real conditions as nearly as possible. Of particular importance is a consideration of the changing chemistry of the test solutions when "static" conditions are used; i.e., when the test solution remains in contact with the test specimen for long periods of time.

Preliminary data suggest that the glass-ceramic would lose about 0.1% of thickness in the first 1000 years of storage. The figure shown below presents a comparison of estimated corrosion rates as a function of temperature for a flowing and a static water system.

There is little evidence to suggest that the corrosion rate in ground water/bentonite/sand mix or in salt water would differ by more than an order of magnitude.

It appears that the spodumene fraction of the glass-ceramic is very corrosion resistant, even at 180°C. This suggests that modifications in bulk composition or processing could dramatically decrease overall corrosion rates.



1. Introduction

At the request of ASEA-ATOM, Sweden, a study was initiated in July, 1977, to evaluate glass-ceramic Code 9617 as a cannister material for the containment of spent nuclear fuel rods. Corning was to have one year to determine whether it could fabricate the large (1½ by 8 foot) cylinders, seal them and assure that they would survive the environment in which they would be stored.

There were three areas of study:

1. feasibility and cost of fabrication
2. physical durability of the cannister in the proposed environment, and
3. chemical durability of the cannister in the proposed environment.

The project was discontinued October 31, 1977.

This report details the chemical durability study (item 3) up to that time.

The objectives were to:

1. evaluate methods of testing, and
2. use the best methods to predict corrosion over a 1,000 year span.

We have completed objective 1 and have some preliminary data concerning objective 2.

In this report, I:

1. discuss the proposed site conditions,
2. describe the experimental approach
3. provide an evaluation of the various methods and techniques, and
4. estimate corrosion rates from the preliminary data and discuss possible mechanisms of attack.

2. Problem Definition-Repository Conditions

The proposed repository conditions are summarized below:

a mixture of 9 parts sand and 1 part bentonite will be packed around the spent fuel rods contained in the cannister,

the cannisters will be stored in shafts 500 meters deep in granite and surrounded with the 9 sand and 1 bentonite mix,

the temperature is initially 100°C, cooling to 60°C after 100 years, 40°C after 500 years and 25°C after 3000 years, (see zeolite, Figure 2),

the ground water flow through the granite will be 1 liter/meter²/year,

the ground water composition will be as shown in Table 1.

Figure 1, represents the proposed storage configuration as we understand it:

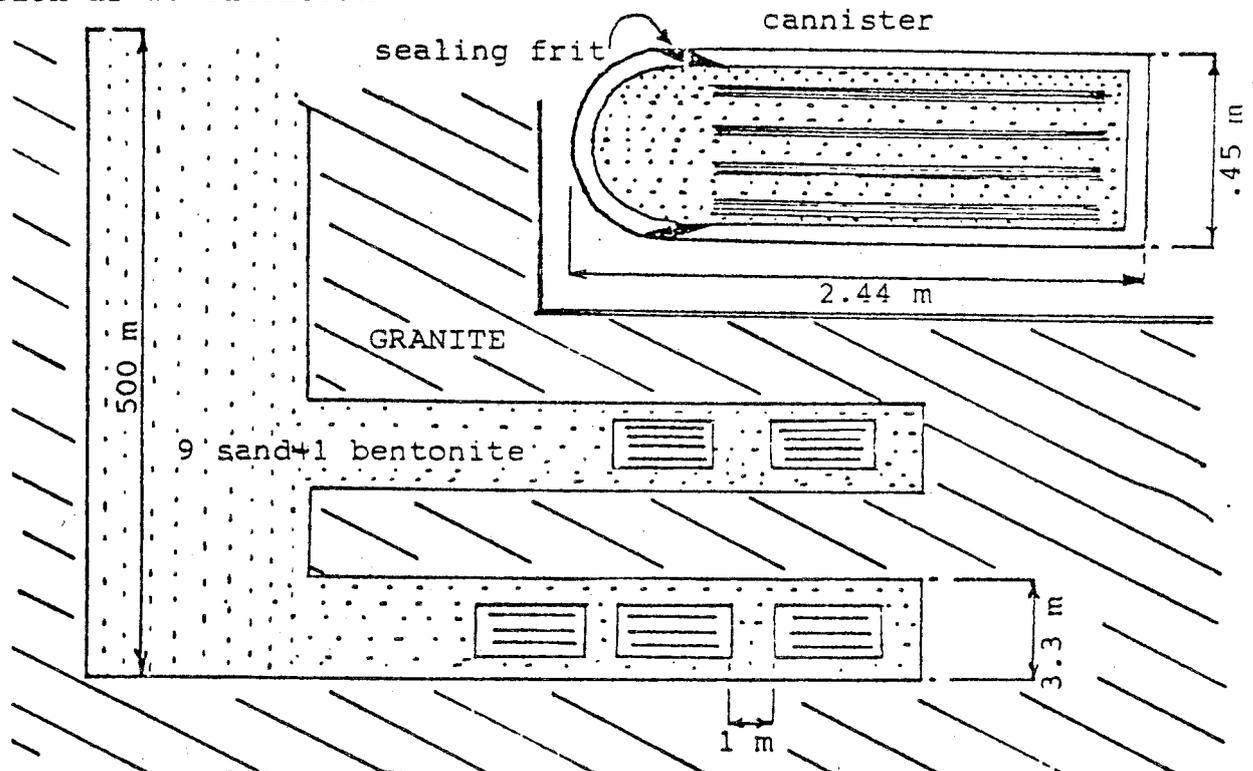


Figure 1. Proposed Repository Configuration.

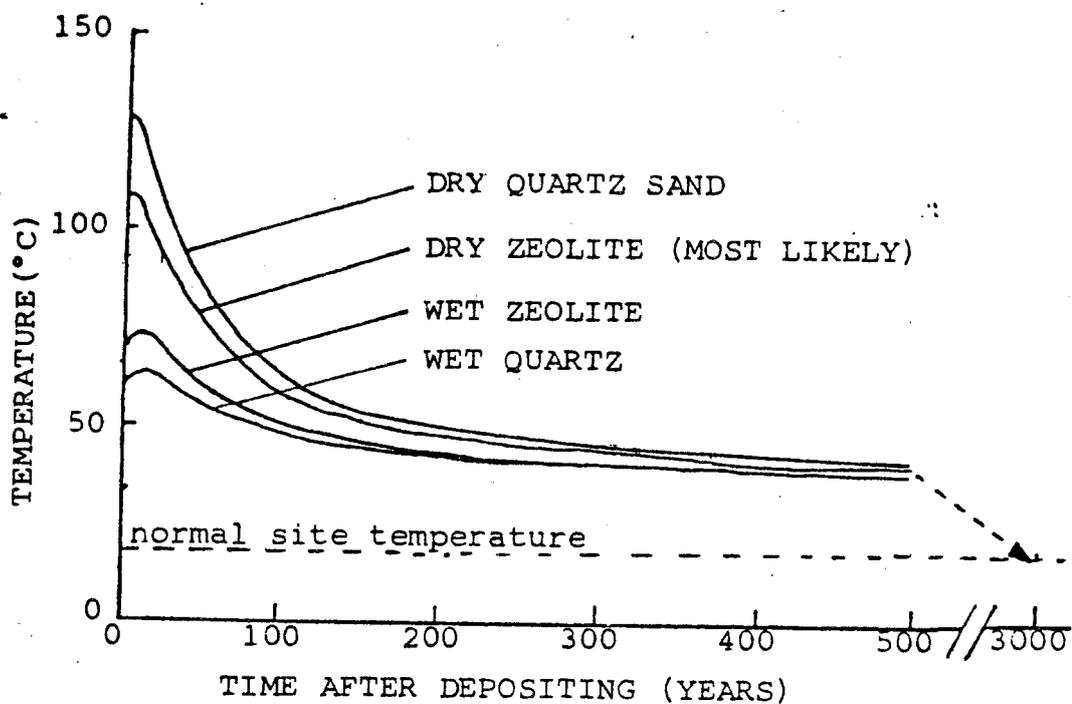


Figure 2. Temperature decay curve in granite repository. (Spent fuel rods are stored 10 years before placing in cannisters, then stored 40 years before being placed in the repository.) (Reference 1)

ASEA-ATOM

Addendum to Inquiry of February 21, 1977

Water chemistry for materials selection:

pH	6.5 - 8.6
Ca	5 mg/l
Mg	1 mg/l
Na	270 mg/l
K	1 mg/l
Cl	280 mg/l
SO ₄	35 mg/l
HCO ₃	240 mg/l
NO ₃	1.7 mg/l
PO ₄	0.3 mg/l
F	4.3 mg/l
SiO ₂	4 mg/l
Fe ^{II}	3 mg/l
Mn ^{II}	1.3 mg/l

Table 1. ASEA-ATOM Definition of Ground Water
at Repository Site.

For one cannister I have made some calculations from the previous information to aid in our experimental design, method and evaluation:

LATERAL SHAFT: Void Volume	36.8 m ³
Surface Area	53.6 m ²
VOLUME OCCUPIED BY CANNISTER	0.39m ³
OUTSIDE SURFACE AREA=	4.86m ²

(SHAFT VOLUME) - (CANNISTER VOLUME) = 36.4 m³ = 26.4 x 10³

(9) sand + (1) bentonite packing density = 1.63 gms/c

sand density = 2.35 gms/c

bentonite density = 2.0 gms/c

sand/bentonite mix = 70% solid and 30% void

FINAL VOID SPACE = (30%) x (36.4 x 10³)L 10.9 x 10³ L

WATER LEAK RATE IN = 56.6 L/year



TIME TO FILL VOID = 200 years



SOLUTION VOLUME TO CANNISTER SURFACE AREA RATIO = 220 cc/cm²



RATE OF CHANGE OF SOLUTION = 1.1 cc/year/cm² of canniste

3. Equipment and Test Conditions

The time interval over which we seek to predict corrosion is extremely long. To shorten laboratory testing time we frequently accelerate tests by increasing temperature and/or solution corrosivity and then extrapolate back to the real-life condition. This is relatively easy when predicting for a few years. To apply this technique to a 1,000 year interval requires further consideration. The changing test solution chemistry becomes critical as does the changing on-site chemistry. Also, the increased temperature required may introduce new variables. In this light our present and proposed test procedure and methods of evaluation were studied in detail.

