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LEACH TESTS AT SAVANNAH RIVER LABORATORY FOR RADIOACTIVE AND NONRADIOACTIVE NUCLEAR WASTE GLASSES (U)

by

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Slides and a Summary of the Slides for an Invited Review Presentation at the 197th National Meeting of the American Chemical Society, April 9-14,1989, Dallas, Texas.

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LEACH TESTS AT SAVANNAH RIVER LABORATORY FOR RADIOACTIVE AND NONRADIOACTIVE NUCLEAR WASTE GLASSES*

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ABSTRACT

The high-level nuclear wastes currently stored at Savannah River Plant as caustic slurries will be immobilized into borosilicate glass. To characterize the glass resulting from this vitrification process, the Savannah River Laboratory (SRL) has developed and performed a variety of leach tests on both radioactive glass containing actual nuclear waste and nonradioactive glass containing simulated waste. These leach tests range from simple tests using a monolithic samples in deionized water to complex tests using radioactive glass and performed remotely. Some of the tests were designed to determine the effects of factors such as radiation or groundwater oxidation potential (Eh) on the leaching process. Other tests were designed to obtain a measure of how the waste glass will respond in geologic environments. This paper describes the rationale used in the development of these tests and presents a brief survey of the tests including some of their special procedures and pertinent results.

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TITLES OF SLIDES

1. Title slide: "Leach Tests at Savannah River Laboratory for Radioactive and Nonradioactive Nuclear Waste Glasses," by Ned E. Bibler and Carol M. Jantzen.

2. Rationale used in Leach Test Development.

3. Composition of a Typical Radioactive SRP Nuclear Waste Glass.

4. Experimental Factors Studied at SRL to Determine their Effect on Leaching SRP Nuclear Waste Glasses.

5. Procedure for the MCC-1 Leach Test.

6. Effect of pH on Release of Si from a Nonradioactive Simulated Nuclear Waste Glass.

7. Normalized Si Release in the MCC-1 Test from Various Glasses as a Function of the Free Energy of Hydration Calculated from the Glass Composition and Solution pH.

8. Special Procedures for Low Eh Leach Tests.

9. Achievement of Low Redox Potential in Simulated Basalt Groundwater for Leach Tests.

10. Boron Release from Leaching a Nonradioactive Simulated Nuclear Waste Glass in Reducing and Oxidizing Simulated Basalt Groundwater.

11. Effect of Heavy Ion Irradiation of SRP Glass on the Release of B and Li in Leach Tests in Deionized Water.

12. Stainless Steel and Teflon® Leach Vessels for Leach Tests with Radioactive SRP Nuclear Waste Glasses.

13. Release of B, Li, and Cs-137 in Leach Tests with Radioactive SRP Nuclear Waste Glasses in Stainless Steel and Teflon® Vessels.

14. Comparison of Leaching B, and Li from Radioactive and Nonradioactive SRP Nuclear Waste Glasses.

15. Comparison of Pu-238 in Solution and Total Pu-238 Leached from a Radioactive SRP Waste Glass.

16. Apparatus for Leach Tests to Simulate a Saturated Tuff Repository Environment.

17. Partially Assembled Apparatus for Leach Tests to Simulate a Saturated Tuff Repository Environment.

18. Release of Boron from Radioactive and Nonradioactive SRP Nuclear Waste Glasses in Leach Tests to Simulate a Saturated Tuff Repository Environment.

19. Release of Cs-137 from a Radioactive SRP Nuclear Waste Glasses in Leach Tests to Simulate a Saturated Tuff Repository Environment.

20. Special Procedures for the Crushed Glass Leach Test, the Product Consistency Test (PCT) to be used during DWPF Operation.

21. Comparison of the Release of B and Li from Gamma Irradiated and Unirradiated Simulated Nuclear Waste Glass in the PCT.

22. Normalized B Release in the PCT Leach Test for Various DWPF Glasses as a Function of the Free Energy of Hydration Calculated from the Glass Composition and Solution pH.

23. Conclusions.

SUMMARY OF SLIDES

Slide 1:

The high-level nuclear wastes currently stored at Savannah River Plant (SRP) as caustic slurries will be immobilized into borosilicate glass. Currently the Defense Waste Processing Facility (DWPF) is being built at SRP to accomplish this. To characterize the glass resulting from this vitrification process, Savannah River Laboratory (SRL) has performed a variety of leach tests on both radioactive glass containing actual nuclear waste and nonradioactive glass containing simulated waste. These leach tests range from simple tests using a monolithic samples in deionized water to complex tests using radioactive glass and performed remotely. Some of the tests were designed to determine the effects of factors such as radiation or groundwater oxidation potential (Eh) on the leaching process. Other tests were designed to obtain a measure of how the waste glass will respond in geologic environments. This paper describes the rationale used in the development of these tests and presents a brief survey of the tests themselves including some of the special procedures and pertinent results.

Slide 2:

When developing a leach test, several things need to be considered. First, the purpose of the test should be very specific. In some cases, a test procedure may be general enough so that the test may be multipurpose; however, the generality of the test should not jeopardize the specific purpose for which the test was designed. Second, the procedures should be carefully developed so that the results of the test truly reflect the durability of the glass. This means the elimination or control of all experimental factors that could artificially influence the results of the test. Examples of such factors are the radiolysis of air in the leach vessel, the effects of sample preparation such as the surface finish on the glass, or the material of the leach vessel itself. Third, the use of blanks, replicates, and standard glasses as controls should be evaluated. Blanks and replicates are necessary to establish the reliability of the data and to define a good statistical base. Leaching standard glasses as controls is especially valuable when several tests are performed independently and the results need to be compared. Last, thought should be given as to exactly what data to collect and how it will be used. For example, if the purpose of the test is to determine the durability of the glass, then only data for soluble elements in the leachate are appropriate. Data for elements slightly soluble in the leachate would be unsuitable for this purpose. However, if the test is designed to measure the leachability of a slightly soluble element (such as Fe or Pu), then a complete inventory of that element must be performed after the test. This includes acid rinses of the vessel and any other materials in contact with the leachate. For completeness, analysis of the leached surface of the glass is appropriate to determine which elements concentrated on the surface of the glass or which became depleted.

