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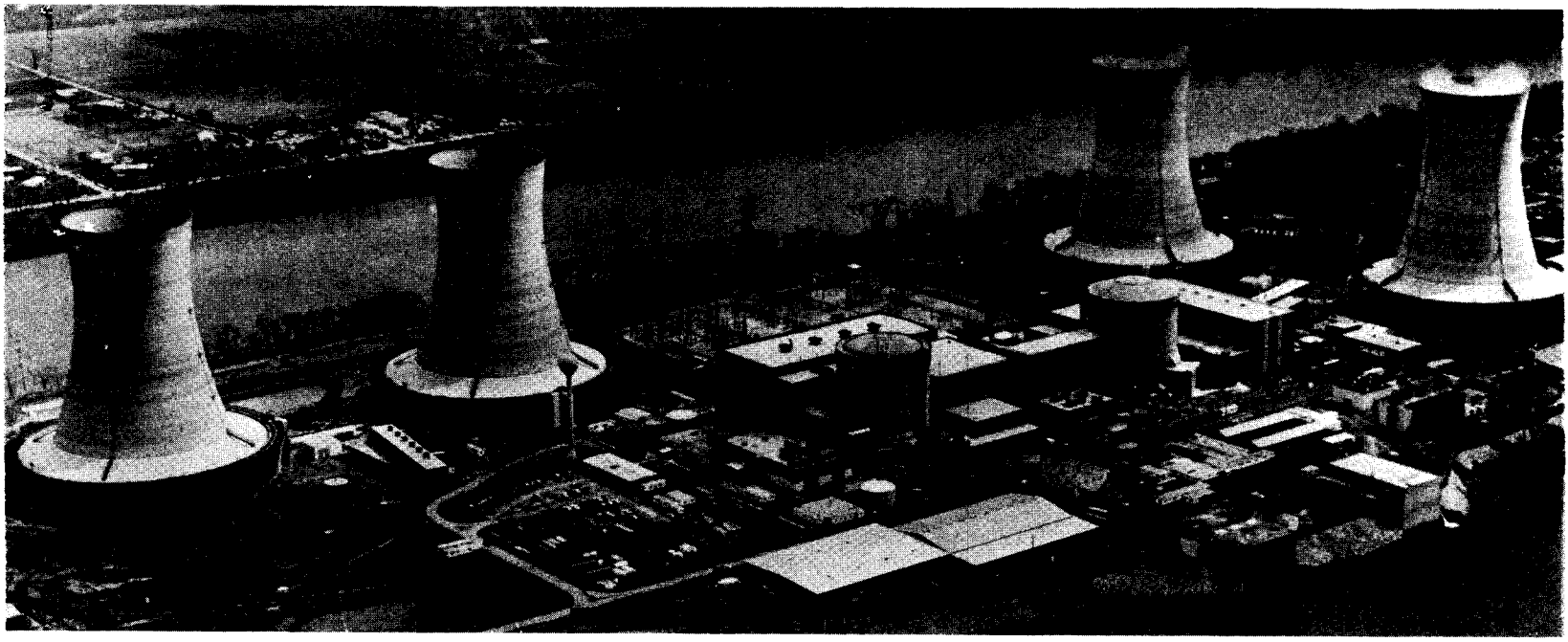

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ZEOLITE VITRIFICATION DEMONSTRATION PROGRAM: CHARACTERIZATION OF RADIOACTIVE VITRIFIED ZEOLITE MATERIALS

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Prepared for the
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Three Mile Island Operations Office
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Pacific Northwest Laboratory
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ABSTRACT

The leach behavior of radioactive vitrified zeolite material was studied as part of the Three Mile Island (TMI) Zeolite Vitrification Program conducted by Pacific Northwest Laboratory (PNL). Experimental procedures, test results, and discussions of the results are presented. The leach behavior of material from three canisters of vitrified zeolite is discussed in terms of the normalized weight loss of the glass-formers and the normalized activity loss of the fission products cesium and strontium. The leach behavior of the radioactive vitrified zeolite material is also compared to the leach behavior of MCC 76-68 reference glass. The effects of changes in the surface microstructure of the vitrified zeolite that occurred during leaching are also presented.

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SUMMARY

Leach behavior and microstructure of radioactive vitrified zeolite material were studied as part of the Three Mile Island (TMI) Zeolite Vitrification Demonstration Program conducted by Pacific Northwest Laboratory (PNL). The leach tests were conducted in deionized, deaerated water according to the Materials Characterization Center (MCC)-1P Static Leach Test Method. Specimens were tested to compare the leach behavior of the radioactive vitrified zeolite contained in three separate canisters with that of the nonradioactive zeolite tested previously, and with an MCC reference glass. The following conclusions were reached:

- The normalized mass loss of cesium and of the glass-former elements from the radioactive vitrified zeolite after the 28-day leach test is significantly lower (factor of 2 or more) than that from the nonradioactive reference glass MCC 76-68.
- The average leach rate for the radioactive vitrified zeolite is slightly lower than the rate for the nonradioactive vitrified zeolite, for all elements reported and all leach test periods longer than seven days.
- The leach behavior of radioactive vitrified zeolite from all three canisters is essentially the same.
- There is no significant systematic difference in leach behavior relative to location of radioactive vitrified zeolite (top, middle, or bottom) in the canisters tested.
- Microstructure of the radioactive material was consistent with that observed in the nonradioactive material, and no second phase components were detected.

INTRODUCTION

The cleanup of the high-activity water at Three Mile Island (TMI) by sorption onto a blend of zeolites provided an opportunity to further develop radioactive waste management technology. Under the Zeolite Vitrification Demonstration Program (ZVDP), the U.S. Department of Energy (DOE) authorized the Pacific Northwest Laboratory (PNL)[a] to take a portion of the zeolites, demonstrate the vitrification of the zeolites on a full scale basis, and determine the dissolution (leach) characteristics of the resultant glass. One low-activity and two high-activity liners of zeolite have been vitrified. These liners were processed in the Submerged Demineralizer System (SDS) that was used to clean cesium and strontium from the TMI water.[1] The characteristics of nonradioactive glass that simulated the vitrified zeolite have been reported.[2] This report describes the dissolution characteristics of specimens of the high-activity vitrified zeolite, and also compares these characteristics with those of the nonradioactive, simulated vitrified zeolite. These dissolution tests on the radioactive glasses were carried out using the Materials Characterization Center (MCC) procedure "MCC-1P Static Leach Test Method", dated December 30, 1982.[3] Because in-cell equipment was lacking, measurements of gas generation, tensile strength, impact strength, and canister corrosion were not conducted. A summary of the tests conducted on the radioactive vitrified zeolite is listed in Table 1.

a. Operated by Battelle Memorial Institute.

TABLE 1. MCC CHARACTERIZATION TESTS ON RADIOACTIVE VITRIFIED ZEOLITE

Test	Sampling Location[a]		
	Canister Z2	Canister Z4	Canister Z5
Chemical analysis on solid product	M	M	M
Phase characterization	M	M	M
Density (geometric)	Averaged	Averaged	Averaged
MCC-1P Static Leach Test Method	T, M, B	T, M, B	T, M, B

a. T, M, and B represent specimens taken from cores located near the top, middle, and bottom of the canisters, respectively.

EXPERIMENTAL PROCEDURE

The objectives of this study were to determine and compare the dissolution (leach) characteristics of radioactive vitrified zeolite to those of a nonradioactive reference vitrified zeolite and to the reference MCC 76-68 glass. The following sections describe the test materials, test equipment, analytical techniques, and leachant. A description of the testing equipment is also contained in Appendix A.

Test Materials

The materials that were tested, the methods for their preparation, and the testing matrix and conditions are described in the following subsections.

Description of Materials

The primary materials that were leach-tested in this study were vitrified zeolites. The zeolites were originally used to remove cesium and strontium from the radioactive water in the TMI-2 Reactor Building basement. Liners containing the zeolites were shipped to PNL, dried, and vitrified to demonstrate the capability of providing a waste material suitable for disposal.[1] The sources of the zeolites for this study were the two high-activity SDS liners, numbers D10012 and D10016. The material from these liners was used to fill three 20.3-cm-diameter, 2.6-m-long, Schedule 40 Type 304L stainless steel canisters--Z2, Z4, and Z5 (Table 2). Samples were taken at three locations along the length of each canister using a 12.7-mm inside diameter (ID) diamond core drill and distilled water as a coolant/lubricant. Cores were taken near the top, midplane, and bottom of each canister in order to identify any differences in leach characteristics that might be attributable to segregation during the vitrification process. Some cores were nearly intact, while others were composed of several pieces. An example of the cores is shown in Figure 1.

In addition to the primary vitrified zeolite specimens used in the study, MCC reference glass [MCC 76-68 (Bar 303)], and the nonradioactive simulated

TABLE 2. DESCRIPTIONS OF CANISTERS USED FOR CORE/SPECIMEN PREPARATION

Canister Number	Canister Fill Height (m)	Amount of Glass (kg)	Zeolite Source
Z2	2.0	167	Entire canister filled with material from SDS liner D10012
Z4	2.0	164	Lower two-thirds of canister filled with material from SDS liner D10012; upper one-third of canister filled with material from SDS liner D10016
Z5	1.9	161	Entire canister filled with material from SDS liner D10016

vitrified zeolite glass, ZVDP-4, were tested to permit comparison of the results of this study with results from other leach studies.

Test Matrix and Testing Conditions

The basis for determing the test matrix for the material from the three canisters of vitrified zeolite was the MCC-1P Static Leach Test Method, Matrix B, December 30, 1982.[3] This method/matrix was developed by the MCC to permit a preliminary comparison of the chemical durability between different waste forms. The method permits initial ranking of different waste forms leached in water by testing for periods up to 28 days at 90°C. It specifies triplicate tests at 28 days and single tests at the lower testing periods. Because of limited in-cell testing space, it was not possible to complete a MCC-1P, Matrix B for each canister-level combination. Therefore, a MCC-1P, Matrix B (except for one 28-day test), was specified only for the cores taken at mid-length from each of the three canisters (Table 3). Modified matrices specified for the cores at other elevations permitted a general comparison between core locations. Blank samples (using only the water leachant), reference MCC 76-68 glass, and nonradioactive simulated vitrified zeolite from earlier studies (ZVDP-4) were included in the matrix in order to permit:

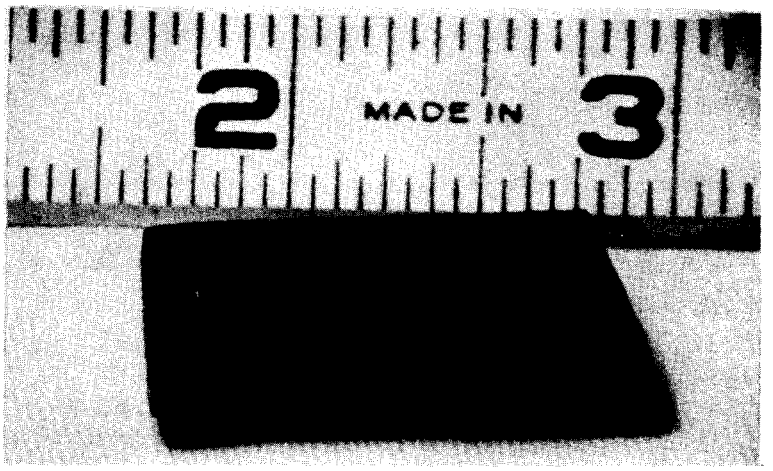


Figure 1. Example of core from one-foot level of canister Z2.

(a) corrections to the data for dissolution of elements from the leach container, and (b) comparison to other leach results, including the earlier non-radioactive vitrified zeolite material.[2]

Specimen Preparation

The sample cores were sliced into leach specimens using a diamond blade and distilled water as a coolant/lubricant. Canisters Z2 and Z4 yielded specimens that had very few gas-bubble holes, although the specimens from the upper level from canister Z2 were very friable. The specimens from Canister Z5 contained several gas-bubble holes. The specimens from the upper (5-foot) level contained the largest holes. Examples of the typical specimens and those with a moderate amount of bubbles are shown in Figure 2. Dimensions, surface areas, weights, and a description of the specimens are listed in Tables 4, 5 and 6 for canisters Z2, Z4, and Z5, respectively. All specimens that were taken from the cores are listed in the tables, even though they were not all used in this study. Because the specimens must be weighed immediately before and after leach testing, the weights of non-used specimens are not included in the tables. The non-used specimens remain available for further tests or experiments if desired.