3.1 Equipment

The glass-ceramic candidate (CGW Code 9617) and frit (experimental) were tested at temperatures of 60, 90, 130, and 180°C. We purchased high temperature ovens and constant temperature baths from the Blue M Company, Long Island, New York and reaction vessels (high temperature bombs) from the PARR Instrument Company, Chicago, Illinois. Chemically resistant glass test tubes were obtained from our company. Figures 3 and 4 show some of the equipment.

3.2 Test Conditions

The glass-ceramic and frit durabilities were studied in distilled, deionized water at the four temperatures indicated in 3.1. The variables evaluated included time, temperature and the ratio of test solution volume to test sample surface area. The latter will hereafter be referred to as "the ratio" or "R".

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We also obtained some data on the effect of a 9 sand and 1 bentonite mixture and on the effect of an aqueous solution of sodium chloride on durability.

Our evaluation methods included weight loss, atomic absorption analysis of the test solution, and 5 methods of sample surface analysis. These methods were scanning electron microscopy, transmission electron microscopy, transmission x-ray energy dispersion analysis; x-ray diffraction and H⁺ profiling by ¹⁵N bombardment.

4. Data

A considerable amount of data was obtained. Because the project was terminated many experiments were not concluded. Other experiments, though completed, have not been validated with replicates.

In Sections 5 and 6 I discuss the results which we feel are significant.

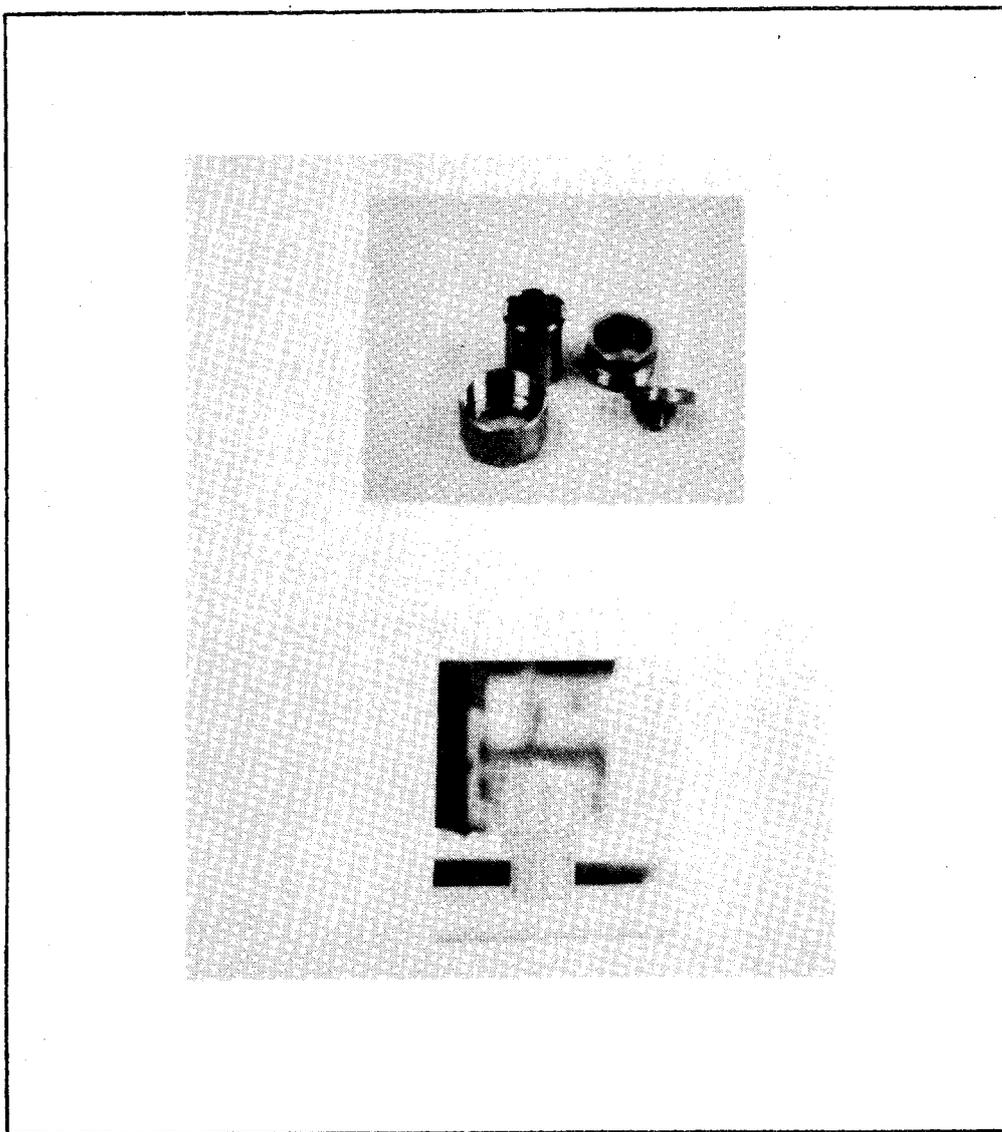
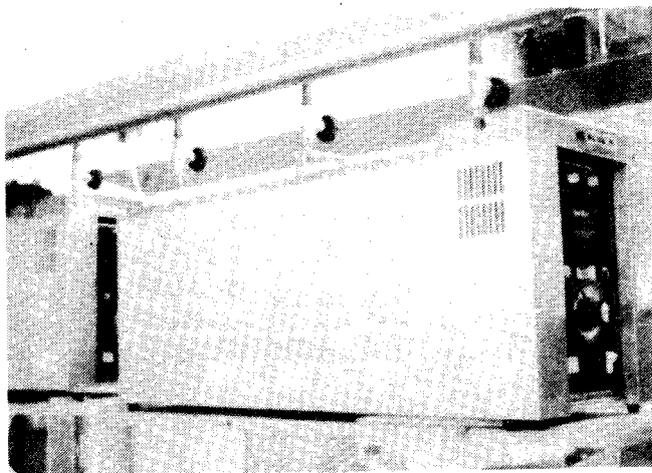
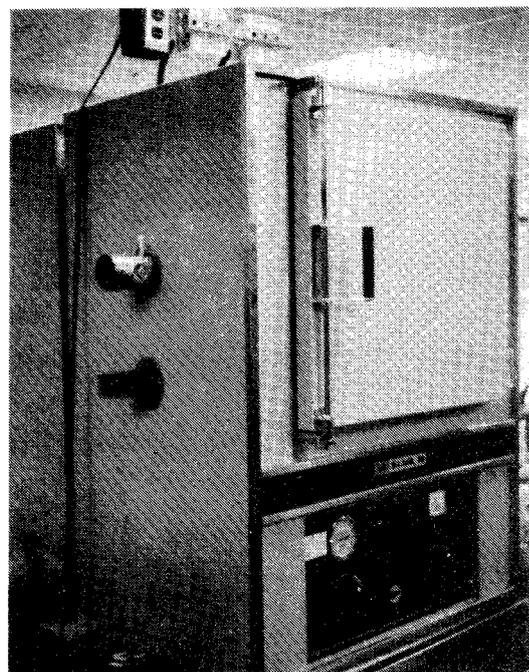


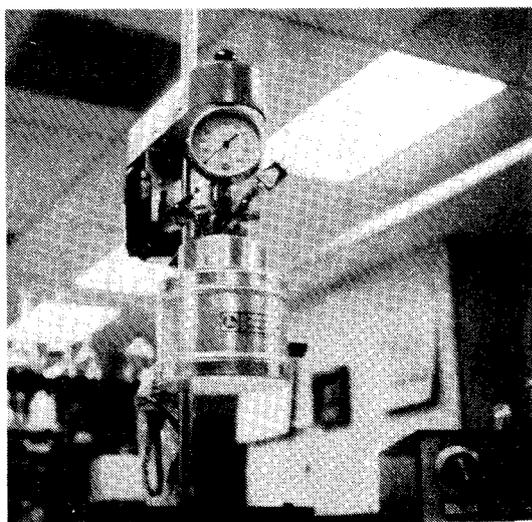
Figure 3. 20 ml High-Temperature Nickel Coated
Reaction Vessels (Bombs)



(A)



(B)



(C)

Figure 4: Photographs of some of the equipment. (A)-constant temperature bath. (B)-oven. (C)- mini-reactor.

5. Study of Test Methods

This section includes the evaluation of the test equipment and an evaluation of the various analytical tools that were employed throughout the study.

5.1 Equipment

High temperature reaction vessels ("bombs") of various composition and size were considered. Materials considered were tantalum, monel, stainless steel and nickel. Corrosion resistance, cost and delivery time led us to select nickel-plated 22 milliliter bombs.

Fifty milliliter Corning Code 7740 test tubes with screw tops were used for most of the low temperature work. They were pre-aged in hot hydrochloric acid. As the tests proceeded the decision was made to switch to plastic test tubes since the relatively large surface area of the tubes compared to the samples gave high blank values.

The ovens and baths functioned well.

The mini-reactor was not evaluated since it was received after the work was finished.

5.1.1 Corrosion of Stainless Steel Bombs. We rejected high quality stainless steel bombs as test containers because of their extremely poor resistance to attack by NaCl. The steel, #316, is molybdenum doped which improves its resistance to chloride attack.