Slide 3:

This slide shows the composition of a typical radioactive SRP nuclear waste glass. It was prepared by mixing the waste (primarily Fe, Al, and Mn hydrous oxides contaminated with radioactive fission products and actinides) with glass forming chemicals (primarily Si, B, Na, and Li oxides) in the form of a prefabricated nonradioactive frit. The mixture is then melted at 1150°C in a joule heated melter. The final glass is nominally 30 wt% waste and 70 wt% frit. All of the glasses leached at SRL in this program have compositions similar to that shown here. On a mass basis, the amount of radioactive material is less than 1%; thus, radioactive and nonradioactive glasses should have similar properties. Radioactive glasses are prepared remotely because of their high radiation levels. Nonradioactive glasses containing simulated waste are prepared in laboratory furnaces or large scale melters.

Slide 4:

This slide lists the many experimental factors investigated at SRL to determine their effect on the leaching of SRP nuclear waste glasses. The efforts at SRL are only part of an international program to understand the leaching processes of borosilicate glasses immobilizing nuclear wastes. Most of the leach tests at SRL were initially based on the MCC-1 leach test developed by the Materials Character Center at Battelle Northwest Laboratory [1]. This leach test is an excellent test to screen glass compositions as well as to determine the effects of experimental factors on the leaching process. Examples of specific factors investigated at SRL include temperature, leachate composition, glass composition, surface finish of the glass, crystalline content of the glass, and radiation effects on the glass. A brief outline of the MCC-1 procedure is given in the next slide.

Slide 5:

The MCC-1 leach test [1] uses a monolith of glass of known geometrical area. The glass is leached in deionized water at 90°C for a known amount of time (usually 3, 7, 14, or 28 days). For each square centimeter of glass surface area leached, 10 mL of leachate are used. After the test, the leachate is acidified and analyzed for components of the glass. Carefully cleaned Teflon® vessels are used for the test. A disadvantage of the MCC-1 test is that its response is strongly affected by the surface finish on the glass. The rougher the surface (120 grit vs. 600 grit), the higher the initial leach rate. This makes the application of the test to radioactive glasses difficult since special techniques and equipment are necessary to remotely prepare reproducible surface finishes on the radioactive glass.

Slide 6:

The effect of pH on the leaching process was examined by Wicks [2] using MCC-1 tests. Results are shown in this slide. At low and high acidities, the leaching is accelerated over that at neutral pH values. In the highly acidic and basic solutions, leaching occurs by Si network dissolution as well as an ion exchange process. Note that at the pH of the groundwaters typical of candidate geologic repositories, leaching is the slowest.

Slide 7:

This slide shows results of a study comparing the durabilities in 28-day MCC-1 tests of glasses of many different compositions. The comparison is based on a hydration free energy model adapted by Jantzen and Plodinec [3] for SRP nuclear waste glasses. In this study, glasses with widely different compositions were leached and the normalized releases of Si compared. The normalized release is the actual release normalized for the amount of Si in that respective glass. The free energy of hydration is a weighted sum of the free energies for the hydration of the various orthosilicate components of the glass such as Li, B, and Fe [3]. Note that the SRP glasses have a durability similar to basalt rock.

Slide 8:

Special leach test procedures had to be developed [4] to determine the effect of Eh on leaching. Some necessary modifications to the MCC-1 procedure are shown in this slide. Reduced or low Eh groundwaters can be present in many geologic formations such as deep basalts. For leach tests at low Eh, all air has to be excluded from the solution both during leachate preparation and the test itself. This requires an Ar glovebox (using 99.999% pur Ar) and airtight leach vessels. Also, reducing agents such as Fe^{2+} or H_2 are necessary in the leachate to achieve reducing conditions. With basalt, the rock furnishes the reducing agent (Fe2+).

Slide 9:

This slide shows the measured Eh in a simulated basalt groundwater leachate as a function of pH and Ar purge time. These data were developed by Jantzen [4] and are presented in the form of a Eh-pH diagram showing the various species of Fe present. The Eh was measured with a clean, calibrated Pt electrode. To prepare this leachate, the solution was heated at 90°C with crushed basalt rock while purging with very pure Ar in an inert box. Ninety hours were necessary to completely convert a leachate from oxidizing to reducing conditions expected in pristine basalt groundwater. Leach vessels were then filled in this box in air tight containers.

Slide 10:

This slide shows the results for a series of modified MCC-1 tests performed with the oxidizing and reducing leachates [5]. A large effect on the B release is not evident, suggesting that the glass reacts similarly under both conditions. For glass components that can be reduced in the low Eh leachate and precipitate, the apparent release is much lower. For example, under oxidizing conditions the radionuclide Tc-99 is released from glass at the same rate as B [6]. Under reducing conditions, Tc is reduced to the 2+ or 4+ state that is insoluble and precipitates. The actual release of Tc-99 from the glass may be the same, but once the Tc-99 reaches the solution, it precipitates. Clearly, one should be cognizant of this behavior when choosing an element in a leach test to measure the durability of a glass.

Slide 11:

This slide shows the results of a study performed at SRL to investigate whether irradiating the glass with heavy ions such as Pb affected the durability of the glass [7]. This study was performed to gain information on how alpha recoils such as Pu-240 from alpha decay of Cm-244 in the glass would affect the glass. For this study, two modifications to the MCC-1 procedure were necessary. These modifications were to decrease the SA/V and leach times. The SA/V was decreased to $0.01 cm^{-1}$ in order to obtain enough solution to analyze. The leach times were shortened considerably to ensure that only the glass damaged by the radiation was being leached. In some cases, the time was only 0.5 hour. Based on B and Li no effect of the ion irradiation is evident.