The reference specimens were prepared in a similar manner and are described in Table 7.

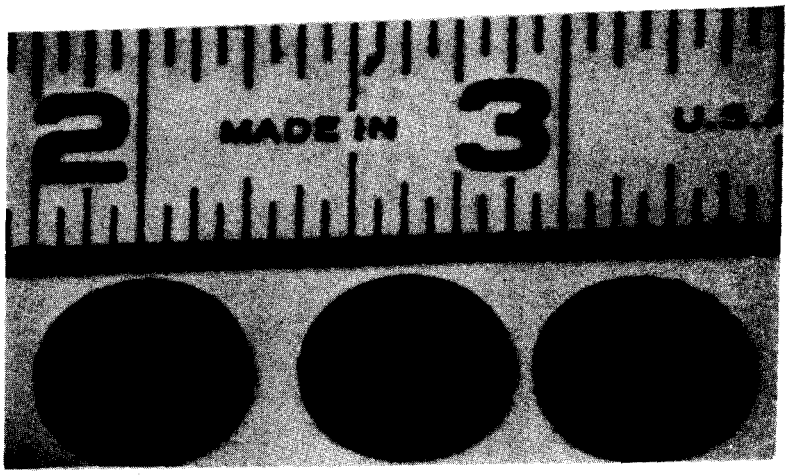
TABLE 3. VITRIFIED ZEOLITE LEACH-TEST MATRIX

Specimen Source[a]	Specimen Identifications			
	3-Day	7-Day	14-Day	28-Day
Midplane and Reference[b]				
Z2-3	2(3)	15a(14)	8(14)	1,2,3(28)
Z4-3	1(3)	12a(14)	2(14)	6,15(28)
Z5-3	SH2M3	16a(14)	10(14)	7,8,9(28)
Blank	5,8(3)	--	5,14(14)	5,14(28)
76-68	6(3)	--	4(14)	4(28)
ZVDP-4	4(3)	--	13(14)	13(28)
Canister Top and Bottom[c]				
Z5-1	9(3)	17b(14)	11(14)	18(28)
Z5-5	7(3)	18a(14)	6(14)	17(28)
Z2-1	--	16b(14)	9(14)	16(28)
Z2-5	--	17a(14)	7(14)	12(28)
Z4-1	--	15b(14)	3(14)	11(28)
Z4-5	--	12b(14)	1(14)	10(28)

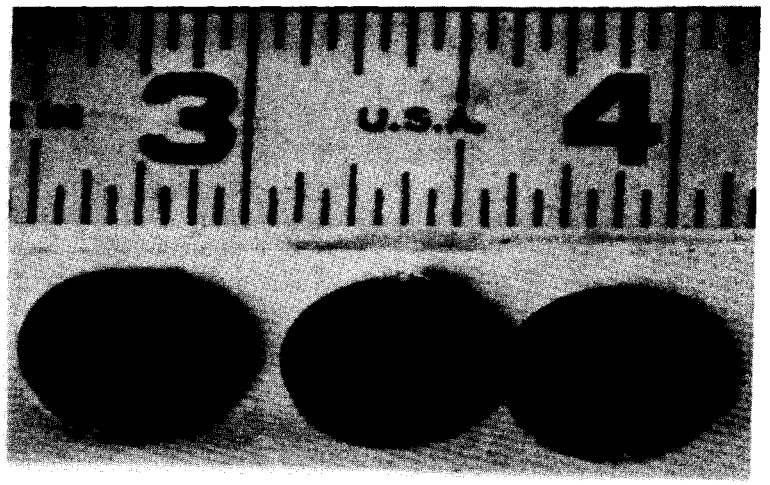
a. Canister-core identification: ZX-Y, X = canister number, Y = core location in feet from bottom of canister. Blank = deaerated water only. 76-68 = MCC 76-68 reference glass. ZVDP-4 = nonradioactive simulated vitrified zeolite.

b. Matrix is MCC-1P, Matrix B; i.e., 28-day, 90°C matrix with one sample each at 3, 7, and 14 days and triplicate samples at 28 days. One 28-day sample was omitted from canister Z4-3 because of limited test space.

c. MCC-1P, Matrix B was modified by conducting single 28-day test and omitting the 3-day specimens from some cores.



CL2 CL1 6(28) Canister Z4



Extra SH2T3 SH2T2 Canister Z5

Figure 2. Example of specimens with bubble holes from canister Z5 (5-foot level) and without holes from canister Z4 (3-foot level).

TABLE 4. DESCRIPTION OF SPECIMENS FROM CANISTER Z2

Specimen Number[a]	Core Location[b]	Distance from Canister Surface (mm)[c]	Dimensions (mm) Thickness	Dimensions (mm) Diameter	Surface Area (mm^2)[d]	Weight (g)	Condition
9(14)	Z2-1	8	1.23	12.88	310.4	0.4144	No bubbles
16(28)		9.5	1.18	12.88	308.3	0.3871	No bubbles
16b(14)		11.0	1.22	12.88	294.5[e]	0.3573	No bubbles, broken edge
SH1B1		83	1.23	12.75	289.4[e]		No bubbles, broken edge
SH1B2		81.0	1.25	12.75	290.1[e]		No bubbles, broken edge
3(28)	Z2-3 (Core 1)	13	1.13	12.71	298.9	0.3681	No bubbles
2(3)		14.3	1.13	12.71	298.9	0.3831	No bubbles
8(14)		15.8	1.19	12.71	301.3	0.3922	No bubbles
15a(14)		17.3	1.23	12.71	302.9	0.4084	No bubbles
2(28)		18.9	1.11	12.71	298.1	0.3559	No bubbles
1(28)	Z2-3 (Core 2)	6	1.27	12.70	304.0	0.3580	One bubble, ~1 mm diameter
CH11		45	1.18	12.70	300.4		No bubbles
CH12		42.9	1.27	12.70	304.0		One bubble, ~1 mm diameter
SH1M1		41.4	1.21	12.69	301.2		No bubbles
SH1M2		38.4	1.09	12.69	296.4		One bubble, ~1 mm diameter
SH1M3		36.9	1.28	12.73	305.7		One bubble, ~1 mm diameter
7(14)	Z2-5	70	1.22	12.66	300.3	0.3910	No bubbles
17a(14)		68.3	1.13	12.71	298.9	0.3683	No bubbles
12(28)		66.9	1.13	12.70	298.4	0.3850	No bubbles
SH1T1		65.4	1.19	12.73	302.1		No bubbles
SH1T2		63.8	1.16	12.70	299.6		No bubbles

a. Identification was based on original intent for specimen. X(YY). X = oven position number, YY = 3, 14, or 28-day tests series; X(a or b)(14): a = 1st 7 days of 14-day test period, and b = 2nd 7 days of 14-day test period; CH11 = chemical sample; CH22 = SEM sample; SH = spare specimen.

b. Z2-X: X indicates axial location of core in feet from bottom of canister Z2; e.g., for X = 3, the core was taken from the 3-foot level.

c. First number (without decimal) in a series is an estimate to ±3 mm of the midplane of the specimen with respect to the inner surface of the canister. Numbers with decimal are with respect to first specimen/number in the series.

d. No correction was made for presence of bubbles because estimates of area change indicated that individual specimen areas would change by less than ±2% of a solid disc specimen.

e. Area adjusted to 95% of solid disc because of chip off side.

TABLE 5. DESCRIPTION OF SPECIMENS FROM CANISTER Z4

Specimen Number[a]	Core Location[b]	Distance from Canister Surface (mm)[c]	Dimensions (mm) Thickness	Dimensions (mm) Diameter	Surface Area (mm^2)[d]	Weight (g)	Condition
3(14)	Z4-1	25	1.25	12.71	303.7	0.4140	No bubbles
15b(14)		28.6	1.21	12.68	300.8	0.4055	No bubbles
11(28)		30.1	1.12	12.68	297.2	0.3699	No bubbles
SLB1		33.1	1.18	12.70	330.4		No bubbles
SLB2		34.7	1.17	12.64	292.4		No bubbles
6(28)	Z4-3	13	1.13	12.79	298.0	0.3742	No bubbles
CL1		38	1.19	12.69	300.4		No bubbles
CL2		41.2	1.25	12.75	305.4		No bubbles
12a(14)		44.4	1.25	12.74	305.0	0.4143	No bubbles
15(28)		46.0	1.27	12.74	305.8	0.4220	No bubbles
SLM3		47.6	0.89	12.70	288.9		No bubbles
SLM1		76	1.13	12.73	299.7		No bubbles
SLM2		74.7	1.14	12.70	298.8		No bubbles
2(14)		73.2	1.25	12.71	303.7	0.4123	No bubbles
1(3)		71.7	1.12	12.73	299.3	0.3738	No bubbles
1(14)	Z4-5	32	1.16	12.74	301.4	0.3769	No bubbles
12b(14)		33.2	1.26	12.78	307.1	0.4016	No bubbles
10(28)		34.8	1.16	12.78	303.1	0.4176	No bubbles
SLT1		36.3	1.11	12.76	300.3		No bubbles
SLT2		37.9	1.23	12.78	305.9		No bubbles

a. Identification was based on original intent for specimen. X(YY): X = oven position number, YY = 3, 14, or 28-day test series; X(a or b)(14): a = 1st 7 days of 14-day test period, and b = 2nd 7 days of 14-day test period; CL1 = chemical sample; CL2 = SEM sample; SL = spare specimen.

b. Z4-X: X indicates axial location of core in feet from bottom of canister Z4.

c. First number (without decimal) in a series is an estimate to ±3 mm of the midplane of the specimen with respect to the inner surface of the canister. Numbers with decimals are with respect to the first specimen/number in the series.

d. No correction was made for presence of bubbles because estimates of area change indicated that individual specimen areas would change by less than ±2% of a solid disc specimen.

TABLE 6. DESCRIPTION OF SPECIMENS FROM CANISTER Z5

Specimen Number[a]	Core Location[b]	Distance from Canister Surface (mm)[c]	Dimensions (mm) Thickness	Dimensions (mm) Diameter	Surface Area (mm^2)[d]	Weight (g)	Condition[e]
9(3)	Z5-1	60	1.22	12.73	303.3	0.3948	A few bubbles to 1.5 mm diameter
17b(914)		61.9	1.18	12.73	301.7	0.3714	A few bubbles to 1 mm diameter
11(14)		63.4	1.16	12.73	300.9	0.3684	A few bubbles to 1 mm diameter
18(28)		64.9	1.19	12.75	303.0	0.3648	Three bubbles to 3 mm diameter
SH2B1		66.4	1.21	12.80	306.0		Four bubbles to 3 mm diameter
SH2B2		67.9	1.22	12.80	306.4		A few bubbles to 1.5 mm diamete
SH2B3		69.5	1.22	12.75	304.2		A few bubbles to 1 mm diameter
CH21	Z5-3 (Core 1)	51	1.16	11.76	260.1		Four bubbles to 1 mm diameter
CH22		52.3	1.09	11.96	265.6		Three bubbles to 1 mm diameter
SH2M1		53.7	1.18	11.99	270.3		One bubble ~1 mm diameter
SH2M2		55.2	1.30	11.96	273.5		Two bubbles to 1 mm diameter
7(28)	Z5-3 (Core 3)	32	1.16	12.75	301.8	0.3596	No bubbles
3(3)		33.2	1.16	12.75	301.8	Sample lost	One bubble ~1 mm diameter
10(14)		34.7	1.21	12.74	303.4	0.3853	No bubbles
16a(14)		36.3	1.27	12.75	306.2	0.4063	One bubble ~1 mm diameter
8(28)		37.9	1.21	12.73	302.9	0.3878	No bubbles
SH2M3		39.4	1.19	12.75	303.0	0.3847	One bubble ~1 mm diameter
9(28)		42.5	1.24	12.75	305.0	0.4071	No bubbles
7(3)	Z5-5	19	1.17	12.71	300.5	0.3827	Four bubbles to 1.5 mm diameter
6(14)		23.8	1.17	12.73	301.3	0.3647	A few bubbles to 1.5 mm diamete
18a(14)		25.3	1.17	12.70	300.0	0.3725	A few bubbles to 1 mm diameter
17(28)		26.8	1.17	12.70	300.0	0.3695	A few bubbles to 1 mm diameter
SH2T1		28.2	1.14	12.70	298.8		A few bubbles to 1 mm diameter
SH2T2		29.7	1.17	12.73	301.3		A few bubbles to 1 mm diameter
SH2T3		31.2	1.17	12.70	300.0		A few bubbles to 1 mm diameter

a. Identification was based on original intent for the specimen. X(YY): X = oven position number, YY = 3, 14, or 28-day test series; X(a or b)(14): a = 1st 7 days of 14-day test period, and b = 2nd 7 days of 14-day test period; CH21 = chemical sample; CH22 = SEM sample; SH = spare specimen.

b. Z5-X: X indicates axial location of core in feet from bottom of canister Z5.

c. First number (without decimal) in a series is an estimate to ±3 mm of the midplane of the specimen with respect to the inne surface of the canister. Numbers with decimal are with respect to the first specimen/number in the series.

d. No correction made for presence of bubbles.

e. "A few" means greater than 4 but less than 10.