In a sodium chloride solution at both 130 and 180°C for four days the steel bomb was deeply corroded and the test solutions discolored. Figure 5 shows a photograph of the tops of the two bombs after they had been "cleaned" by sand blasting compared with an unused bomb.

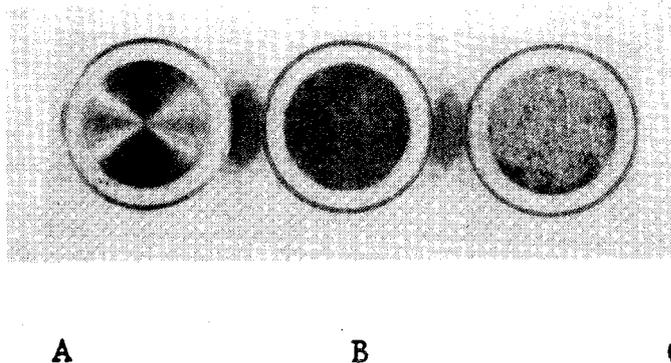


Figure 5. Photograph of High Quality Stainless Steel (316) Containers After Exposure to an Aqueous NaCl Solution for Short Periods of Time.
A = Blank, B = 130°C/4 days, C = 180°C/4 days.

Incidentally, Code 9617 glass-ceramic by comparison is hardly touched!

5.1.2 Corrosion of Code 7740 Glass Tubes. Chemically resistant CGW glass Code 7740 test tubes were used in the constant temperature baths at temperatures less than 100°C. Code 7740 glass corrosion rate in water is 0.15 cm/1000 year (Y). However, because of the relatively large surface area of the test tube compared to the test specimen (ranging from 100/1 to 50/1) the blank contribution by the tubes becomes significant. Because of this high blank it is recommended that plastic tubes be used for small samples.

5.2 Analytical Tools

Several analytical techniques were evaluated including weight loss, flame spectrometric analysis, microscopy and hydrogen ion profiling.

Weight loss has the advantages of ease and speed as well as the availability of much historical data on other materials that can be used as references. It has the disadvantage of requiring a major disruption of a test when interim data is required as the test proceeds. This disruption is not only inconvenient but will affect the results since it introduces a sample cooling and drying step before weighing.

A better method for monitoring tests as they progress is the flame spectrometric analysis of test solution aliquots. This can be done with less disturbance, especially if the solution volume is large (> 100 ml) and the test is not being run in high temperature bombs. Disadvantages include the possibility that all the products of corrosion might not be in solution, the decrease in solution volume or the change in solution chemistry if the removed volume is replaced with fresh water, the fact that the use of the bombs requires the cooling of the test solution before an aliquot can be removed and the fact that the bombs are of a small volume (22 ml). The effect of these drawbacks can be lessened with the use of the 300 ml mini-reactor which we purchased. This bomb permits the addition and removal of solution in situ.

Scanning and transmission electron microscopy are useful in visually assessing surface effects.

A technique for H⁺ profiling of the surface of materials to a depth of a few micrometers has also been used.² A linear accelerator produces ¹⁵N atoms which collide with hydrogen in the sample to yield gamma rays in proportion to the concentration of hydrogen present. Depth of analysis is changed by changing the energy of the ¹⁵N. This method was used to profile reacted glass-ceramic surfaces. The technique is also capable of

providing other ion profiles such as lithium which is present in the glass-ceramic composition. It is not yet clear whether this method can be used to predict corrosion rates.

5.3 Experimental Variables

Experimental variables studied include the ratio (R) of the test solution volume to test sample surface area (ml/cm²), pH, time, temperature, and the effects of the sand plus bentonite mix, and sodium chloride.

Ground water composition effects were not evaluated initially since we preferred to study the principal variables in a simple matrix - pure water. Once these were understood we planned to introduce ground water as an effect.

We used very small sample sizes (0.5 to 2 cm²) in order to fit them into the small bombs. This small sample size introduces two additional variables, neither of which we have evaluated. They are the error in determining the geometric area of small samples and the "edge effect". With large samples (> 5 cm²) this effect is negligible but becomes more important as one uses sample sizes in the regime in which we have. The corrosion rate at the sharp edges of a material is accelerated over that rate for a flat surface. The smaller the sample size, the greater the proportion of edge length to total surface area and the greater this effect. This relationship for various shapes of rectangles is plotted in Figure 6.

6. Preliminary Rate Prediction

This section presents our comments and conclusions regarding various aspects of the data. None of the conclusions and predictions have been supported with replicate experiments. The corrosion rates were calculated assuming linear attack (etch).

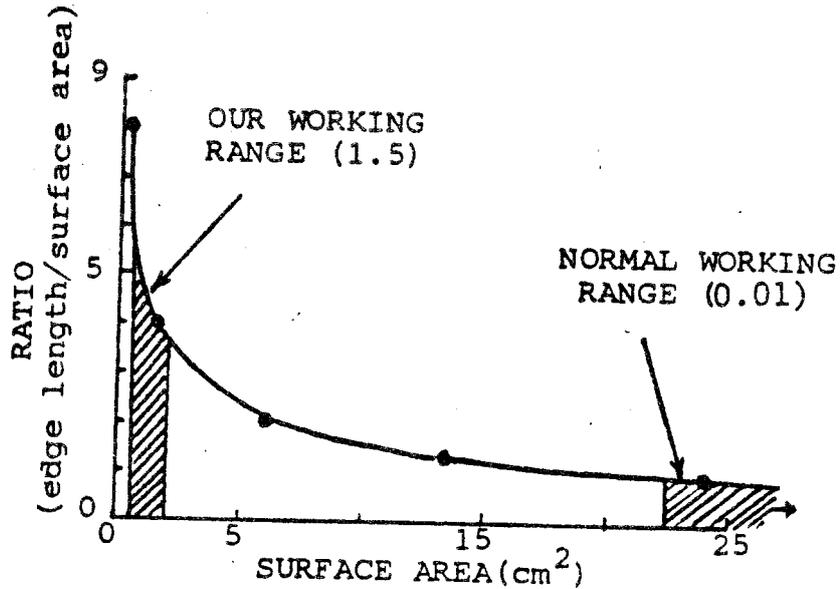


Figure 6. Increased Importance of Edge Effect with Lower Surface Area Samples. Rate of Change of Ratio in Parentheses

This is probably not the case, but it provides a high estimate.

6.1 Ratio Effect

The first variable studied in detail was the effect of test solution volume to sample surface area (R). This is an important consideration since R affects the rate of change of solution chemistry with time. An example of this effect is shown in Figure 7.

Here, corrosion rate in cm/1000 years is plotted versus time at 180°C for two values of R. Apparently, the lower ratio test solution becomes saturated with corrosion products at a faster rate than the higher one and reduces the rate of attack by the solution.

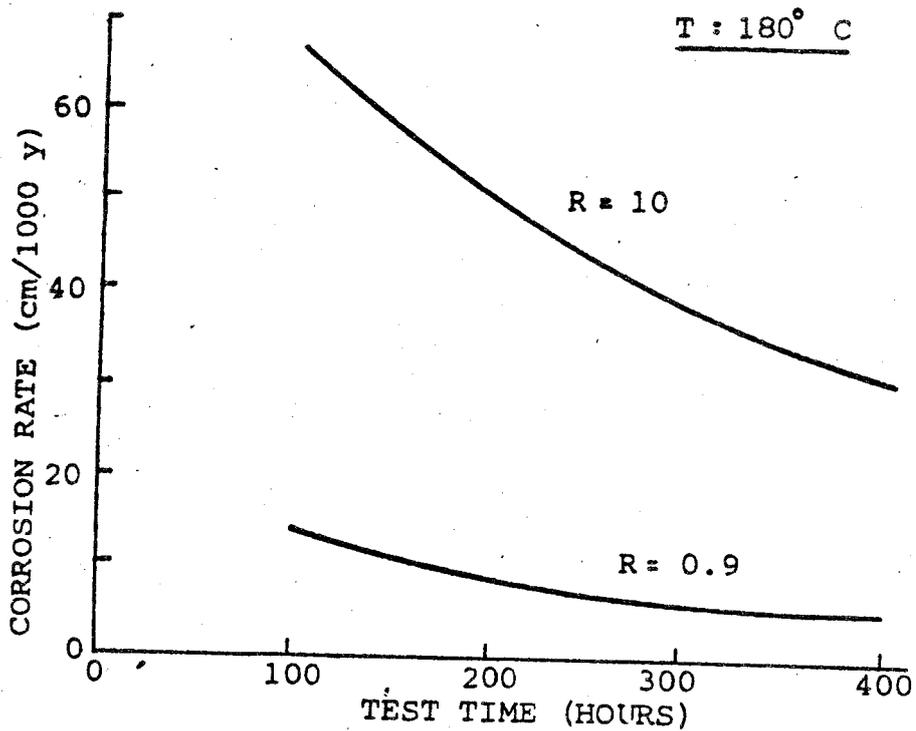


Figure 7. The Effect of the Ratio of Test Solution Volume to Sample Surface Area (ml/cm^2) on Corrosion Rate for the Code 9617 Glass-Ceramic

Since the proposed system is nearly static (a key assumption from the calculated rate of change of solution which was $\sim 1 \text{ ml}/\text{cm}^2$ of glass-ceramic/year) we need to determine the saturated solution corrosion rate to predict the long term corrosion. One can determine this equilibrium corrosion rate by extending the time of experimentation to the point where the corrosion rate becomes constant. To assure that this holds true for any ratio the experiment should be done for more than one starting ratio. Also, if the experimental time becomes prohibitive one can extrapolate a family of curves to a point where they coincide. This is shown graphically in Figure 8.