Slide 12:

This is a picture of the leach vessels and other components for the leach tests with actual radioactive SRP glass performed by Bibler [8]. These tests had to be performed remotely in a shielded cell because of the high radiation levels from the glass. Both Teflon and stainless steel (SS) vessels were used initially. Samples were prepared by pouring the glass into 3/4' ID SS cylinders that were then sliced to obtain samples for leaching. No attempt was made to separate the slice of glass from the SS ring around it. A 600 grit surface was put on the glass slices and their associated SS rings. For the tests, three slices were used to obtain a SA/V 10X greater than that for the MCC-1 test. The slices (separated by SS spacers) were placed in a SS basket. After the test the basket and glasses were removed in the cell. The radioactivity of the leach solution was then low enough so that it could be taken out of the shielded cell to a hood where the leach vessel and solution could be handled directly. During the operations in the shielded cell, a great deal of care had to be exercised in the cell to prevent contamination of the leach solution with radioactivity already present in the cell.

Slide 13:

This slide shows the normalized release of B, Li, and Cs-137 from radioactive glass in both Teflon® and SS vessels [8]. At long times in Teflon™ vessels, glass dissolution increases tremendously as indicated by the high normalized releases of B and Li. Ion chromatography of these leachates indicated high concentrations of F^- ions formed by radiolysis of the Teflon vessel. The high leach rates were apparently caused by reaction these ions with the glass. It is interesting to note that even though the glass is dissolving at a faster rate, the concentration of Cs-137 is not increasing. Apparently, Cs-137 is being retained on the surface of the glass in some secondary phase.

Slide 14:

This slide compares the normalized releases of B and Li from a radioactive glass and from a nonradioactive glass of similar composition and surface finish. Although the data are scattered, there is general agreement among the results and they follow the same trend. On this basis, it appears that the radioactive glass is leaching no differently than the nonradioactive glass. The scatter of the data is attributed to slight differences in the surface finishes of the samples. Optical microscopy indicated that this was especially true where the glass met the SS ring.

Slide 15:

One factor that has to be considered in leach tests is the sorption of the element of interest on other components in the leach test. An example is shown here where the amount of Pu-238 found in the solution is compared to the total amount leached from the glass. The total amount was determined by acid rinses of the vessel and other components of the test until no more Pu-238 was detected in the rinse. In the case of Pu and other slightly soluble elements, more of the element can be sorbed on the test components than is present in solution.

Slide 16:

This slide shows the special equipment necessary to perform a leach test simulating a saturated tuff repository [9]. The tuff geologic formation in the Yucca Mountain in Nevada is being considered as the first USA repository for high level nuclear waste. The leach vessel itself is fabricated of tuff and actual tuff groundwater is used. The entire assemblage is placed in a Teflon vessel so that water is not lost during the test at 90°C. To get a high surface area, three slices of glass separated by SS spacers were used in the SS basket.

Slide 17:

This slide shows the partially completed test apparatus with the groundwater present inside and outside the tuff vessel. After the test, care was exercised so that these two waters are not mechanically mixed. As with the other tests with radioactive glass, the glass was remotely removed from the tuff vessel. Its lid was then replaced and the Teflon capped. The unit could then be removed from the cell and placed in a hood where the solution was removed for analysis.

Slide 18:

This slide compares the release for B from radioactive and nonradioactive glass in the tuff tests with the rock present and absent. Again, within the precision of the data, the radioactive and nonradioactive leach are essentially identical. There is also no large effect due to the tuff rock versus leaching in the groundwater itself.

Slide 19:

The results for Cs-137 do indicate a large effect of the rock. When rock is present, the concentration of Cs-137 is nominally 160 times lower due to sorption of the radionuclide on the rock. Such sorption is beneficial for repository performance since it hinders release of Cs-137 to the biosphere.

Slide 20:

This slide summarizes the procedures for a crushed glass leach test called the Product Consistency Test (PCT) being developed at SRL for use during DWPF operation [10,11]. Crushed glass tests have the advantage in that the glass has a very high surface area and high concentrations of leached components can be achieved quickly. Another big advantage of such tests is that reproducible surfaces can be prepared remotely on radioactive glass samples since a fractured surface is being leached. SRL is currently developing this test to be used routinely during production of actual radioactive waste glass.

Slide 21:

This slide shows the results for the PCT for gamma irradiated and unirradiated simulated nuclear waste glass [12]. Purpose of this study was to determine if gamma irradiation affected the durability of the glass. The results for the irradiated and unirradiated glasses for triplicate tests are in excellent agreement, indicating no affect of the gamma radiation. Also the precision of the data is much better than when slices of glass were leached, presumably because of the reproducible surface finishes of the glass.

Slide 22:

The slide shows the results of the crushed glass leach test for glasses of several compositions representative of those that will be produced at SRP [11]. One radioactive glass and several nonradioactive glasses are included. The normalized release of B adheres well to the linear relationship prescribed by the free energy of hydration model [3]. Since the result for the radioactive glass also adheres to the model, radiation is not affecting the leaching process.

Slide 23:

This slide summarizes some of the points made in this presentation concerning leach tests used at SRL. Conclusions are drawn with regard to the development of the tests and some important results. Tests have to be designed for specific purposes with careful attention given to the test procedures to ensure that no artifact is being introduced which would influence those results. Perhaps the three most important results presented here are that radioactive and nonradioactive glass leach similarly, that gamma and ion radiation do not affect the durability of the glass, and that tuff rock does not affect the leaching process.