TABLE 7. DESCRIPTION OF NONRADIOACTIVE REFERENCE SPECIMENS

Specimen Number	Standard Identification	Dimensions (mm) Thickness	Diameter		Surface Area (mm^2)	Weight (g)[a]
4(3)	ZVDP-4[b]	1.20	12.90 to 12.95		311.1	0.4035 (0.4047)
13(14)	ZVDP-4	1.21	12.90 to 12.95		311.5	0.4098 (0.4102)
13(28)	ZVDP-4	1.22	12.90 to 12.95		312.0	0.4100 (0.4105)
ZVDP-Extra	ZVDP-4	1.19	12.90 to 12.95		310.7	(0.4044)
		Width	Height	Length		
6(3)	76-68-303[c]	10.06	9.98	2.52	301.8	0.7209 (0.7218)
4(14)	76-68-303	10.08	10.00	2.55	304.0	0.7345 (0.7349)
4(28)	76-68-303	10.10	10.08	2.53	305.7	0.7276 (0.7279)
76-68-Extra	76-68-303	9.99	9.99	2.50	299.5	(0.7202)

a. The first weight listed is as-measured in the hot cell and was used for calculating weight loss during the experiments. The second weight was measured in the laboratory and is listed to show the accuracy of the in-cell instrument even though it is subject to degradation in the radioactive field.
b. Reference material is nonradioactive simulated vitrified zeolite ZVDP-4.
c. Reference material is MCC 76-68 glass from Bar 303.

Testing Equipment

The expected degradation of standard Teflon®[a] containers in the radiation environment of the hot cell necessitated the design of quartz leach containers. The geometric restrictions of transfers into the hot cells required small special ovens. This specialized equipment is described below. The standard equipment used during the testing is listed in Appendix A.

Environmental Chambers. Two specially designed ovens were built for use in the hot-cell leach tests. Each oven consisted of a stack of aluminum plates with nine cavities for specimen containers. Each cavity had a lead liner to increase the heat capacity of the oven. Oven heating was provided with two cartridge-type heaters located in the bottom aluminum plate. The aluminum stack was 25 x 25 x 10 cm with asbestos insulation on the sides, top, and bottom. A stainless steel shell enclosed the oven. Inlet and outlet ports were provided to permit the flow of argon through the specimen cavities during the tests. The temperature of each oven body was measured with three chromel-alumel thermocouples and was controlled with one iron-constantan thermocouple. Located out-of-cell were two LOVE controllers, one for each oven, to maintain constant oven temperature, and a Fluke data logger which recorded the oven temperatures on paper tape at a selected frequency. The testing arrangement for the hot-cell leach tests is shown in Figure 3.

During hot-cell leach testing, the oven body temperature was measured and controlled to ensure that specimen cavity temperatures were maintained at 90 ±2°C. The oven calibration procedure and data for ensuring that the specimen cavity temperatures were within the specified limits are presented in Appendix B.

a. ®Teflon is a registered trademark of E. I. du Pont de Nemours and Company.

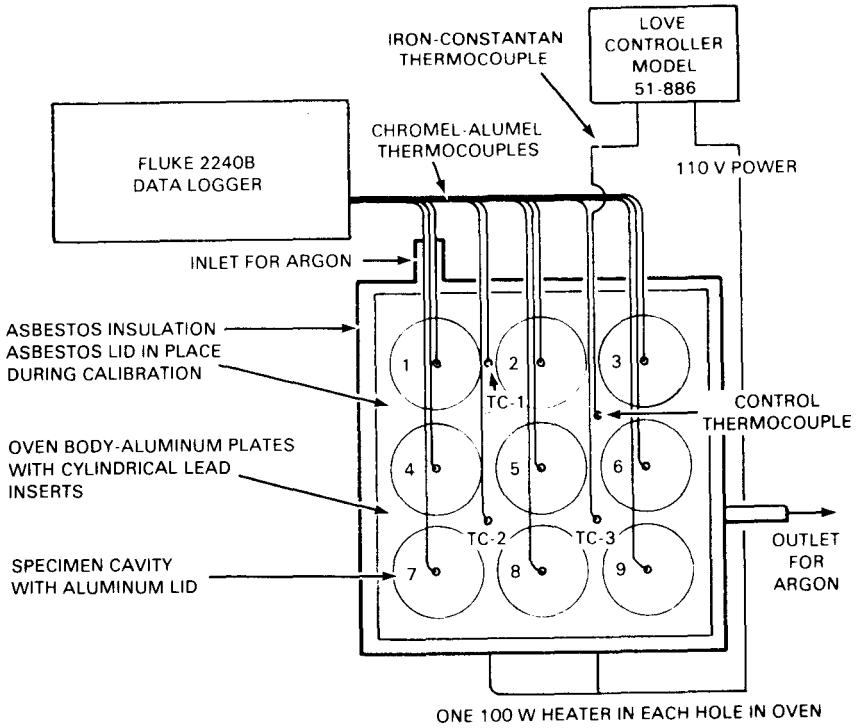


Figure 3. Ovens for leach tests.

Leach Container. The hot-cell leach containers were mated pairs of clear fused quartz standard inner and outer taper joints, size 40/50. The outer joint (i.e., top of the leach container) was fused at the top, the inner joint (i.e., the bottom of the leach container) was fused at the bottom. An hour-glass shape holder also made from quartz was used to position the specimen in the leach container. The leach container was sealed by wrapping a strip of Para Film "M"®[a] approximately 6 mm wide around the bottom of the container and permitting the material to soften in the oven after mating the two halves of the leach container.

The design of the container permitted a maximum loss of 9 mL of leachant during at-temperature testing before the specimen became uncovered by leachant.

a. ®Registered trademark of American Can Company, Dixie/Marathon, Greenwich, Connecticut, 06830.

Description of Leachant

The leachant used in all the tests was distilled, deaerated water. The water was deaerated by bubbling argon through the distilled water for 15 minutes at room temperature. One batch of leachant, used in the 14-day tests and some of the 7-day tests, was discovered to have been slightly contaminated with brine during the deareation. Judging from the results, this contamination apparently had no effect upon the dissolution characteristics of the vitrified zeolite; leach test data were blank-corrected appropriately in calculating the sodium elemental mass loss. Because of the difficulty of hot cell experimentation, no attempt was made to purge the 5 to 10 mL of residual air from the specimen containers.

Analytical Techniques

The 26 elements routinely analyzed on radioactive materials by inductively-coupled plasma (ICP) spectrometry (Appendix D) were determined analytically using a lead-shielded Applied Research Laboratories Model 3400 ICP analyzer. The as-cast material specimens were prepared for analysis by fusion in both KOH in a nickel container and Na_2O_2 in a zirconium container at red heat followed by dissolution in distilled water. The results of the analyses were averaged (Table C-1).

The ^{137}Cs concentration in the as-cast material specimen was determined by gamma ray spectrometry on small (~10 mg) pieces of vitrified zeolite and also on the fused and dissolved material. The results of the analyses were averaged (Table C-2). The ^{137}Cs concentration in the leachate was also determined by gamma ray spectrometry, using a standard geometry of 15 mL of leachate in a 25 mL standard polyethylene bottle.

Microstructural examination of radioactive vitrified zeolite specimens, both as-cut from the canister and after a 28-day leach test, was conducted using scanning electron microscopy (SEM). The instrument was a JEOL 35C SEM, specially shielded for MCC use with fully radioactive specimens. The zeolite

specimens were mounted in the hot cell for microscopy, and transferred to the SEM using a tungsten alloy transfer device. In-cell leach-tested nonradioactive vitrified zeolite and MCC 76-68 glass were also examined as reference materials.

TEST RESULTS

The basis for comparison of the leach test results, a summary of the leach results, results of the SEM examinations, and vitrified zeolite densities are presented in the following subsections.

Basis for Comparison

The leach test results are compared in terms of normalized weight loss (i.e., grams of material leached per square meter of specimen surface area) and normalized elemental weight loss. For the nonradioactive elements, the normalized weight loss, $(NL)_i$, was calculated from the equation

$$(NL)_i = \frac{(\text{ppm - blank})(\text{grams leachate})}{(f_i)\ (SA)}$$

where

ppm - blank	=	ICP analysis for element by weight in leachate minus analysis for the appropriate blank
gram leachate	=	final weight of leachate
f_i	=	fraction of element in specimen by weight
SA	=	dimensional surface area of specimen in square meters.

The individual f_i values for the specimen materials are listed in Tables C-1 and C-3. The f_i for elements determined by ICP was determined using the average values for analyses of two specimens prepared by fusion of nonleached glass (Table C-1). The blank analyses are listed with the leach results in Appendix D. Because no blank was run for the 7-day tests, interpolated values from other testing periods were used.

The ^{137}Cs results were calculated from the equation

$$(NL)_i = \frac{(ai)(W_o)}{(a_o)(SA)}$$

where

a_i	=	activity/mL of leachate times the volume of leachate in mL
W_o	=	weight of specimen in grams
a_o	=	activity/mg of control sample times the weight of the specimen in mg
SA	=	dimensional surface area of specimen in square meters.

The f_i values for ^{137}Cs were determined by averaging the counting results of the two ICP fusions and the counting of a solid piece of nonleached glass (Table C-2). The resultant averages for ^{137}Cs, 166 to 190 μCi/mg, compare favorably with that expected to be present based on the initial cesium loading in the TMI liners.

Summary of Results

Summaries of the leach results for the vitrified zeolite and the reference glasses are presented in Tables 8 and 9. For the vitrified zeolite, the leach values for all specimens for each leach period have been averaged, because no significant systematic difference was found between specimens. The individual experimental parameters, weight loss, and pH results for the 3-, 7-, 14-, and 28-day tests are summarized in Tables D-1, -2, -3, and -4, respectively. Individual ICP analytical results and the resultant $(NL)_i$ values for the tests are listed in Tables D-5, -6, -7, and -8. Gamma spectroscopy results for ^{137}Cs for the individual tests are listed in Table D-9. The summary for the radioactive vitrified zeolite (Table 8) represents average values from which questionable data has been omitted.