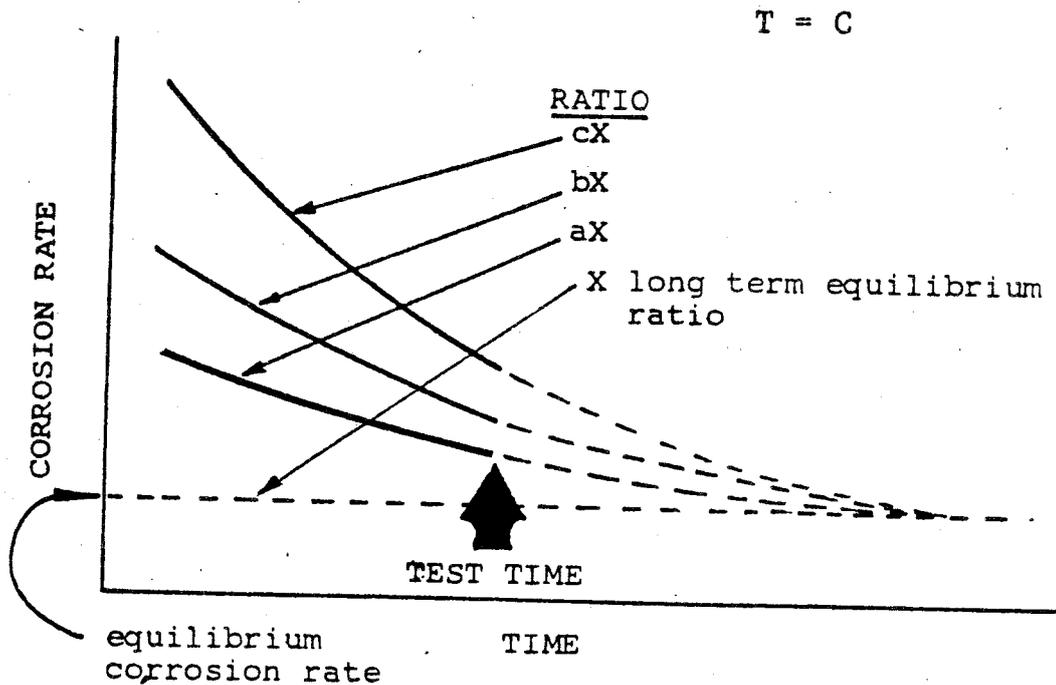


Figure 8. Predicting Equilibrium Long-Term Corrosion Rate and Ratio

This should then be done for several different temperatures so that one could extrapolate to the actual test site temperature range.

After having gathered some data in this manner, it has become apparent that the use of high temperature tests creates many problems. Control of the experiment is difficult and we have indications that there is a change in the corrosion mechanism at the higher temperatures that prevents a straight forward extrapolation to the lower temperatures. (This change is discussed later in this report.)

Rather than accelerating the corrosion of the materials and extrapolating down to actual conditions to obtain the long term corrosion rate, I feel that the best approach in the future

would be to take corrosion data under conditions very close to those predicted for the repository site. By using a low enough solution volume to surface area ratio, we would probably approach the equilibrium quite rapidly even at the lower temperatures of 60° and 90°C.

6.2 Corrosion Rate Estimates

Based on data so far obtained I have calculated the ceramic and frit water corrosion rate as a function of temperature and ratio:

Sample	R	Test		Corrosion Rat (cm/1000 Y)
		Time(days)	Temperature(°C)	
Glass-ceramic	20	4	180	78
	10	4	180	62
	5	4	180	41
	20	4	130	18
	10	4	130	10
	5	4	130	7
	20	4	90	1.8
	10	4	90	0.9
	5	4	90	0.5
Frit	20	4	180	68
	10	4	180	34
	5	4	180	18

Table 2. Estimated Corrosion Rate of the Code 9617 Glass-Ceramic and Experimental Sealing Frit.

NOTE: That as the ratio is lowered the corrosion rate drops.

Extrapolation of these data back to a zero ratio should give an estimate of the equilibrium (long term) corrosion rate under static conditions. In Figure 9 below I have done this for the data in Table 2. The glass-ceramic 180°C data are not linear. This may be a change in glass corrosion mechanism or an error in the data.

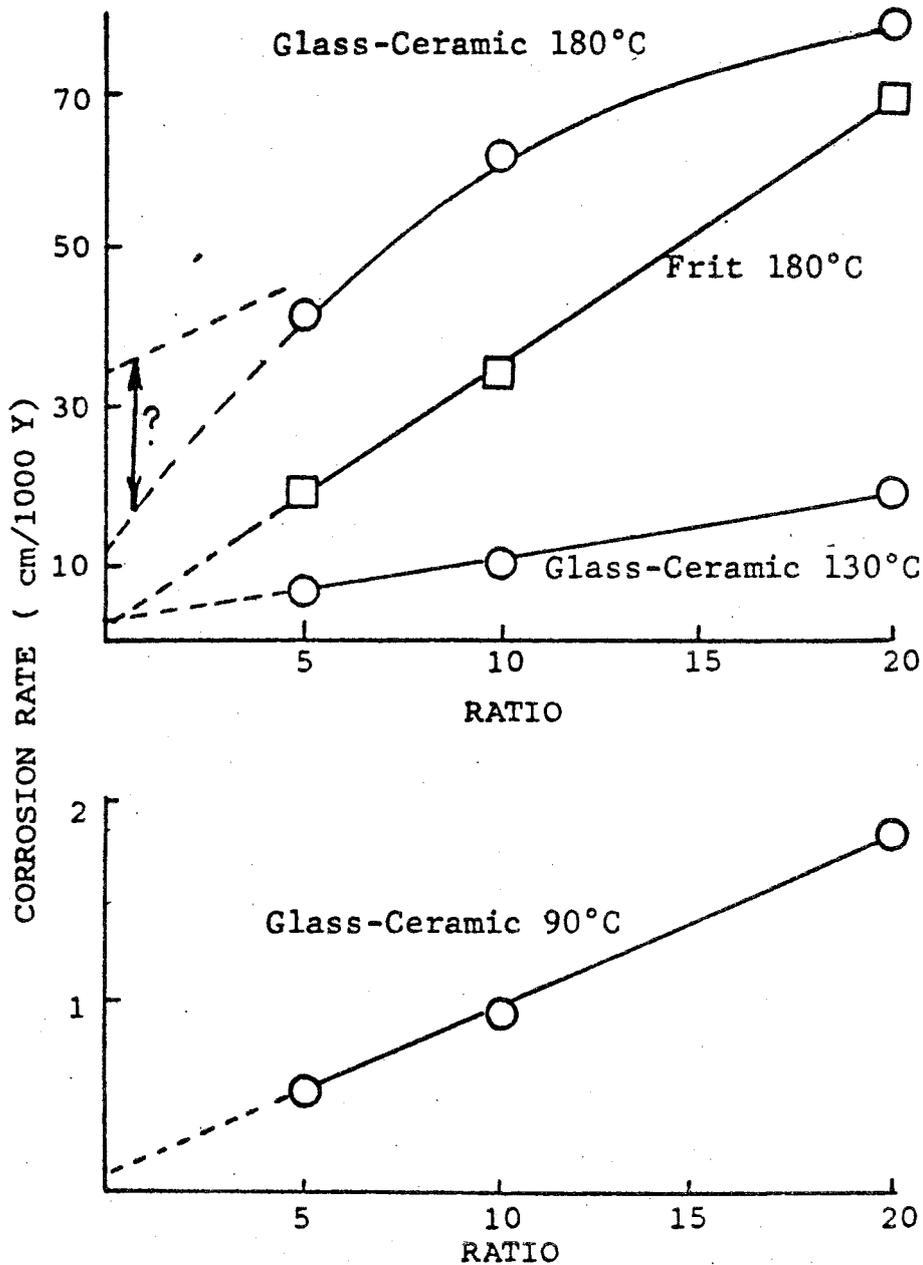


Figure 9. Corrosion Rate as a Function of the Ratio of Solution Volume to Surface Area. Zero Ratio is Equivalent to Corrosion Over the Long Term.

The predicted long term corrosion rates from this extrapolation are shown below in Table 3.

<u>Material</u>	<u>T(°C)</u>	<u>Corrosion Rate (cm/1000 Y)</u>
Glass-Ceramic	180	8 to 33
	130	3
	90	0.1
Frit	180	2

Table 3. Predicted Long Term Corrosion Rates from the Extrapolation of Data to a Zero Ratio.

Figure 10 shows the glass-ceramic corrosion rate plotted as a function of temperature for ratios of 0 (obtained by extrapolation), 5, 10, and 20 ml/cm². The zero ratio curve represents our best guess for the long term corrosion rate.

To obtain an accurate prediction of actual total corrosion we need to consider the effects of cooling. Using the cooling curve shown earlier in Figure 2, the following calculations can be made:

<u>Average Temperature (°C)</u>	<u>Time Interval (yrs)</u>	<u>Rate (cm/1000 Y)</u>		<u>Material Removed (cm)</u>	
		<u>R = 20</u>	<u>R = 0</u>	<u>R = 20</u>	<u>R = 0</u>
100	20	3.5	0.28	0.07	0.006
75	80	0.5	0.08	0.04	0.006
50	400	0.023	< 0.001	0.009	< 0.0004
40	<u>500</u>	0.005	< 0.001	<u>0.003</u>	< <u>0.0005</u>
Total	1000			0.12	0.013

Table 4. Estimated Glass-Ceramic Corrosion Rate in Water Taking Into Account the cooling of the repository Site. Sample is Assumed Wet at Time of Deposit.