ACKNOWLEDGMENT

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LEACH TESTS AT SAVANNAH RIVER LABORATORY FOR RADIOACTIVE AND NONRADIOACTIVE NUCLEAR WASTE GLASSES

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RATIONALE USED IN LEACH TEST DEVELOPMENT

- PRECISE DEFINITION OF PURPOSE
- CAREFUL DEVELOPMENT OF PROCEDURES
- COMPLETE USE OF BLANKS, REPLICATES, AND CONTROLS
- CONSIDERATION OF EXACT USE OF DATA

COMPOSITION OF A RADIOACTIVE SRP NUCLEAR WASTE GLASS

Nonradioactive Composition (Weight % Oxide)

Oxide	Wt. %	Oxide	Wt. %
SiO_2	55.4	Al_2O_3	9.8
Na_2O	11.0	Fe_2O_3	6.0
Li_2O	4.9	MnO_2	1.9
B_2O_3[b]	8.4	CaO	0.24
MgO	1.0	NiO	0.9

Radioactive Composition (mCi per gram glass)

Radionuclide	mCi/g glass	Radionuclide	mCi/g glass
Cs-137	0.111	Sb-125	0.0099
Sr-90	7.18	Eu-154	0.045
Pu-238	0.042	Eu-155	0.016
Zr-95	0.005	Co-60	0.0055

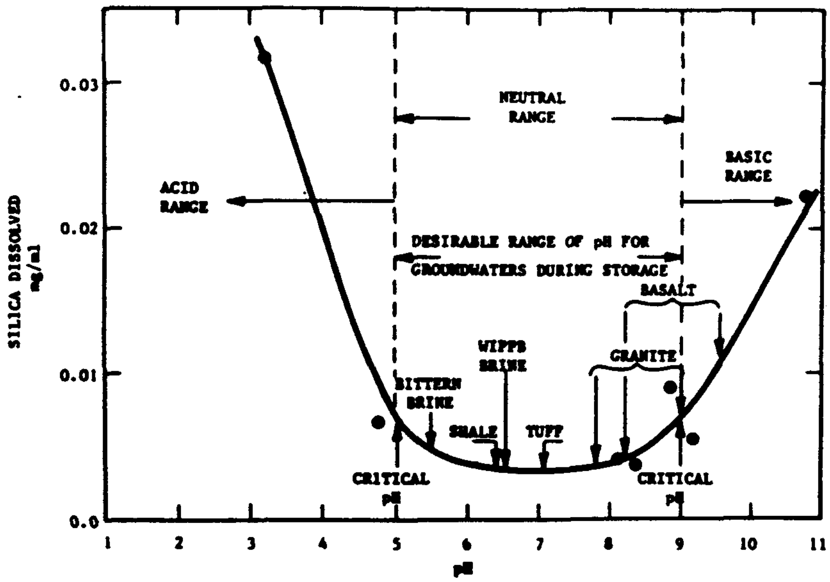
EXPERIMENTAL LEACH FACTORS STUDIED AT SRL

LEACHATE EFFECTS	GLASS EFFECTS	SYSTEMS EFFECTS
TEMPERATURE	SURFACE FINISH	STEEL
pH	SURFACE AREA	IRON
Eh	RADIATION	TEFLON
COMPOSITION	COMPOSITION	ROCK
RADIOLYSIS	CRYSTALLINITY	AIR

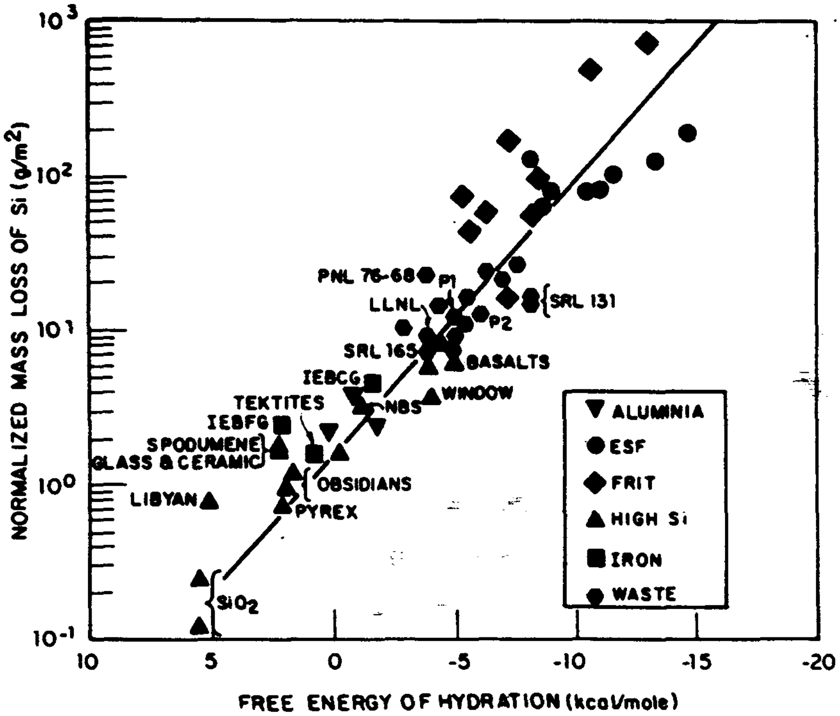
GENERAL PROCEDURES FOR MCC-1 LEACH TEST

- GLASS MONOLITH - KNOWN SURFACE AREA
- REPRODUCIBLE SURFACE FINISH ON MONOLITH
- LEACH IN DI WATER IN TEFLON VESSELS AT 90°C
- LEACH TIMES = 3,7,14,28, ---DAYS
- ANALYZE ACIDIFIED LEACHATES