In some cases, such as sample 5(3) from the 3-day test series, the fused quartz leach container broke during the test. In other cases, such as sample

TABLE 8. SUMMARY OF LEACH RESULTS FOR RADIOACTIVE VITRIFIED ZEOLITE

	Average Leaching Result (g/m^2)							
	3-Day Tests		7-Day Tests		14-Day Tests		28-Day Tests	
Item	$(NL)_i$	Standard Deviation	$(NL)_i$	Standard Deviation	$(NL)_i$	Standard Deviation	$(NL)_i$	Standard Deviation
Weight loss	3.1	1.6	6.7	0.9	5.9	2.0	7.0	0.6
Al	5.0	0.9	6.8	0.8	7.1	0.5	7.5	1.1
B	5.0	0.8	7.8	0.8	8.4	0.8	9.5	1.2
Ba	--[a]		0.4	0.2	0.5	0.4	1.0	1.0
Ca	2.5	0.4	2.2	0.2	2.3	0.3	1.0	0.7
Ce	--						[12.4][b]	7.2
Cr			[2.5]	0.8	[3.2]	0.7	[5.7]	1.3
Dy							--	
Fe	0.1	--	[0.3]	0.2	7.0	2.5	0.1	0.4
K	5.4	3.2	4.6	1.7			7.2	3.7
La							--	
Li	6.1	0.6	8.2	0.7	8.9	0.5	9.6	1.6
Mg	1.9	0.3	1.4	0.6	0.6	0.6	--	
Mn							--	
Mo							--	
Na	4.3	0.7	5.8	1.1	5.8	0.7	6.9	1.1
Nd	--						[21.0]	8.8
Ni							[9.0]	4.6
Rh	--						[20.9]	12.6
Ru	--						--	
Si	6.1	0.8	8.6	1.6	9.9	2.0	7.7	2.0
Sr	2.2	0.6	3.2	0.4	3.0	0.5	2.5	0.6
Te	--						--	
Ti							[0.1]	
Zn	--						--	
Zr							--	
^{137}Cs	6.4	1.1	8.6	1.1	9.1	1.1	9.4	1.2

a. Dash (--) indicates that either $(NL)_i$ <0.05, was negative after correction for blank, or analyses of original material was not available to calculate f_i; data omission indicates element not detected.

b. Brackets [ ] indicate that this average pertains only to specimens with determined value; not all specimens indicated a value.

TABLE 9. SUMMARY OF LEACH RESULTS FOR REFERENCE MCC 76-68 AND ZVDP-4 GLASSES

	$(NL)_i$ (g/m^2)					
	MCC 76-68			ZVDP-4		
Item	3-Day Test	14-Day Test	28-Day Test	3-Day Test	14-Day Test	28-Day Test
Weight loss	2.0	6.5	8.8	1.3	7.4	7.1
Al	3.3	0.9	21.1	4.8	9.0	9.4
B	6.5	14.3	0.3	3.5	9.0	10.2
Ba	0.7	0.3	0.4	--[a]	0.4	1.6
Ca	3.4	2.0		1.4	1.5	--
Ce	--			--		
Cr		0.6				9.9
Dy						--
Fe	--	--	0.1	--	--	1.0
K	--			--		--
La	--					--
Li				4.7	9.1	9.7
Mg	5.2	--	--	0.3	--	--
Mn						
Mo	--	--	--		--	--
Na	7.5	13.0	21.8	4.6	7.8	9.5
Nd	--			--		--
Ni						--
Rh				--		--
Ru	--			--		
Si	11.4	24.2	40.2	5.4	10.5	12.6
Sr	3.7	2.4	--		0.9	1.8
Te		--				--
Ti		0.1		--		--
Zn						--
Zr						6.3

a. Dash (--) indicates that either $(NL)_i$ <0.05, was negative after correction for blank, or analyses of original material not available to calculate f_i. Data omission indicates element not detected.

TABLE 10. SUMMARY OF ADEQUACY OF TEST RESULTS

Test Number	Loss of Water (g)	Specimen Weight Change (%)	Normalized Mass Loss $(NL)_{Si}$	Conclusion[a]
1(3)	1.40	0.37	6.8	All OK
2(3)	0.00	0.31	7.4	All OK
3(3)	0.05	0.36	5.7	All OK
4(3)	1.55	0.10	5.4	Chem. OK, weight NG
5(3)	All	--	--	All NG
6(3)	0.35	0.08	11.4	Chem. OK, weight NG
7(3)	0.90	0.08	5.3	Chem. OK, weight NG
8(3) (blank)	+0.05	--	--	All OK
9(3)	+0.05	0.10	5.4	Chem. OK, weight NG
1(14)	0.70	0.53	11.8	All OK
2(14)	0.50	0.51	13.1	All OK
3(14)	0.75	0.70	9.9	All OK
4(14)	0.95	0.27	24.2	All OK
5(14)(blank)	0.30	--	--	All OK
6(14)	23.40	--	--	All NG
7(14)	0.35	0.51	9.4	All OK
8(14)	2.40	0.28	9.8	All OK
9(14)	0.40	0.31	11.3	All OK
10(14)	1.70	0.23	7.0	All OK
11(14)	5.10	0.57	7.2	All OK
12a(14)	0.15	0.56	8.1	All OK
12b(14)	2.30	0.55	8.2	All OK
13(14)	0.05	0.56	10.5	All OK
14(14) (blank)	1.35	--	--	All OK
15a(14)	0.25	0.52	9.2	All OK
15b(14)	0.20	0.47	11.7	All OK
16a(14)	1.60	0.02	0.1	Specimen NG
16b(14)	All	0.20	--	All NG
17a(14)	0.35	0.35	9.0	All OK
17b(14)	0.65	0.43	7.5	All OK
18a(14)	0.85	0.56	6.2	All OK
1(28)	8.00	0.67	7.4	All OK
2(28)	21.30	0.62	6.4	All NG, exposure time unknown
3(28)	All	0.08	--	All NG
4(28)	0.35	0.37	40.2	All OK
5(28) (blank)	+0.05	--	--	All OK
6(28)	0.65	0.67	5.3	All OK
7(28)	All	0.25	--	All NG
8(28)	2.00	0.08	9.7	Chem. OK, weight NG

TABLE 10. (continued)

Test Number	Loss of Water (g)	Specimen Weight Change (%)	Normalized Mass Loss $(NL)_{Si}$	Conclusion
9(28)	18.60	0.52	7.7	All NG, exposure time unknown
10(28)	0.35	0.53	8.5	All OK
11(28)	1.05	0.49	11.6	All OK
12(28)	3.35	0.52	5.4	All OK
13(28)	1.15	0.54	12.6	All OK
14(28)(blank)	All	--	--	All NG
15(28)	17.40	0.43	5.6	All NG, exposure time unknown
16(28)	2.30	0.54	7.3	All OK
17(28)	All	0.11	--	All NG
18(28)	0.00	0.49	6.6	All OK

a. All OK = all data acceptable.
Chem. OK = chemistry data acceptable.
All NG = all data unacceptable due to leachant loss (for those containers having less than two-thirds the original water, the specimen was exposed at an unknown time).
Weight NG = weight measurement not consistent with chemistry measurements.

15(28) from the 28-day test series, the leach container seal leaked to the extent that the specimen was uncovered at an unknown time during the test. For both of these cases, the leach results were not included in the evaluation of results presented in the next section of this report. Table 10 summarizes the adequacy of the test results based upon water loss, specimen weight change (a clue as to whether the specimen was uncovered during the test), and chemical analysis for silicon in the leachate. In addition to those samples for which the leach time was unknown, a single sample, 16a(14), yielded results significantly different from similar specimens. For this case the leaching was very small compared to other specimens and the results were not included in the results in order to be conservative.

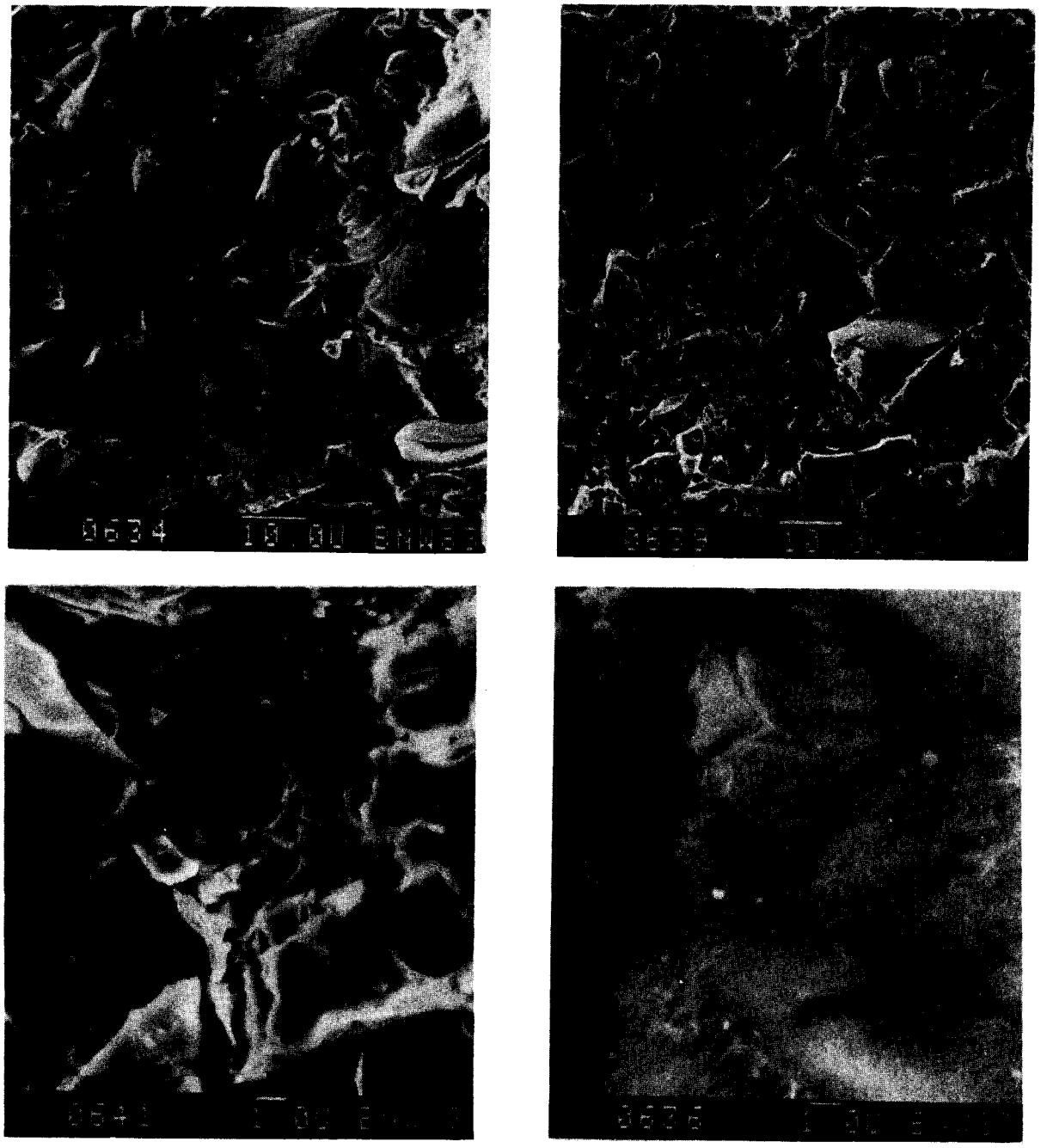
Results from Scanning Electron Microscopy

The microstructure and phase characteristics of radioactive vitrified zeolite specimens from canister Z2 are shown in Figures 4 and 5, both as-cut before leaching (specimen CH12), and after a 28-day leach test (specimen 1-28), respectively. Most of the sharp fracture edges initially present on the raw cut surface have been removed by the leaching action, but otherwise the surface appears to be largely unetched. The leached specimen of non-radioactive vitrified zeolite (Figure 4) is very similar in appearance to the radioactive material after 28 days of leaching, consistent with their very similar leach test analytical results. The MCC 76-68 glass (Figure 5) also shows some edge-rounding effects of leaching, but displays considerably more structural character than do the zeolites. This difference may be indicative of more active leaching for the glass after leaching 28 days, as evidenced by the mass loss data.

No evidence of second phases or component separation was found in the radioactive vitrified zeolite.