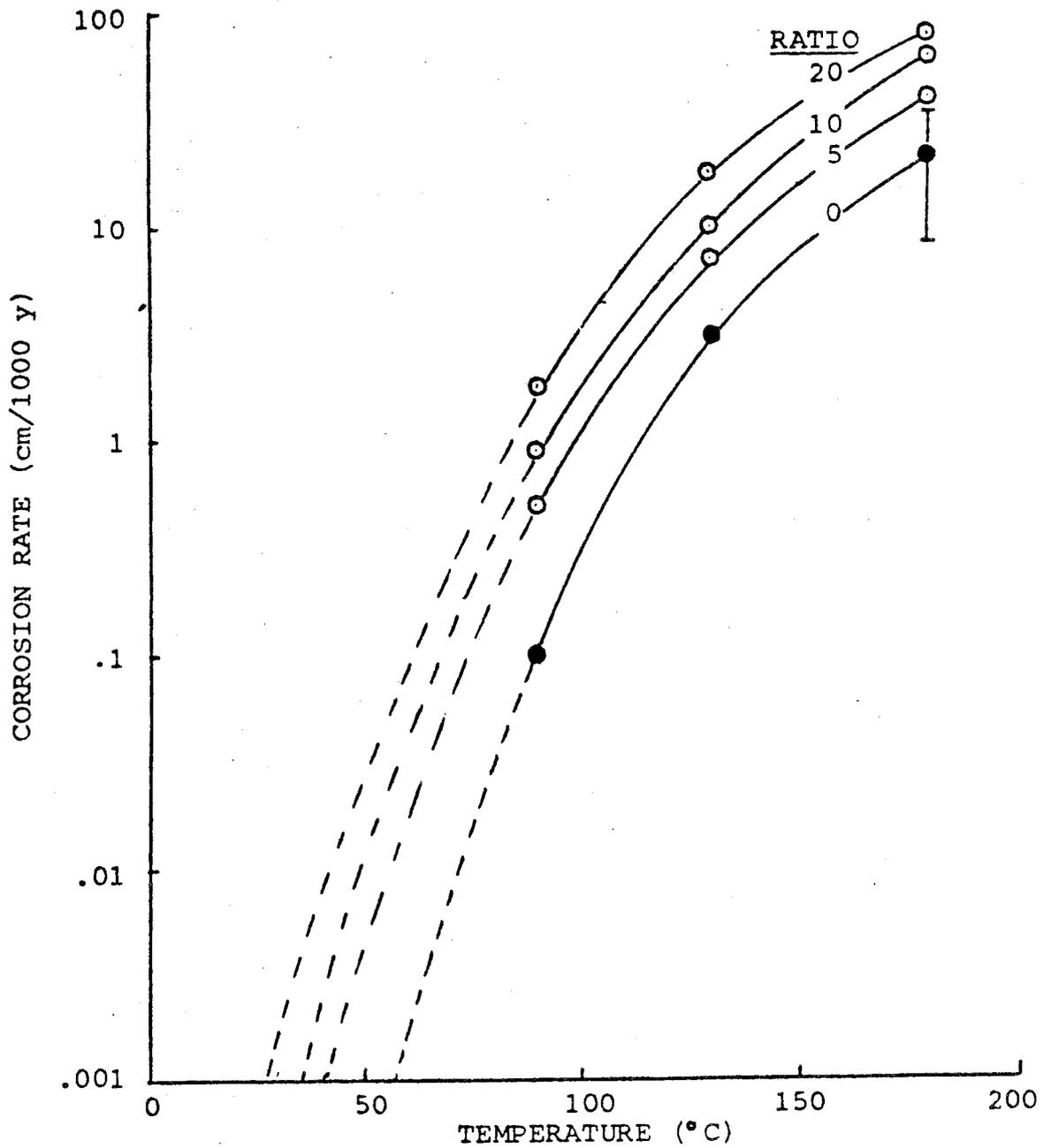


Figure 10. Corrosion Rate as a Function of Temperature and Ratio for the Glass-Ceramic

These estimated rates are well within the limit of 1 cm/1000 years that we were targeting.

Note that if it is taken into account that the repository void space is not filled with water until 200 years these estimates are high.

6.3 The Effect of Sand and Bentonite

The preliminary data on the effect of sand and bentonite on corrosion are shown below:

Material	Test Matrix	R	Temperature °C	Estimated Corrosion Rat cm/1000 Y
Frit	9 sand/1 bentonite/H ₂ O ^c	220	90	4
	9 sand/1 bentonite/H ₂ O ^c	75	90	3
Glass-ceramic	9 sand/1 bentonite/H ₂ O ^c	~75	90	~75 ^a
Glass-ceramic	9 sand/1 bentonite/H ₂ O ^c	2	60	0.7
	H ₂ O	~ 2	~ 60	~ 0.01 ^b

Table 5. Preliminary Corrosion Estimates on the Effect of the Sand Bentonite Mix^c Compared to Pure Water

^afrom Figure 9; ^bfrom Figure 10; ^c9 grams sand + 1 gram bentonite made up to volume with H₂O

These values indicate the corrosion in the mix to be within accepted limits considering that the data are for ratios higher than the expected equilibrium ratio of zero.

6.4 The Effect of Solution Flow

The rate of influx of ground water is estimated to be about 1 milliliter per centimeter of glass-ceramic per year. This low rate was considered insignificant in its effect on the chemistry of the system and was not investigated.

6.5 The Effect of the Composition of the Ground Water

This was to be studied later in the course of our work.

6.6 Sodium Chloride Corrosion

A small amount of data was obtained on corrosion by aqueous salt solution (0.5 gms/ml). Some of the data was confounded by corrosion of the stainless steel test containers. In these instances, the glass-ceramic showed little corrosion relative to the steel. Table 6 shows the corrosion rate estimates for aqueous salt attack on the frit and the glass-ceramic.

<u>Material</u>	<u>Solution</u>	<u>R</u>	<u>Time (Hours)</u>	<u>Temperature °C</u>	<u>Corrosion I cm/1000</u>
Glass-Ceramic	NaCl	20	96	180	30.
	H ₂ O	20	96	180	78.
	NaCl	20	96	130	18.
	H ₂ O	20	96	130	18.
	NaCl	20	96	90	0.4
	H ₂ O	20	96	90	1.8
Frit	NaCl	20	96	130	27.
	H ₂ O	8	96	130	26.
	NaCl	20	96	90	2.8
	H ₂ O	20	96	90	8.2

Table 6. Preliminary Data on the Corrosion Rate of NaCl Solution Compared to H₂O for the Glass-Ceramic and the Frit.

These corrosion rates seem to be comparable to water corrosion rates.

7. Corrosion Mechanism

We have evaluated the corrosion mechanism on a macro and an atomic scale. The next two sections address these topics.

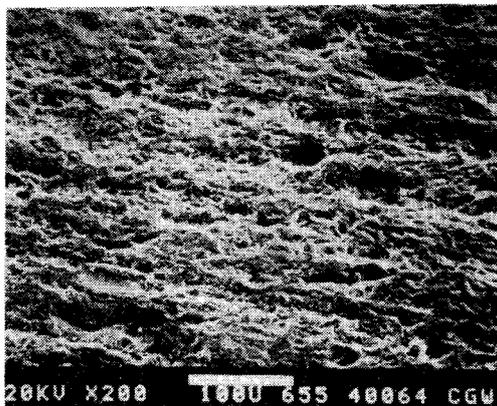
7.1 Selective Attack

The Code 9617 glass-ceramic is a composite of several phases as shown below:

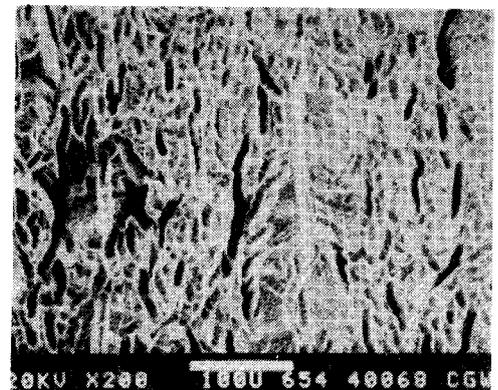
β -spodumene ($\text{Li}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot x \text{SiO}_2$) ($x = 5$ to 7)	~ 90%
rutile (TiO_2)	~ 3
spinel (Mg or $\text{Zn} \cdot \text{Al}_2\text{O}_4$)	~ 3
residual glass (high Zn)*	~ 5

*determined by scanning transmission electron energy dispersion.

Because of this, one would expect the composite material to exhibit different localized corrosion rates. This appears to be the case. Scanning electron microscopy of the H_2O corroded glass-ceramic surface showed the glass phase to be removed leaving the crystalline phases relatively untouched.



A



B

Figure 11. Scanning Electron Micrographs Comparing Water Attacked Surface (B) with Unattacked Surface (A). Attack was 4 Hours at 180°C in H_2O

Figure 12 shows the remaining crystalline phases in greater detail.

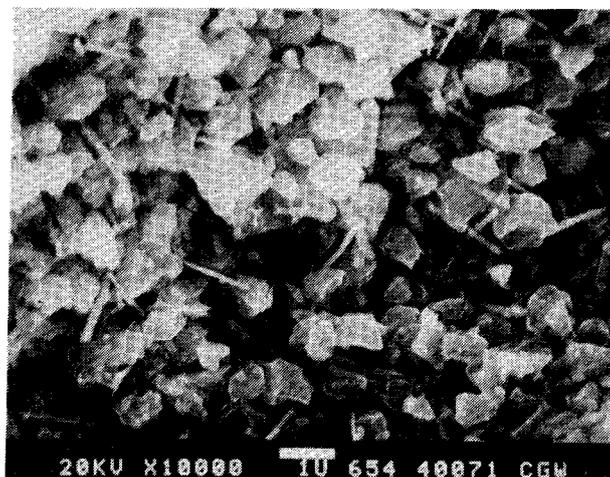


Figure 12. Higher Magnification Scanning Electron Micrograph of Glass-Ceramic Showing Only Crystals Remaining After 4 Days at 180°C in Water

This evidence coupled with the cloudiness of the solution suggests the mechanism of attack described below in Figure 13.