EFFECT OF pH ON LEACHING IN MCC-1 TESTS



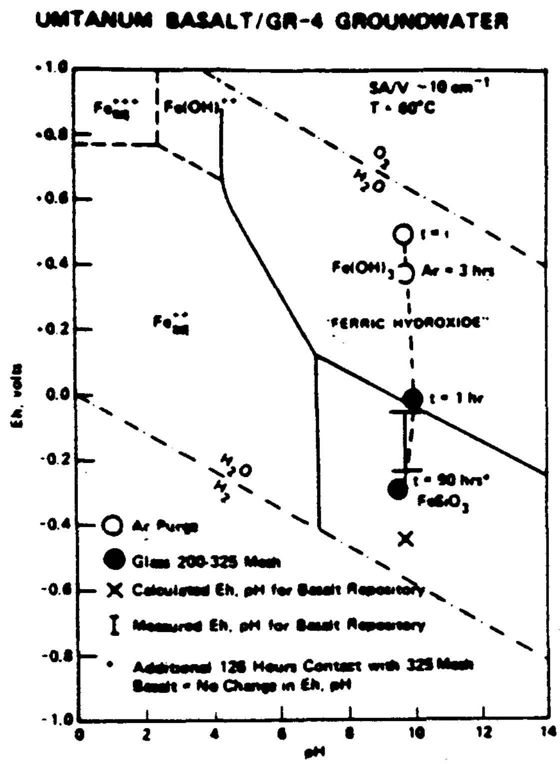
EFFECT OF COMPOSITION ON LEACHING IN MCC-1 TESTS



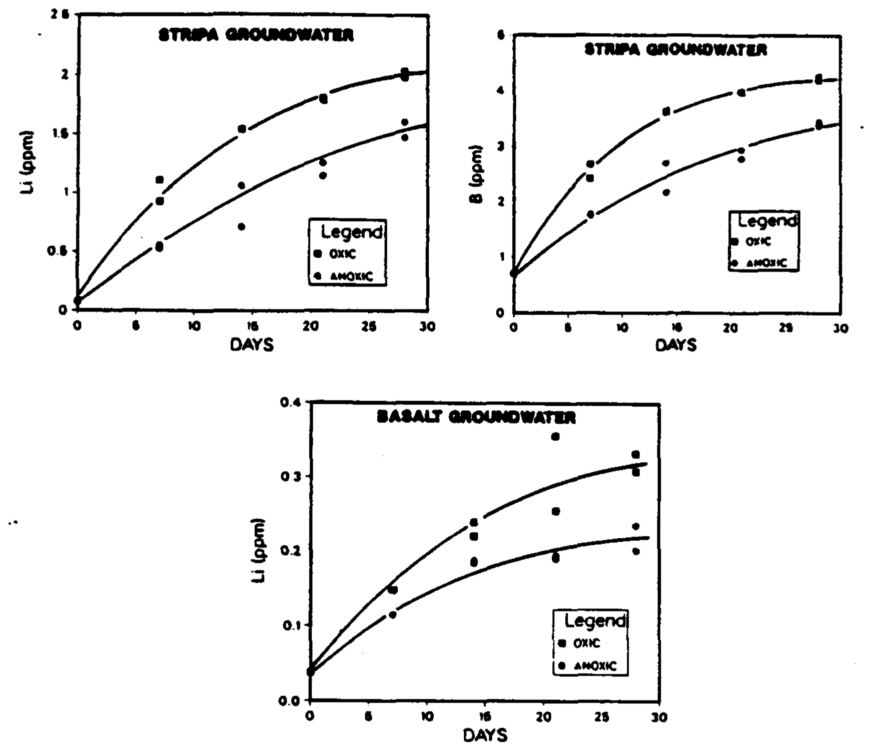
SPECIAL PROCEDURES FOR LOW Eh TESTS

- CAREFUL EXCLUSION OF OXYGEN
- INERT ATMOSPHERE GLOVEBOX
- AIR TIGHT LEACH CONTAINERS
- SOURCE OF REDUCING AGENT IN LEACHATE