Vitrified Zeolite Density

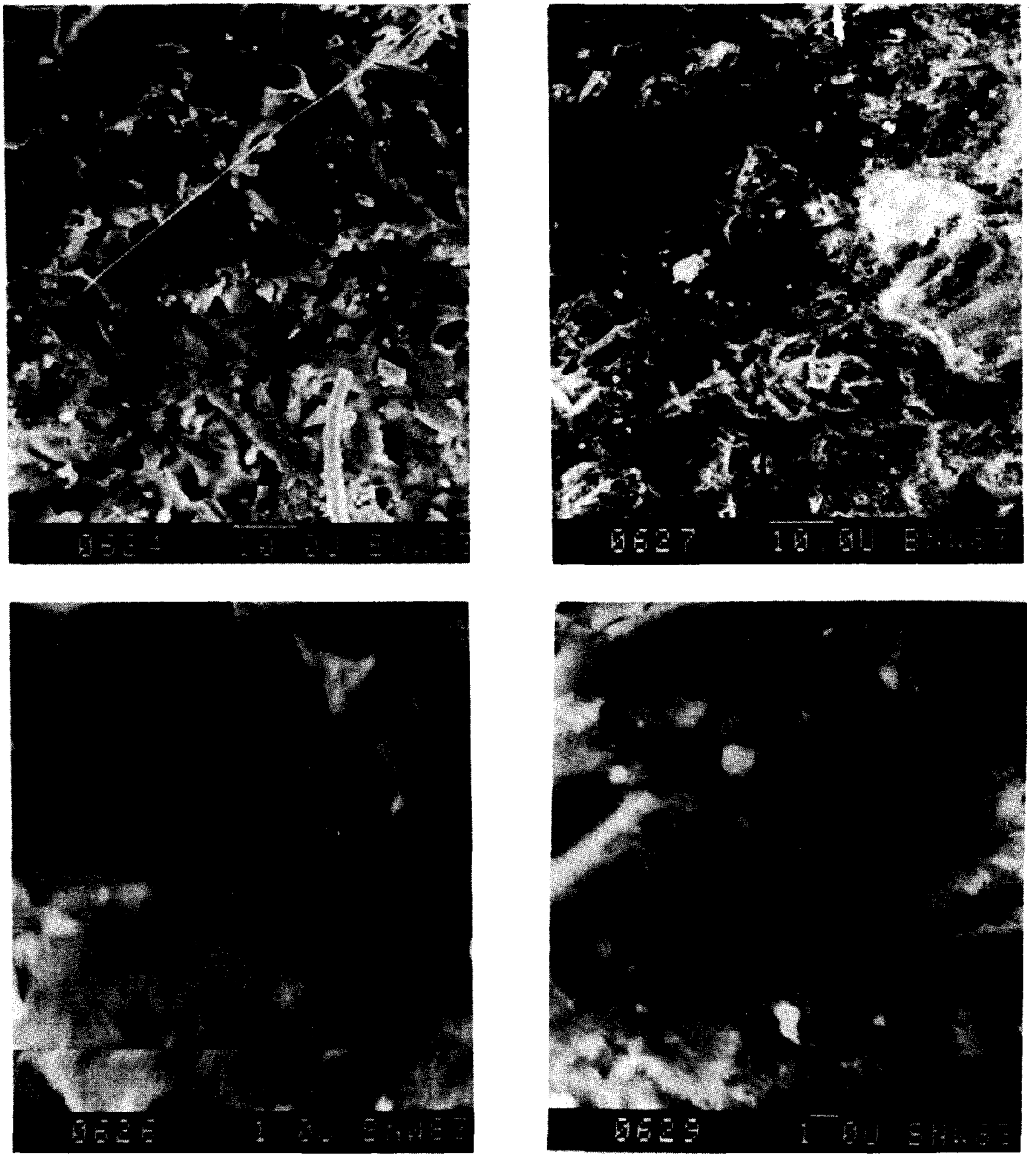
The geometric density of the vitrified zeolite specimens from each of the three canisters is listed in Table 11. The lower density for specimens from



Specimen CH12
(as-cut, before leaching)

Specimen 1(28)
(after 28-day leach test)

Figure 4. Microstructure of radioactive vitrified zeolite specimens from middle level of canister Z2. [Scanning electron micrographs; orginals at 1000X (upper) and 5000X (lower).]



Specimen 13(28)
(vitrified zeolite ZVDP-4)

Specimen 4(28)
(MCC 76-78 glass)

Figure 5. Microstructure of nonradioactive reference specimens after 28-day leach tests in hot cell ovens. [Scanning electron micrographs; originals at 1000X (upper) and 5000X (lower).]

canister Z-5 is probably the result of gas bubbles in the glass (Table 6). The density of the radioactive vitrified zeolite is similar to that of the reference ZVDP-4.[2]

TABLE 11. DENSITY OF SPECIMENS

Canister	Number of Specimens	Density (g/cm^3)	Standard Deviation
Z-2	10	2.61	0.07
Z-4	11	2.61	0.08
Z-5	14	2.50[a]	0.05

a. Geometric density includes contribution of bubbles; no correction was made for presence of bubbles.

DISCUSSION OF RESULTS

The leach results on the materials from the three radioactive vitrified zeolite canisters are compared by level within the canister, between canisters, to each other, and to leach results from nonradioactive glasses MCC 76-68 and ZVDP-4, measured both in- and out-of-cell.

Comparison of Leaching by Level in the Canisters

A comparison of the normalized weight loss of specimens from different levels of the three canisters as a function of the leaching period is shown in Figure 6. Comparisons of the normalized elemental weight loss of the primary glass-formers, silicon, boron, and sodium, with the radioactive fission products strontium and ^{137}Cs for specimens from the different levels of the three canisters are shown in Figures 7, 8, 9, 10, and 11, respectively. The comparisons do not reveal any significant pattern.

Comparison of Leaching between Canister Materials

Comparison of the leach results for specimens from the three canisters did not reveal any pattern (Figures 6 through 11) with respect to location (level) of the specimens within the canisters. Therefore, the individual values for each canister were averaged to permit a comparison between canisters. Averaged results for normalized specimen weight loss and normalized elemental weight loss for silicon, boron, sodium, strontium, and cesium are shown in Figures 12 through 17. Individual averaged values and standard deviations are included in Appendix D. Again, no significant differences in leach behavior were observed.

The ranges in values for the leach results were wider than those for the simulated nonradioactive vitrified zeolite,[2] possibly indicating that the radioactive glass is more heterogeneous than the nonradioactive glass. Based on the overlap in range of the results, it can be concluded that there are no differences in leach behavior between the materials in the three canisters.

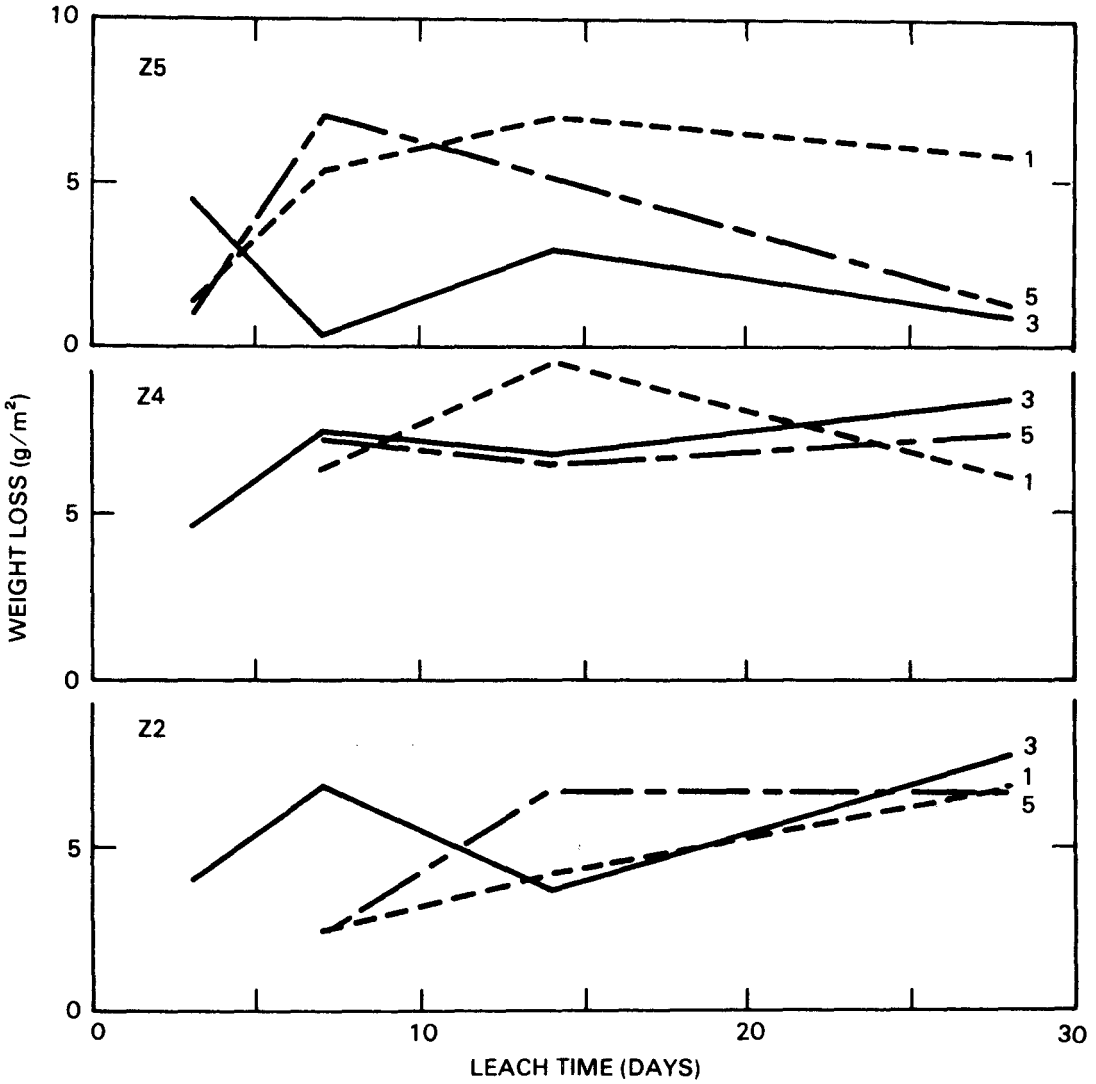


Figure 6. Normalized weight loss in specimens from three levels in three canisters of radioactive vitrified zeolite. (1, 3, 5 = level in feet from bottom of canister.)