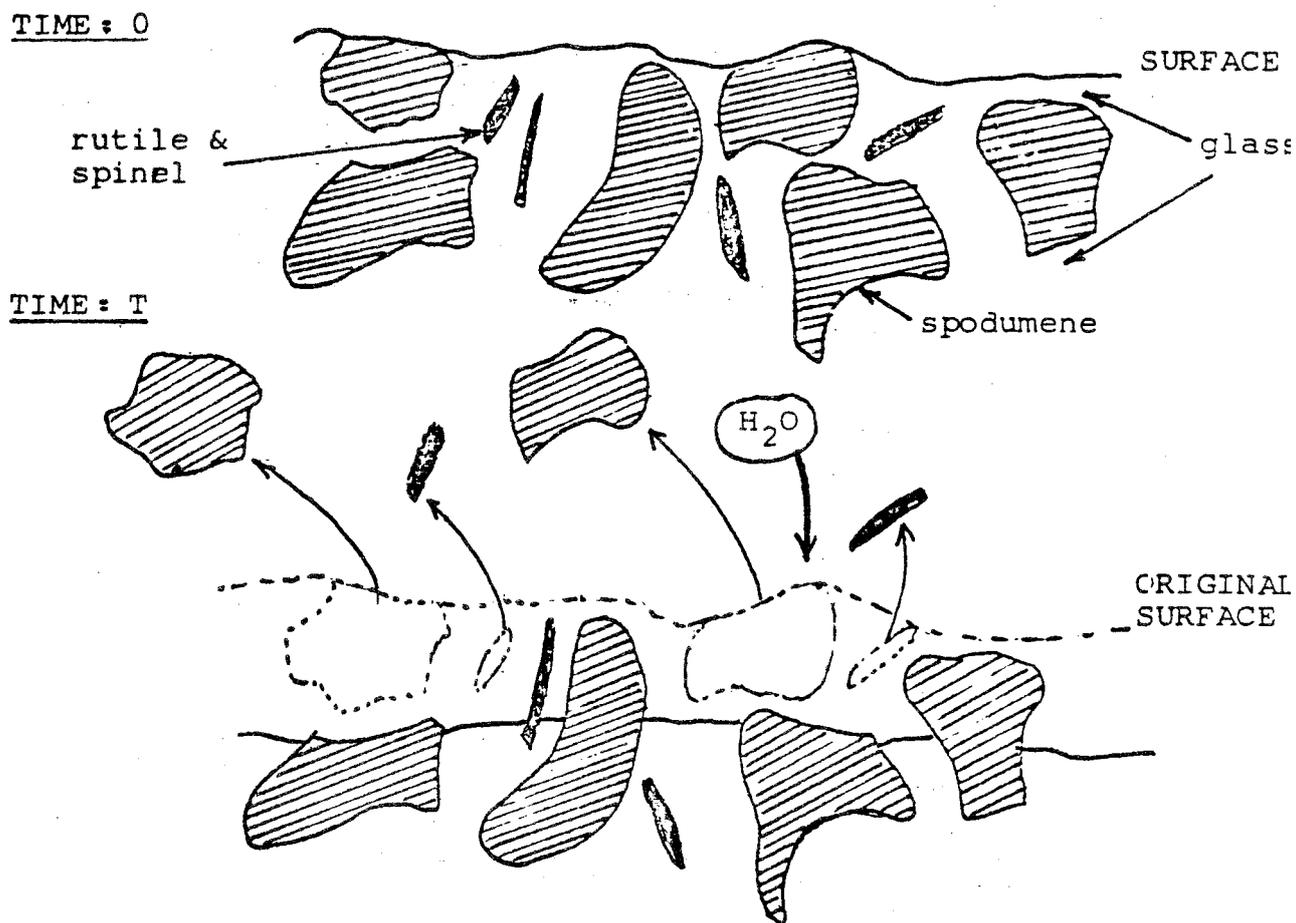


Figure 13. Suggested Corrosion Mechanism: Selective Leaching Away of Glassy Phase Followed by Dislodging of Unreacted Crystalline Phase.

Improving the durability of this glassy phase should improve the overall glass-ceramic durability. This was demonstrated using an SO₂ treatment, a common method in the glass container industry to improve the durability of some glasses. Hot, wet SO₂ provides free H⁺ ions which exchange with alkali at the surface of the glass. The surface then reconsolidates into a more durable high silica skin. At the temperature of treatment

(~ 550°C) the crystalline phases are probably not affected.

Chemical durability results on some SO₂ treated samples is shown in the table below compared with untreated samples.

<u>Sample</u>	<u>Time(Days)</u>	<u>Temperature °C</u>	<u>Corrosion Rate (cm/1000 Y)</u>
Untreated	1	180	27.
SO ₂ Treated	1	180	11.
Untreated	17	180	4.5
SO ₂ Treated	16	180	0.7
Untreated	17	130	1.3
SO ₂ Treated	16	130	0.4

Table 7. The Effect of SO₂ Treatment on Ceramic Durability.
Glassy Phase Durability Is Improved (R = 0.9)

Though the improved durability only lasts until the silica-rich skin is removed the long range durability will be improved by holding off attack until the canister and its surroundings have cooled to a condition of lesser corrosivity.

The proposed corrosion mechanism presents other opportunities for improving chemical durability:

1. reduction of the amount of glassy phase
2. changing the composition of the glassy phase.

It is worth noting that the spodumene phase appears to be highly durable in a water environment and efforts to improve ceramic durability should aim at increasing the amount of β -spodumene.

7.2 Ion Exchange

Hydrogen ion profiling of the reacted sample surface may aid in understanding the corrosion mechanism on an atomic scale.

Hydrogen and/or water migration into the surface of materials, especially glass and glass-ceramics, play a major role in the corrosion process. Until recently, there has been no method available for profiling this reacted surface. Recent work by Dr. William Lanford² at Yale University has provided us with a method for determining hydrogen ion profiles in glass and glass-ceramics.

The generally agreed upon model for H₂O corrosion of glass is shown schematically in Figure 14.

In:

Step 1: H⁺ ions replace free alkali ions in the glass by ion exchange (leaching)

Step 2: The presence of the alkali ions in the water raises its pH and the resulting hydroxyl groups attack the Si-O-Si network breaking the Si-O to form Si-OH (etching).

Lanford's work suggests that hydronium ions (H₃O⁺), not hydrogen ions (H⁺), are the exchanging species. (in Step 1). Figure 15 shows a typical H⁺ profile and how it relates to the glass (or glass-ceramic) surface.