ATTAINMENT OF LOW Eh IN SIMULATED BASALT GROUNDWATERS

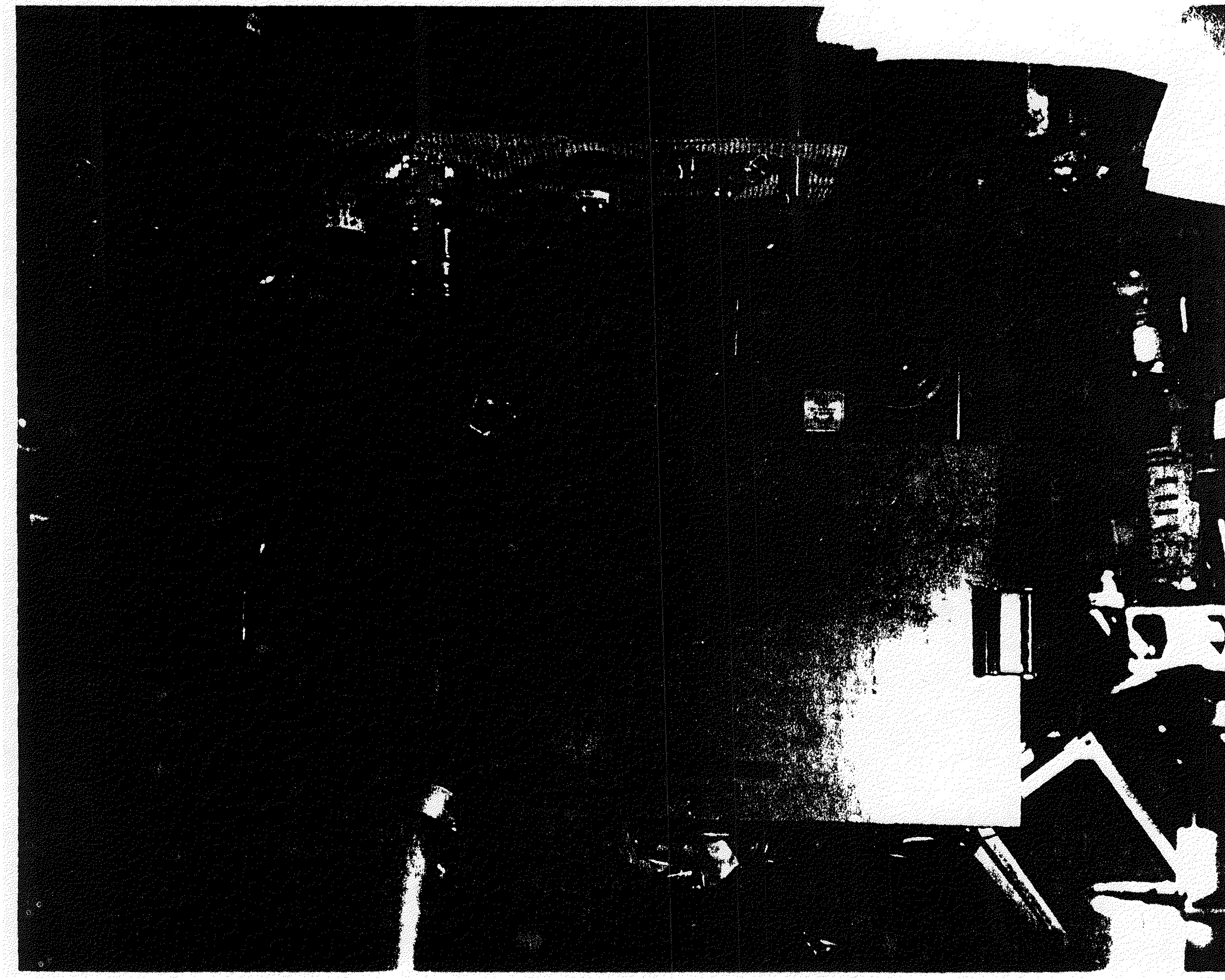


LEACHING Li AND B IN OXIC AND ANOXIC LEACHATES

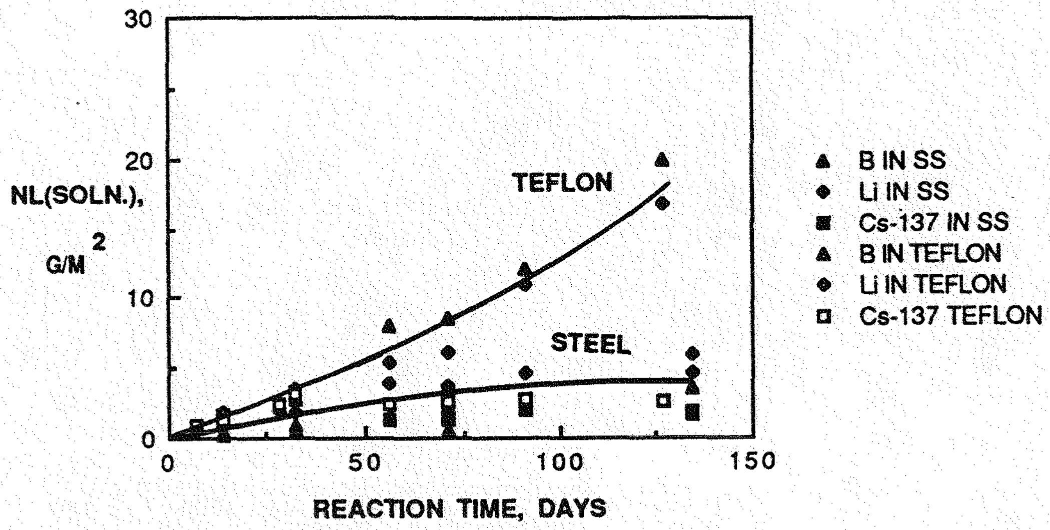


LEACHING Si, B, Na, AND Li FROM ION IRRADIATED NUCLEAR WASTE GLASS IN MODIFIED MCC-1 TESTS

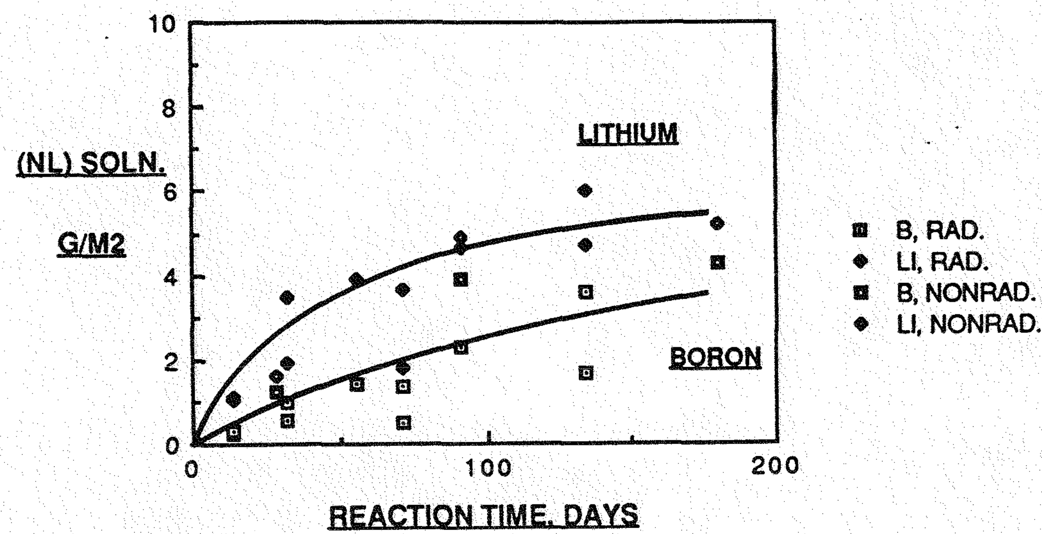
Radiation Dose ions/cm^2	Concentration (ppm) in Leach Test			
	Si	B	Na	Li
0	0.126	0.030	0.183	0.028
0	0.110	0.031	0.109	0.025
5×10^{11}	0.087	0.029	0.204	0.032
10^{12}	0.032	0.023	0.124	0.026
5×10^{12}	0.079	0.026	0.158	0.028
10^{13}	0.127	0.033	0.167	0.032
5×10^{13}	0.109	0.029	0.210	0.029
10^{14}	0.046	0.030	0.171	0.026
10^{15}	0.162	0.042	0.187	0.037
10^{16}	0.187	0.039	0.221	0.041