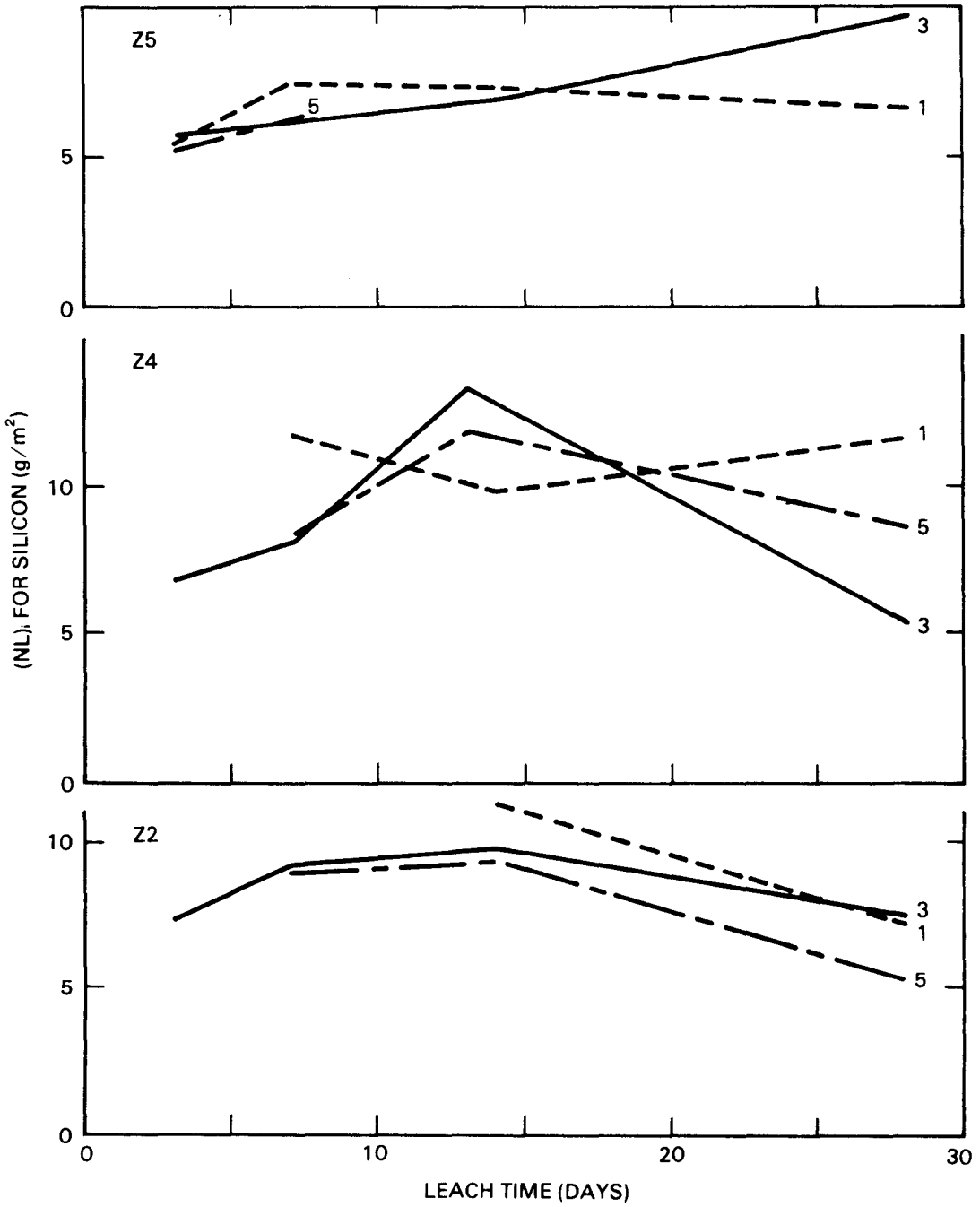


Figure 7. Normalized silicon weight loss from specimens from three levels in three canisters of radioactive vitrified zeolite. (1, 3, 5 = level in feet from bottom of canister.)

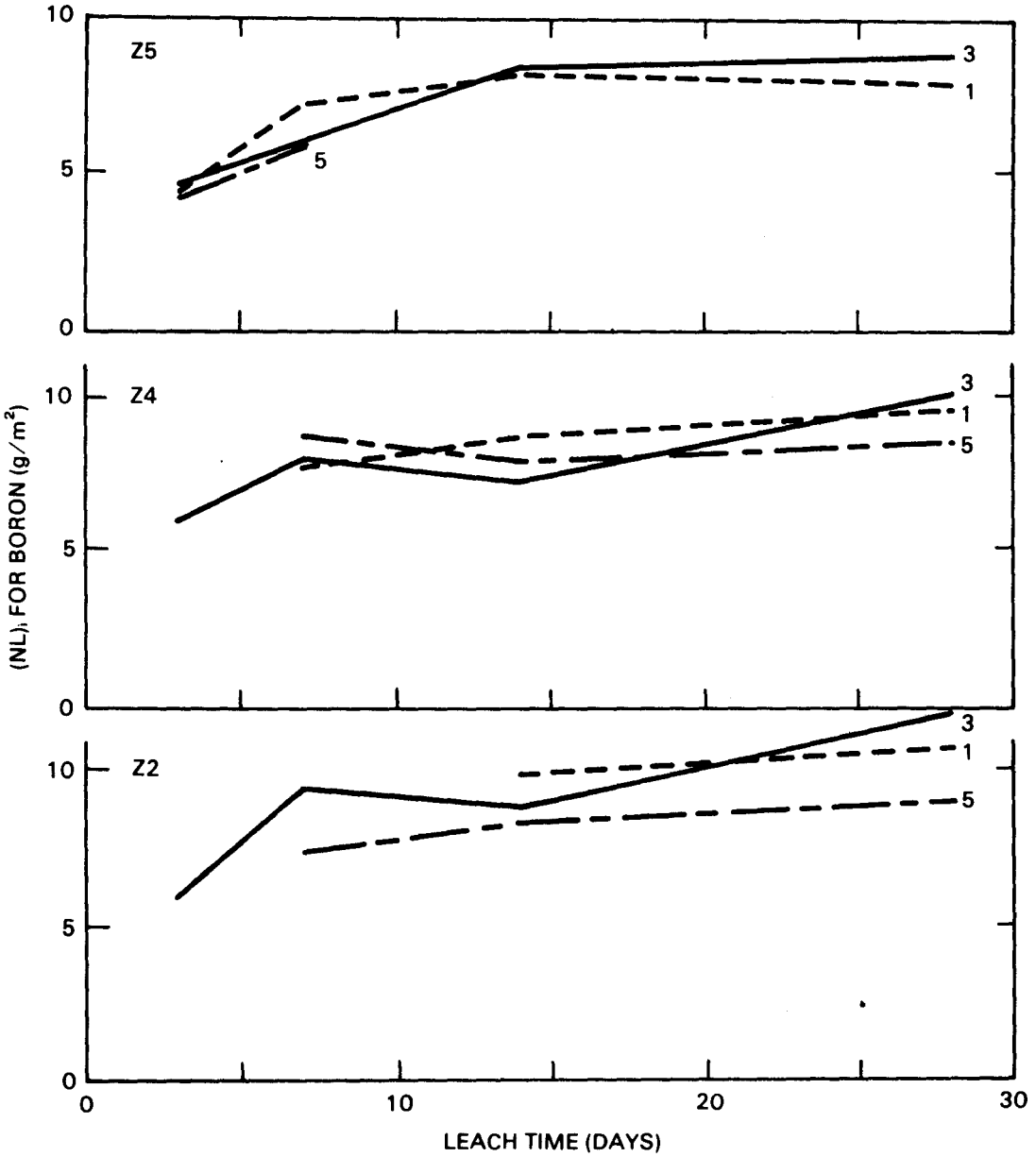


Figure 8. Normalized boron weight loss from specimens from three levels in three canisters of radioactive vitrified zeolite. (1, 3, 5 = level in feet from bottom of canister.)

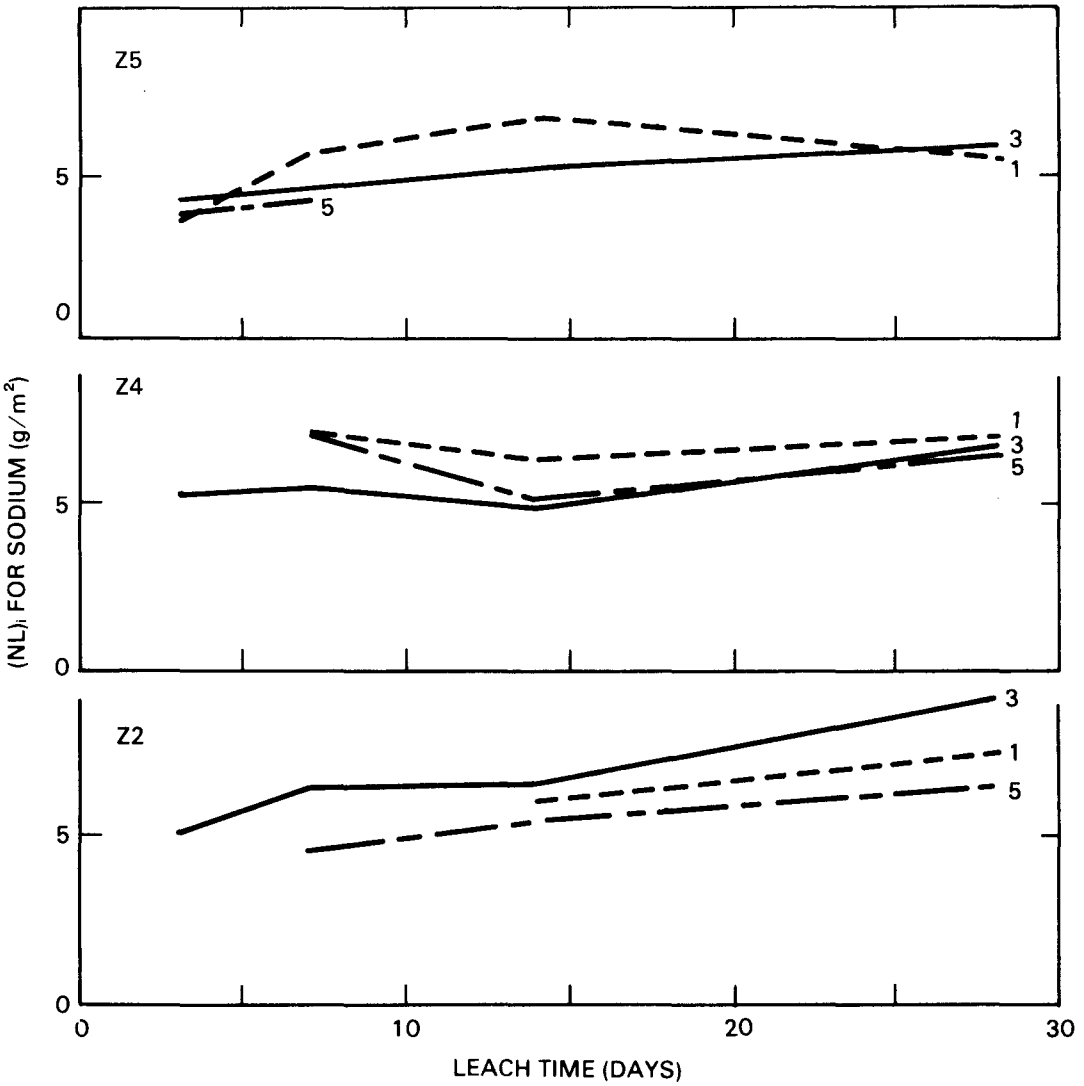


Figure 9. Normalized sodium weight loss from specimens from three levels in three canisters of radioactive vitrified zeolite. (1, 3, 5 = level in feet from bottom of canister.)

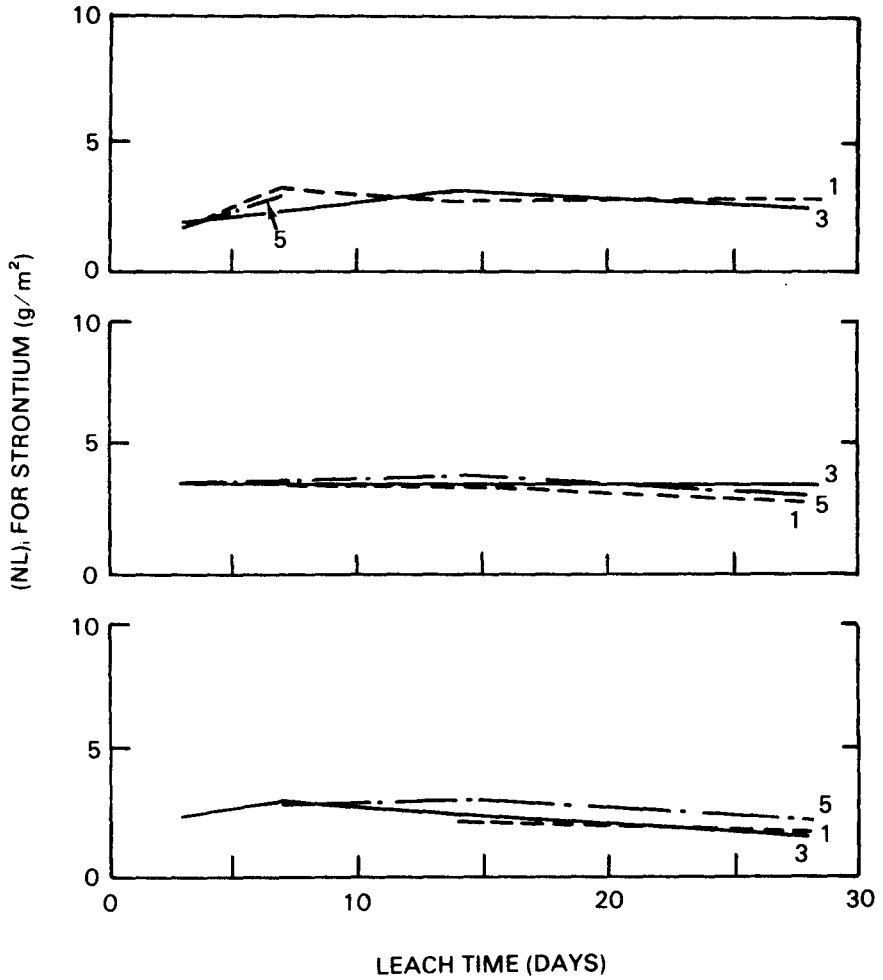


Figure 10. Normalized strontium weight loss from specimens from three levels in three canisters of radioactive vitrified zeolite. (1, 3, 5 = level in feet from bottom of canister.)