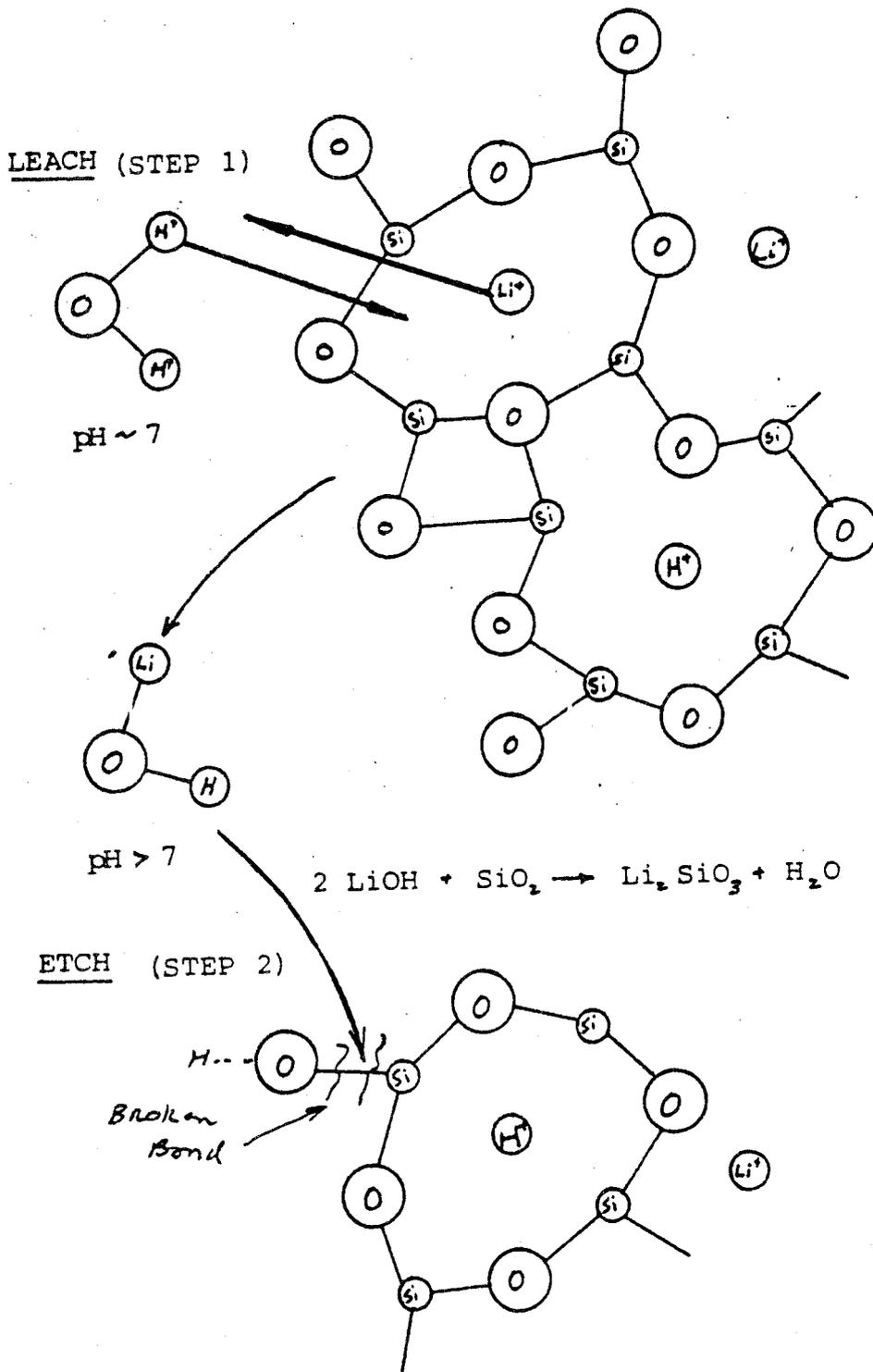


Figure 14. Corrosion Mechanism on An Atomic Level

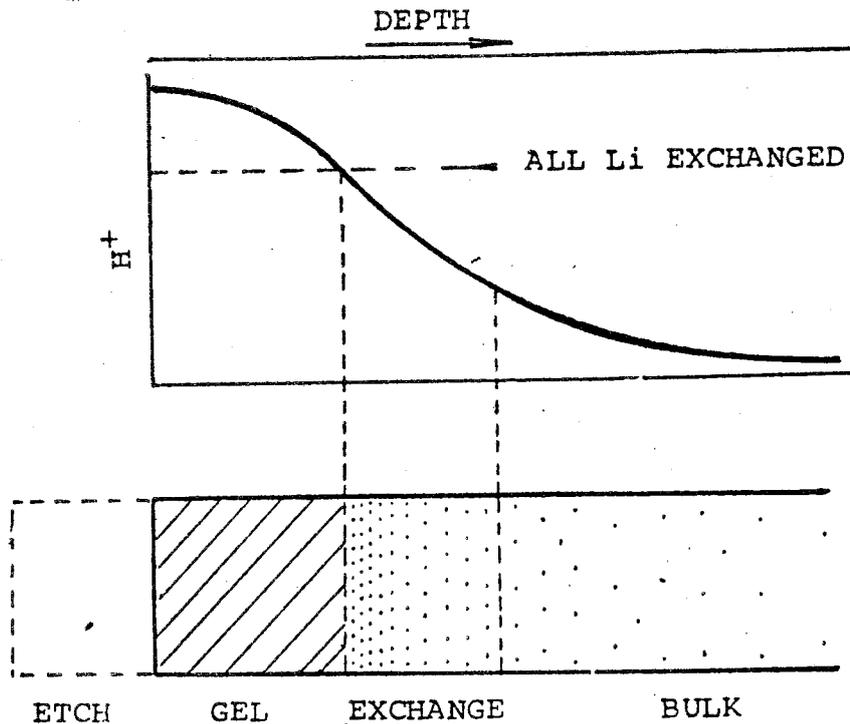


Figure 15. Typical Hydrogen Ion Profile by ^{15}N and How It Relates to the Sample

The horizontal line in the figure labelled "All Li Exchanged" corresponds to the point where the H^+ ion concentration is three times the concentration of the Li^+ ions in the bulk ceramic as calculated from the composition. That is, we assume that one H_3O^+ ion exchanges for one Li^+ ion. The hydrogen ions in excess of this value are assumed to correspond to bulk water that saturates the ion exchanged silica skin. This is called the hydrated gel layer. The depth of exchange below the gel/bulk interface is called the exchanged layer.

From H^+ profiles measured by Lanford and weight loss data we have computed the thickness of the etch, gel and ion-exchanged layers for several test conditions:

Test Conditions		Etch	Gel	Ion-Exchange
Time (hours)	Temperature ($^{\circ}C$)			
4	130	0.04	0.00	0.06
24	130	0.07	0.00	0.15 (?)
96	130	0.47	0.00	0.06
408	130	0.59	0.05	0.08
4	180	0.47	0.00	0.07
24	180	0.74	0.00	0.08
96	180	1.6	0.08	0.08
408	180	2.1	0.72 (?)	0.66 (?)

Table 8. Some Preliminary Calculations of Etch, Gel and Ion-Exchange Layers from Weight Loss and Hydrogen Ion Profile Data ($R = 0.9$)

The sharp increase in the thickness of the gel and ion-exchange layer at 408 hours $-180^{\circ}C$ is suggestive of a mechanism change. More work is necessary to fully understand this.

8. Conclusions

The first objective, learning how to best do experiments for long term prediction under the specified conditions has been achieved. Some tentative conclusions can be drawn from the data regarding the second objective-predicting the extent of corrosion over a 1000 year span. Information has also been obtained regarding other chemical environments such as an aqueous salt system and modifications to the storage site and/or the cannister

material to prolong the storage life.

The optimum test conditions would embrace the actual time, temperature, chemistry and flow conditions to be found at the repository site. Since this cannot be done, it is preferable to conduct the tests at test site temperatures and extrapolate to the longer times required for prediction. Mechanism changes seem to occur when increased temperature is used and this could invalidate conclusions. However, a few tests at elevated temperatures might be used to rapidly identify possible "worst case" situations. Such worst case situations should then be validated under expected site temperatures.

Extreme care must be taken in the design and interpretation of experiments to assure that the chemistry simulation is accurate, and/or that deviations are understood and accounted for. Simply duplicating the initial chemistry of the system is not enough. This is particularly true in a situation that is expected to be "static" regarding ground water movement.

If further work is to be done in the system described in this report, the following is recommended:

1. Temperature - 30°, 60°, 90°, and 120°C
2. Time - as long as practical
3. Initial solution - ground water in equilibrium with 9 sand and 1 bentonite
4. Solution volume to sample surface area ratio - very high, very low
5. Determine - Weight loss, ions in solution (Si, Li, etc.)
- Electron micrographs of surface and cross-section
- Hydrogen and possibly lithium ion profiles.

Composite "sandwiches" of the ceramic-frit-ceramic body should be tested as well as small samples from an actual cannister.

The data obtained at this point indicate that the ceramic would be reduced in thickness by about 0.1 cm in 1000 years under the specified conditions. The experimental frit candidate tested would apparently be corroded to a depth of about 1 cm in 1000 years. The corrosion rates in cm/1000 years for the glass-ceramic are estimated in Figure 10 (zero ratio = static condition). Typically, rates in flowing conditions are 2 cm/1000 Y at 100°C, 0.1 at 50°C; in static conditions, 0.2 cm/1000 Y at 100°C and 0.001 at 50°C.

Corrosion rates in aqueous salt systems do not appear to differ dramatically from those found in water; if anything they appear to be lower. In limited testing we found a static corrosion rate of about 0.1 cm/1000 Y. These data may have been affected by the stainless steel corrosion products from the test container.

The strong dependency of the corrosion rate "on the ratio" of solution volume to sample surface area in actuality represents the effect of different concentrations of dissolved ions, probably silicon and aluminum complexes. This suggests that any potential corrosion of a cannister stored in a water cooled basin could be decreased by appropriate chemical additions to that water.

The β -spodumene crystal fraction of the glass-ceramic seems to be extremely corrosion resistant to near neutral (pH ~ 7) solutions. The glassy phase is apparently the less durable fraction.

It is probable that more resistant glass-ceramics could be designed by either adjusting the composition or the ceramming process.

Any further studies should include the evaluation of the effects of radiation damage on corrosion, the possibility of crystal growth and resultant fracture in the fritted seal joint and the change in physical strength as corrosion proceeds.

Two major concerns emerge:

1. the limited results on water in contact with bentonite and sand suggest that the rate of corrosion could be different than for water, and
2. the H^+ profile results suggest that, long term, there could be a mechanism change to accelerate the corrosion process.

Finally, note that the bulk of these conclusions regarding corrosion rates and mechanisms rests on limited data and should be further studied.

March 14, 1978
(Sundquist, J.D.)

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elc
3/15/78

March 14, 1978
(Sundquist, J.D.)

APPENDIX I. GLASS-CERAMIC CODE 9617 Corrosion Test Data^a

Ref.	Test Description	Ratio (ml/cm ²)	Time (Days)	Temperature (°C)	Final pH	Wt. Loss (mg/cm ²)	Extracted Solution (mg/cm ²)				
							SiO ₂	Al ₂ O ₃	Li ₂ O	MgO	ZnO
5	H ₂ O	1.7	17	60	7.7	0.076	0.005	<0.0002	0.001	0.00005	0.00002
3	H ₂ O/9 sand/1 benton- ite (3 gms)	1.7	17	60	8.9	0.088					
61	H ₂ O	220	4	90	6.3	≤0.01					
64	H ₂ O	220	16	90	7.1	0.04	0.44	<0.02	< 0.002	<0.002	0.007
79	H ₂ O	200	14	90	7.5	<0.01	2.4				
114	H ₂ O	200	54	90	7.5	2.29					
60	H ₂ O	150	4	90	6.1	0.01					
63	H ₂ O	150	16	90	7.0	<0.01					
97	H ₂ O	100	16	90	7.7	0.16					
62	H ₂ O	75	4	90	6.7	0.03					
65	H ₂ O	75	16	90	6.9	<0.01					
78	H ₂ O	20	16	90	7.9	0.05	0.57	0.036	0.007	0.010	0.001
96	H ₂ O	20	16	90	7.3	0.13					
77	H ₂ O	10	16	90	8.3	0.02					
76	H ₂ O	5	16	90	8.1	0.02					
107*	change H ₂ O once/week	76	70	90	5.9	0.04					
128	change H ₂ O once/week	10	22	90	8.4	0.40					
70	H ₂ O/9 SiO ₂ /1 benton- ite (5 gms)	220	4	90	9.0	0.05					
68	H ₂ O/9 SiO ₂ /1 benton- ite (10 gms)	220	16	90	8.7	0.02					
69	H ₂ O/9 SiO ₂ /1 benton- ite (5 gms)	75	16	90	8.7	0.11					
94	H ₂ O/NaCl (0.5 gms/ml)	20	4	90	7.5	<0.01					
74	Buffered ¹ (pH=8.5)	150	16	90	8.5	0.03					
57	Buffered ¹ (pH=8.5)	15	3	90	8.4	<0.01					
59	Buffered ² (pH=8.6)	15	3	90	8.6	0.12					
75	Buffered ¹ (pH=8.5)	15	16	90	8.6	0.02					
73	Buffered ¹ (pH=8.5)	10	4	90	8.4	<0.01					
56	Buffered ¹ (pH=8.5)	2	3	90	8.3	<0.01					
58	Buffered ² (pH=8.6)	2	3	90	8.6	<0.01					

*detailed in Appendix 3

¹(Hydroxy-methyl) amino methane+HCl

²Borate+HCl

^aNote: no data excluded although some may be spurious.