LEACHING B, Li, AND Cs-137 FROM RADIOACTIVE SRP GLASS IN TEFLON AND STEEL VESSELS

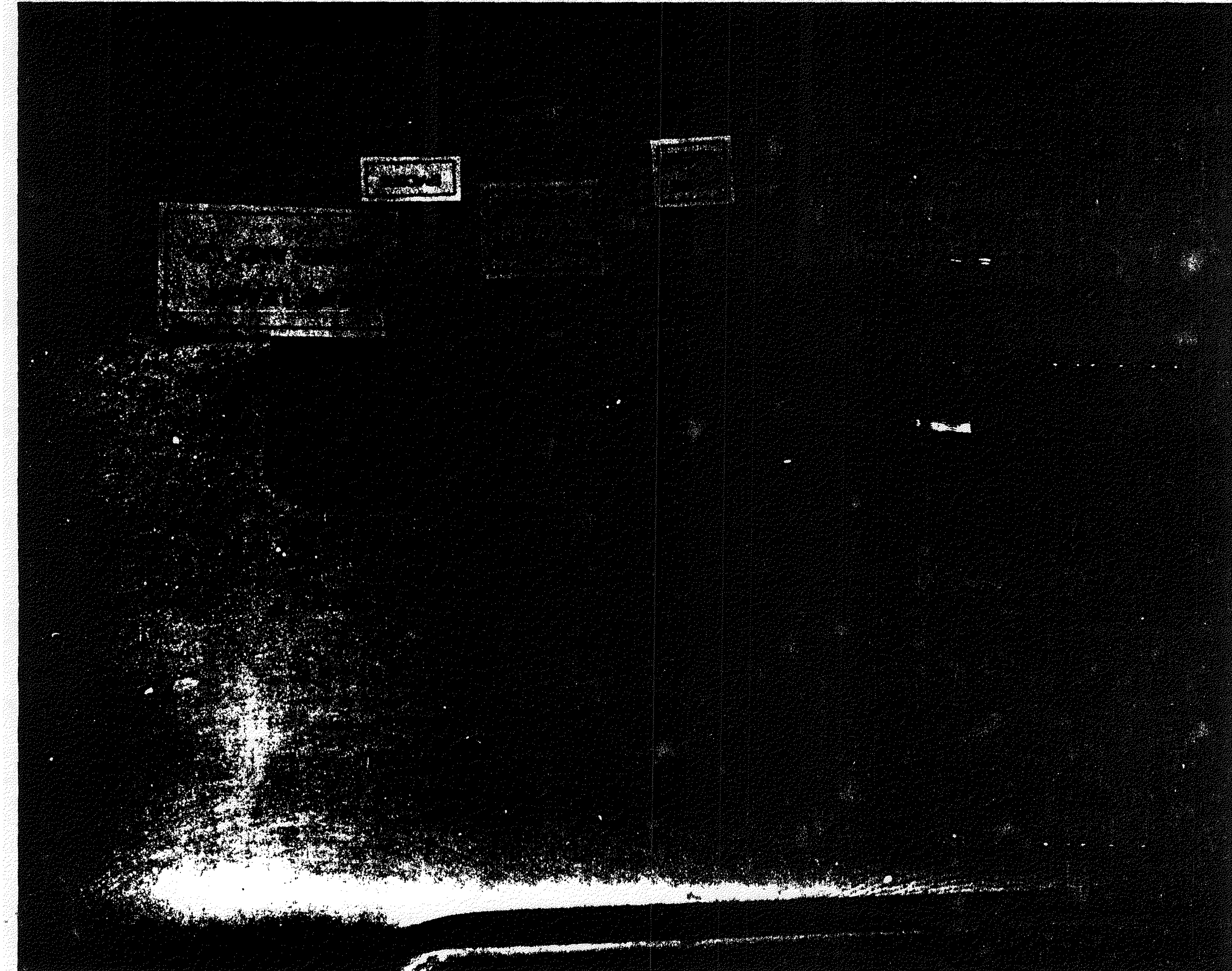


LEACHING B AND Li from RADIOACTIVE AND NONRADIOACTIVE SRP GLASS

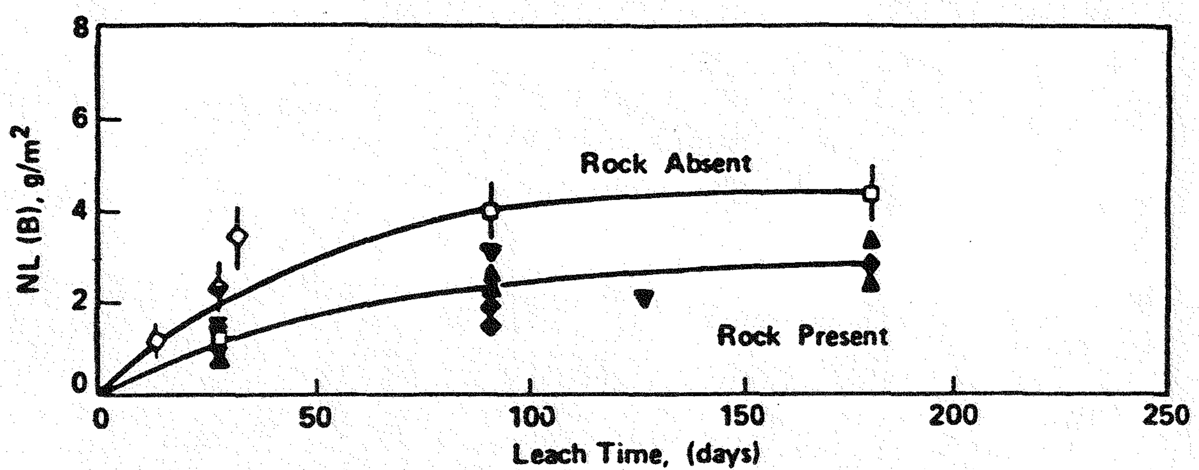


COMPARISON OF Pu-238 IN SOLUTION AND TOTAL LEACHED FROM RADIOACTIVE SRP GLASS

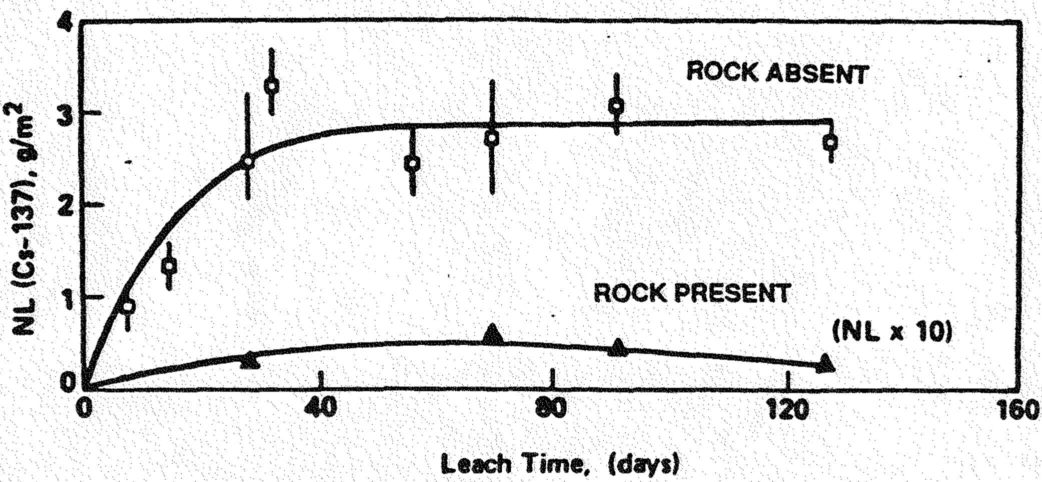
Pu-238			
1st Test		2nd Test	
Soln.	Total	Soln.	Total
.0047	.39	.012	.34
.0077	.31	.029	.30
.0047	.35	.0049	.31
.00034	.25	.0003	.31
.019	.69	--	--
.009	.22	--	--



LEACHING B IN SATURATED TUFF REPOSITORY TESTS



LEACHING Cs-137 IN SATURATED TUFF REPOSITORY TESTS



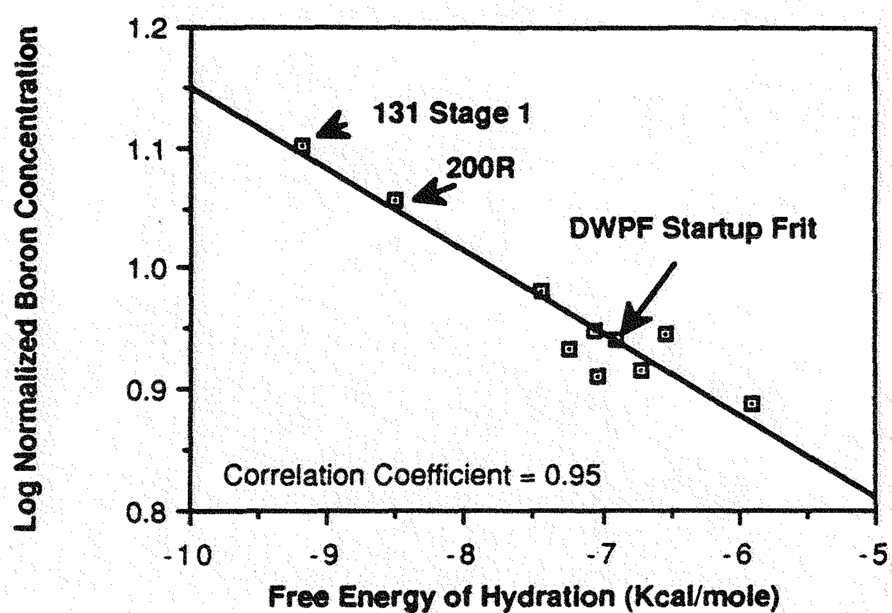
GENERAL PROCEDURES FOR PCT LEACH TEST

- CRUSHED, SIEVED 100-200 MESH GLASS
- GLASS WASHED TO REMOVE FINES
- LEACH IN DI WATER IN SS OR TEFLON VESSELS AT 90°C