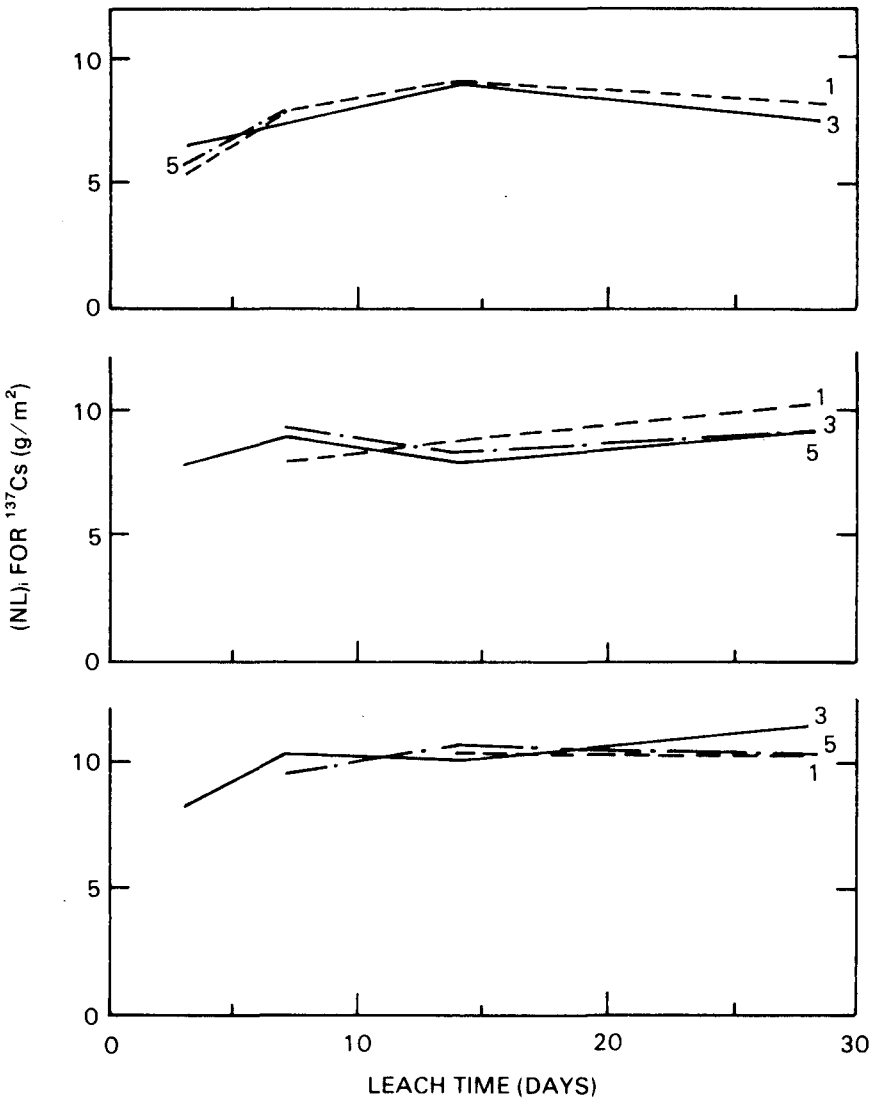


Figure 11. Normalized ^{137}Cs activity from specimens from three levels in three canisters of radioactive vitrified zeolite. (1, 3, 5 = level in feet from bottom of canister.)

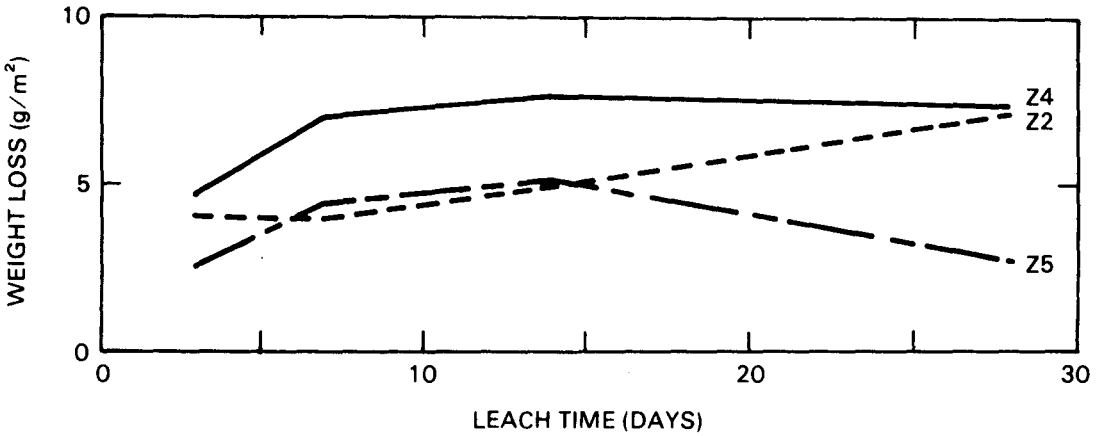


Figure 12. Average of normalized weight loss results for three canisters of radioactive vitrified zeolite.

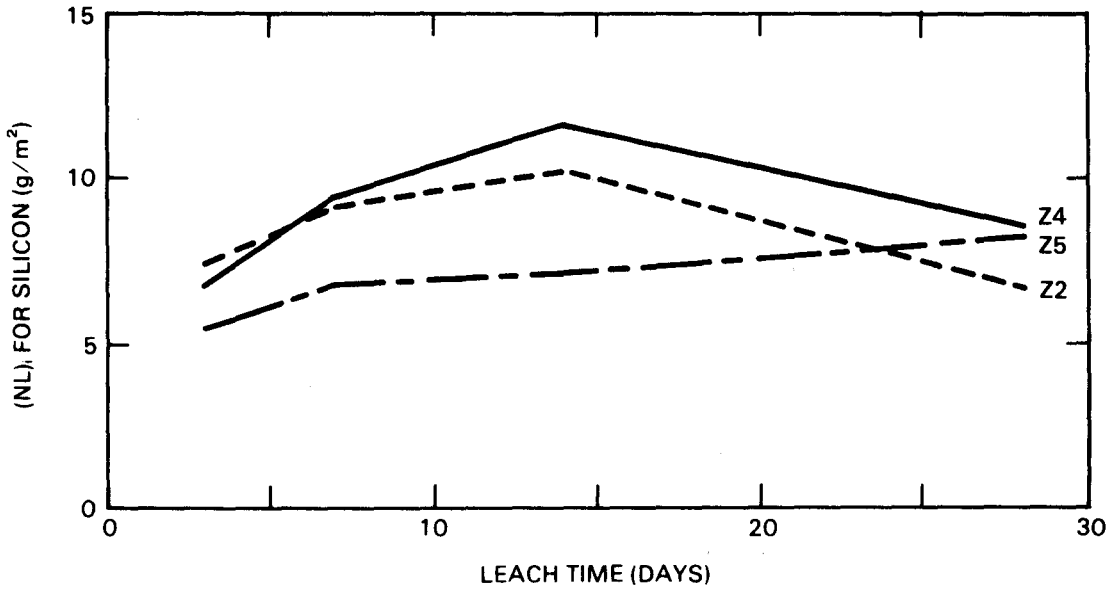


Figure 13. Average of normalized silicon weight loss results for three canisters of radioactive vitrified zeolite.

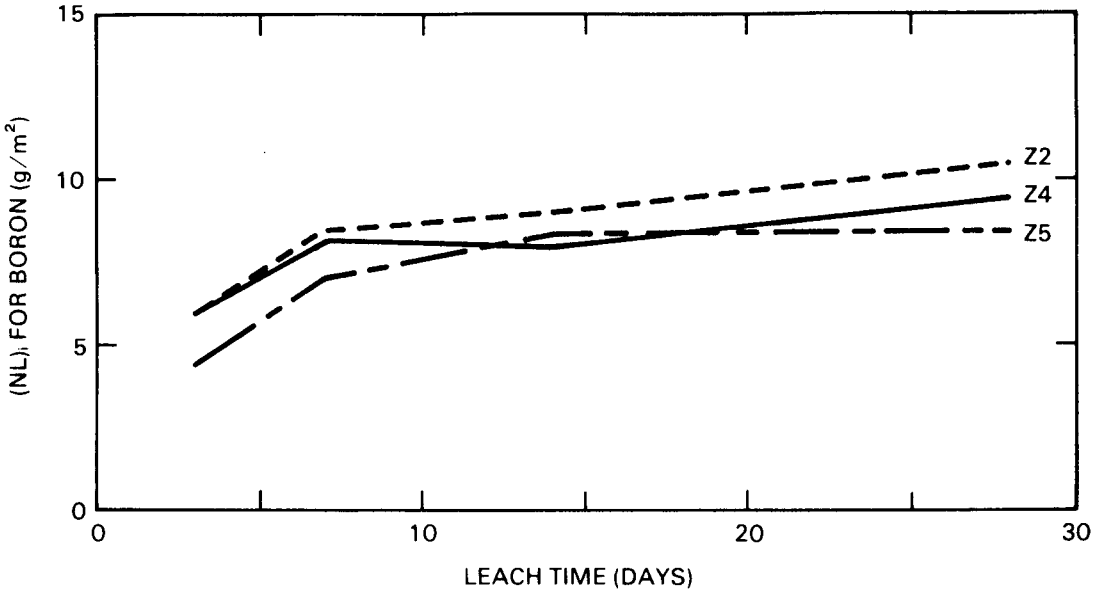


Figure 14. Average of normalized boron weight loss results for three canisters of radioactive vitrified zeolite.

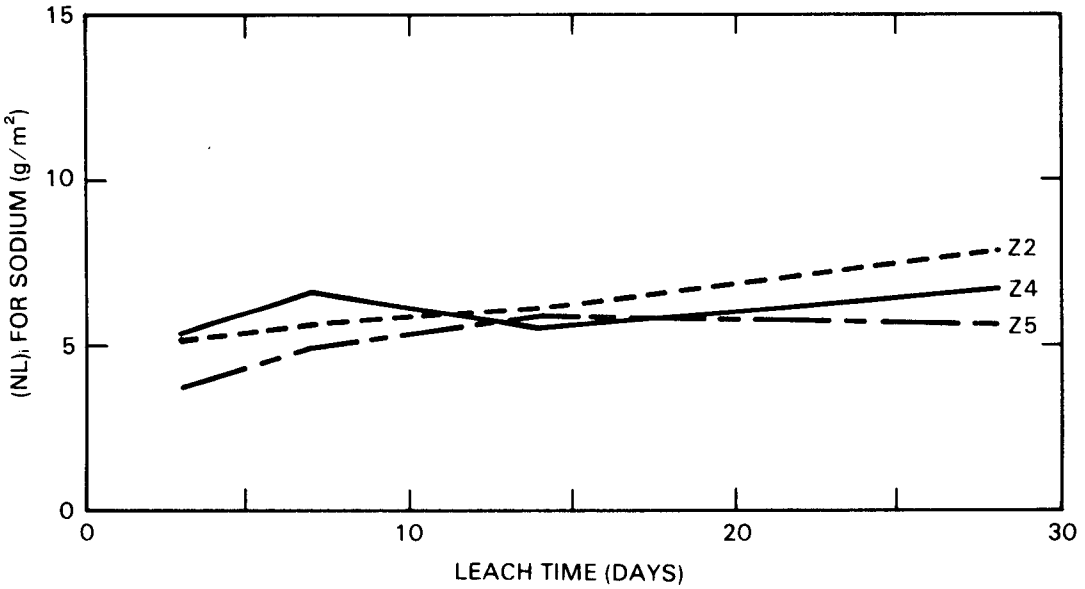


Figure 15. Average of normalized sodium weight loss results for three canisters of radioactive vitrified zeolite.