Ref.	Test Description	Ratio (ml/cm ²)	Time (days)	Temperature (°C)	Final pH	Wt. Loss (mg/cm ²)	Extracted Solution (mg/cm ²)				
							SiO ₂	Al ₂ O ₃	Li ₂ O	MgO	ZnO
22	H ₂ O	11	4	130	7.0	0.32	0.067	< 0.001	0.077	0.043	0.029
48	H ₂ O	0.9	4	130	7.0	< 0.01	0.0057	0.0007	0.0007	0.0002	
50	H ₂ O	0.9	1	130	7.2	0.018	0.011	0.0008	0.0011	0.0001	0.00004
52	H ₂ O	0.9	4	130	7.7	0.12	0.067	0.0052	0.0041	0.0004	0.0002
54	H ₂ O	0.9	17	130	7.5	0.15	0.104	0.0065	0.0063	0.0005	0.00002
93	H ₂ O/NaCl(0.5 gms/ml)	20	4	130	5.3	0.5					
87	Buffered ¹ (pH=8.5)	10	4	130	8.3	0.18					
106	H ₂ O (glass-ceramic SO ₂ treated)	0.9	16	130	7.0	0.04					
21	H ₂ O ³	21	4	180	7.4	2.12 ³	2.21	0.095	0.29	0.28	0.04
100	H ₂ O	20	4	180	7.6	2.2					
121	H ₂ O	18	1	180	7.8	5.4					
124	H ₂ O	11	4	180	8.9	2.1					
123	H ₂ O	10	1/6	180	8.0	0.07					
101	H ₂ O	10	4	180	7.5	1.25					
20	H ₂ O ³	10	4	180	7.9	2.8 ³	1.56	0.12	0.25	0.092	0.17
122	H ₂ O	9	1	180	7.9	0.73					
19	H ₂ O	5.6	4	180	7.8	1.24	0.73	0.036	0.14	0.02	0.15
23	H ₂ O	3.4	4	180	7.5	0.96	0.71		0.081	0.008	0.066
102	H ₂ O	3	4	180	7.4	0.054					
18	H ₂ O	2.7	4	180	7.7	0.32	0.40		0.073	0.009	0.086
49	H ₂ O	0.9	1/6	180	7.7	0.12	0.066	0.0086	0.0039	0.0007	0.0001
88	H ₂ O	0.9	1	180	7.7	0.19	0.12	0.017	0.0069	0.0005	0.00009
53	H ₂ O	0.9	4	180	8.0	0.40	0.27	0.034	0.014	0.0005	0.0006
98	H ₂ O	0.9	4	180	7.7	0.43					
99	H ₂ O	0.9	16	180	8.8	0.72					
55	H ₂ O	0.9	17	180	8.4	0.54	0.40	0.02	0.02	0.0006	0.0006
129	experimental glass-ceramic/H ₂ O	20	14	180	8.2	4.32					
125	experimental glass-ceramic/H ₂ O	11	4	180	7.7	1.67					
115	experimental glass-ceramic/H ₂ O	4.6	1	180	7.0	0.59					
103	experimental glass-ceramic/H ₂ O	0.9	16	180	8.6	0.67					
92	H ₂ O/NaCl(6 gms/ml) ³	20	1/6	180	4.4	0.18 ³					
91	H ₂ O/NaCl(6 gms/ml) ³	20	1	180	4.5	0.46 ³					
90	H ₂ O/NaCl(6 gms/ml) ³	20	4	180	3.6	0.85 ³					
89	H ₂ O/NaCl(6 gms/ml) ³	10	4	180	9.7	0.98					
104	H ₂ O/glass-ceramic -SO ₂ treated	0.9	1	180	8.0	0.08					
120	(#104 continued)	0.9	16	180	8.9	0.62					
105	H ₂ O (glass-ceramic SO ₂ treated)	0.9	16	180	8.9	0.08					

^aNote: no data excluded although some may be spurious.

Experimental Frit Glass Code 186AYU. Corrosion Test Data

Ref	Test Description	Ratio	Time (days)	Temperature (°C)	Final pH	Wt Lc (mg/cm ²)	
85	H ₂ O	220	63	60	5.6	0.19	
33	↓	62	1/6	90	7.2	0.00	
34		67	1-1/6	90	6.7	0.16	
35		63	4	90	6.5	0.25	
66		220	4	90	6.9	0.33	
67		220	16	90	6.9	0.36	
27		8	1/6	130	6.4	0.05	
28		8	1-1/6	130	6.5	0.39	
29		8	4	130	6.7	0.73	
30		8	1/6	180	6.7	0.46	
31		8	1-1/6	180	7.1	1.9	
32		8	4	180	7.0	3.17	
81		3	4	180	7.7	0.52	
82		5	4	180	7.2	0.61	
83		10	4	180	7.2	0.80	
84		20	4	180	7.2	1.93	
110		H ₂ O change solution (10 gms) once/wk	150	70	90	5.5	0.66
71		H ₂ O/sand/bentonite (10 gms)	220	1/6	90	8.6	0.06
72	↓	220	16	90	7.5	0.44	
96	H ₂ O/NaCl	20	4	90	7.6	0.08	
95	H ₂ O/NaCl	20	4	130	6.3	0.76	

*Solution extraction < 0.03 mg/cm² PbO

^aNote: no data excluded although some may be spurious

March 14, 1978

APPENDIX III-A^a -H₂O Solution Analysis for Glass-Ceramic (fresh water once a week)

Ref.	R	Time (Weeks)	Temperature (°C)	mg/cm ²				
				SiO ₂	Al ₂ O ₃	Li ₂ O	MgO	ZnO
107	76	1	90	0.46	< 0.008	0.008	0.002	< 0.0008
		2		0.16	< 0.008	0.008	0.002	< 0.0008
		3		0.15	< 0.008	< 0.0008	0.002	< 0.0008
		4		0.11	< 0.008	< 0.0008	0.0008	0.002
		5		0.09	< 0.008	< 0.0008	0.002	0.002
		6		0.12	< 0.008	< 0.0008	0.002	0.002
108	13	1	130	0.21	0.039	0.033	0.006	0.0001
		2		0.18	0.008	0.019	0.003	0.0004
		3		0.19	0.006	0.02	0.003	0.0001
		4		0.19	0.022	0.023	0.002	0.0008
		5		0.19	0.063	0.021	0.002	0.002
		6		0.19	< 0.001	0.021	0.002	0.001
109	9	1	180	0.70	0.047	0.079	0.002	0.005
		2		0.62	0.067	0.065	0.002	0.005
		3		0.55	0.063	0.058	0.002	0.005
		4		0.57	0.14	0.067	0.001	0.005
		5		0.57	0.13	0.078	0.001	0.005
		6		0.60	0.14	0.077	0.001	0.006

APPENDIX III-B -H₂O Solution Analysis for Frit (fresh water once a week)

	R	Time (Weeks)	Temperature (°C)	SiO ₂	PbO
110	59	1	90	1.3	< 0.0006
		2		0.32	< 0.0006
		3		0.71	0.012
		4		0.59	< 0.0006
		6		0.65	< 0.0006
		10			0.056
111	7.3	1	130	0.035	0.15
		2		0.042	0.03
		3		0.031	0.024
		4		0.024	0.017
		5		0.060	0.032
		6		0.039	0.022
		10		0.016	
112	8.4	1	180	0.21	0.068
		2		0.078	0.029
		3		0.060	0.027
		4		0.066	0.026
		5		0.003	0.023
		6		0.009	0.051
		10		0.02	

etc
3/17/78

^aNote: no data excluded although some may be spurious.

March 14, 1978 APPENDIX IV - Compositions
(Sundquist, J.D.)

A. Water Solution After Contact with 9 Sand & 1 Bentonite Mix^a
(1 gram/20 ml)

<u>Conditions</u>		<u>Concentration in Solution (ppm)</u>					
<u>Time(days)</u>	<u>Temperature(°C)</u>	<u>SiO₂</u>	<u>Al₂O₃</u>	<u>Li₂O</u>	<u>MgO</u>	<u>Na₂O</u>	<u>ZnO</u>
4	25	175	127	0.06	15.	60	0.1
4	130	340	354	0.1	38.	120.	0.6
4	180	390	190	0.1	23.	90.	1.2

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- 02 PM angående värmeledningstal hos jordmaterial
Sven Knutsson
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Högskolan i Luleå 77-04-15
- 03 Deponering av högaktivt avfall i borrhål med buffertsubstans
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Högskolan i Luleå 77-05-27
- 04 Deponering av högaktivt avfall i tunnlar med buffertsubstans
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