- LEACH TIME = 7 DAYS
- ANALYZE FILTERED, ACIDIFIED LEACHATES

LEACHING B AND Li FROM GAMMA IRRADIATED SRP GLASS USING THE PCT CRUSHED GLASS TEST

Dose (gray) and Test No	Concentrations (ppm) in Leach Solutions	
	B	Li
Unirradiated		
Test 1	15.2	17.7
Test 2	15.2	17.7
Test 3	15.4	18.1
4.0×10^6		
Test 1	14.6	17.3
Test 2	15.0	17.8
Test 3	14.3	17.0
4.7×10^7		
Test 1	15.1	18.3
Test 2	15.2	18.4
Test 3	15.1	18.4
3.1×10^8		
Test 1	15.6	18.5
Test 2	15.0	18.1
Test 3	15.6	18.5

EFFECT OF GLASS COMPOSITION ON THE B RELEASE IN THE PCT FOR SRP GLASSES



CONCLUSIONS

TEST DEVELOPMENT

- CAREFUL DEFINITION OF PURPOSE AND PROCEDURES

EXPERIMENTAL RESULTS

- RADIOACTIVE AND NONRADIOACTIVE GLASS LEACH SIMILARLY
- GAMMA OR ION IRRADIATION DOESN'T AFFECT LEACHABILITY
- NO EFFECT OF TUFF ROCK