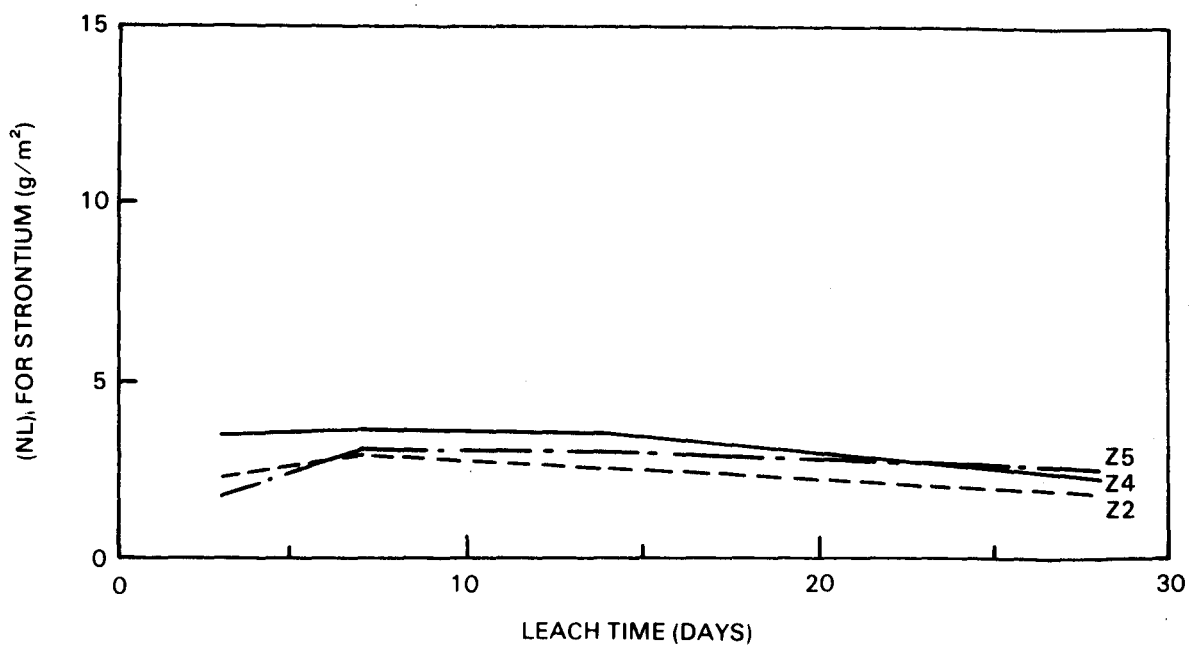


Figure 16. Average of normalized strontium weight loss results for three canisters of radioactive vitrified zeolite.

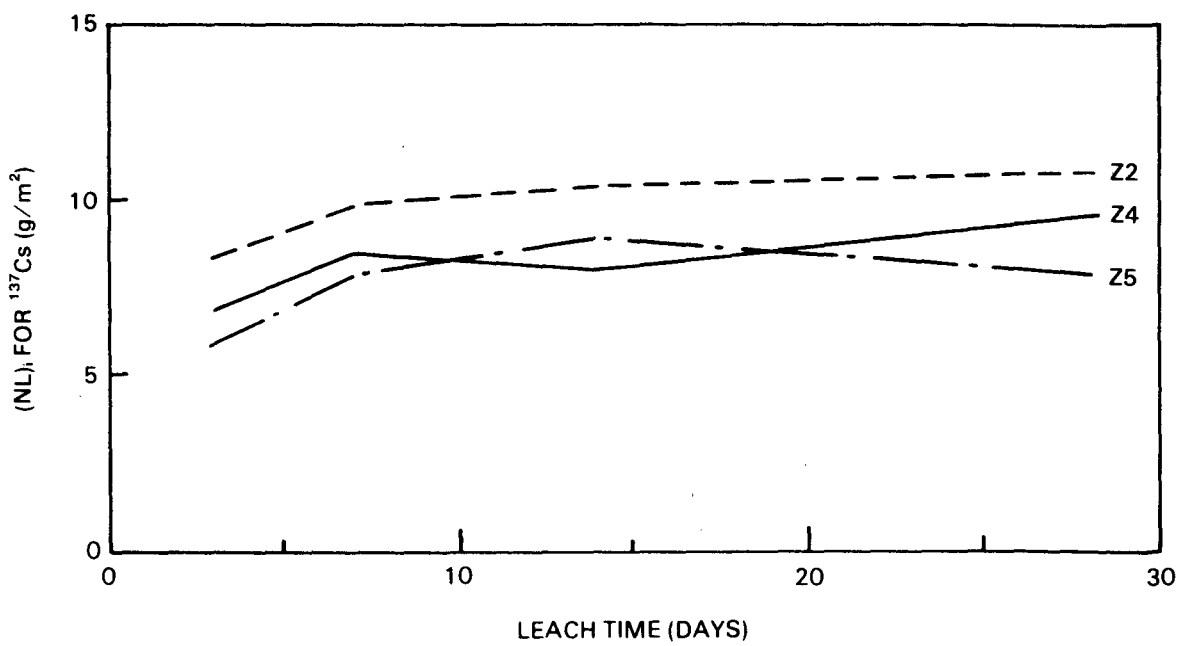


Figure 17. Average of normalized ^{137}CS activity results for three canisters of radioactive vitrified zeolite.

Comparison of Leaching: Vitrified Zeolite and Reference Glasses

Because there are no significant differences between the leaching behavior of the material in the three canisters, all the radioactive vitrified zeolite results were averaged for comparison to the reference MCC 76-68 and ZVDP-4 glasses. The comparisons for normalized specimen weight loss and normalized elemental weight loss for silicon, boron, and sodium, are shown in Figures 18 through 21. The results of leaching of ^{137}Cs from the radioactive vitrified zeolite are compared to leaching of nonradioactive cesium from MCC 76-68 and ZVDP-4 in Figure 22.

The averaged values for the radioactive vitrified zeolite are lower than any of the nonradioactive materials, in all cases, after 14 days of leaching. However, the range of leach results for the radioactive vitrified zeolite often overlaps the leach values for the in-cell measurements for ZVDP-4, indicating that the leach behavior of the radioactive and nonradioactive vitrified zeolites are comparable. As was observed for the out-of-cell measurements,[2] the leach rate of the vitrified zeolite, both radioactive and nonradioactive, was lower than that of the MCC 76-68 reference glass.

The difference in leach behavior between the in-cell and out-of-cell measurements for the ZVDP-4 glass might be attributable to the presence of silicon from the fused quartz specimen container. If the silicon analyses for the blanks are added to the silicon content for the in-cell measurements on the ZVDP-4 glass, the resultant sums are comparable to the silicon leached during out-of-cell measurements on ZVDP-4 glass. The silicon content of the leachant at any time might affect the leaching of the glass. However, this interpretation is speculative because silicon leaching of the MCC 76-68 glass was higher in-cell than out-of-cell.

The leach rate of ^{137}Cs in-cell from the vitrified zeolite was comparable to the leach of the glass-formers (Figure 23). The same correlation was

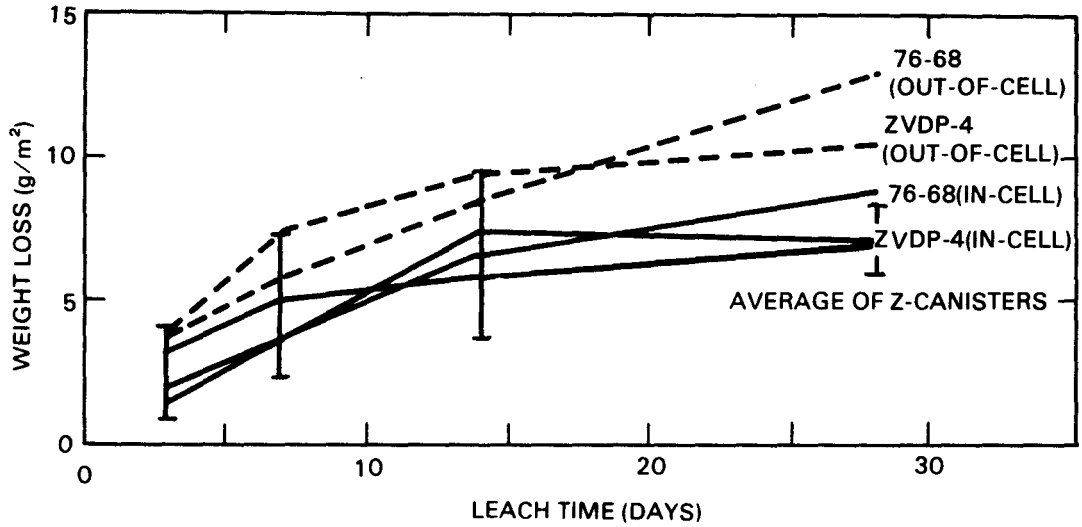


Figure 18. Comparison of normalized weight loss in radioactive vitrified zeolite, MCC 76-68 glass, and ZVDP-4 glass. (Bars represent the range of values for vitrified zeolite.)

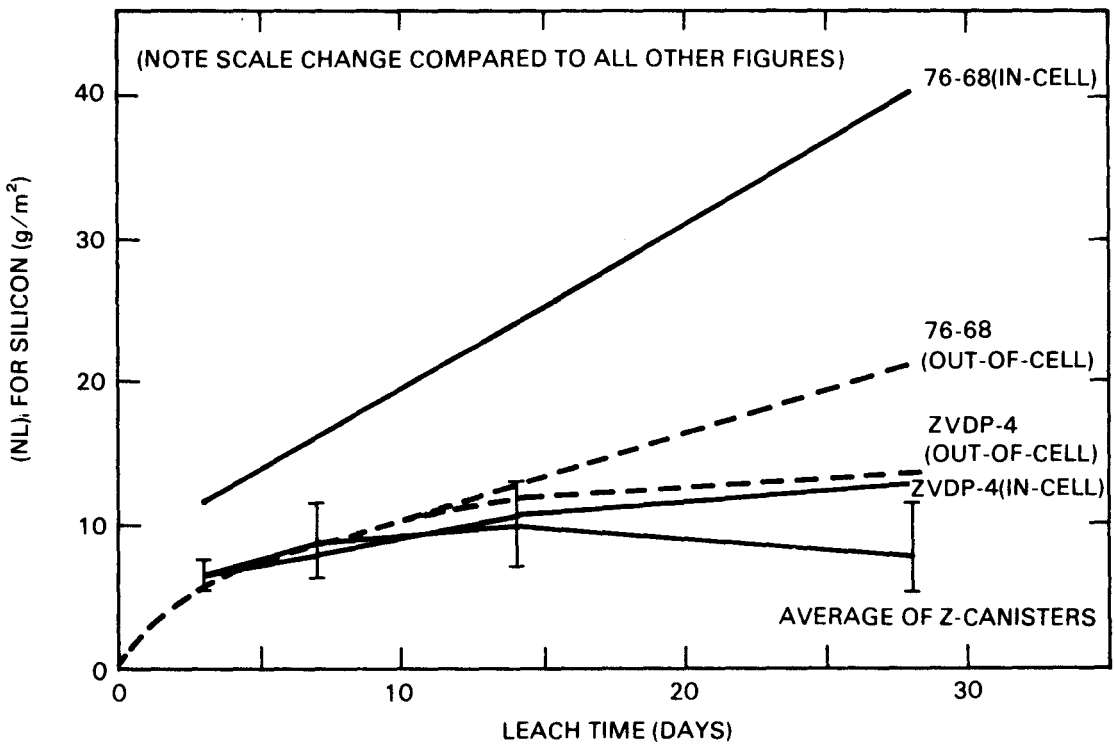


Figure 19. Comparison of normalized silicon weight loss in radioactive vitrified zeolite, MCC 76-68 glass, and ZVDP-4 glass. (Bars represent the range of values for vitrified zeolite.)

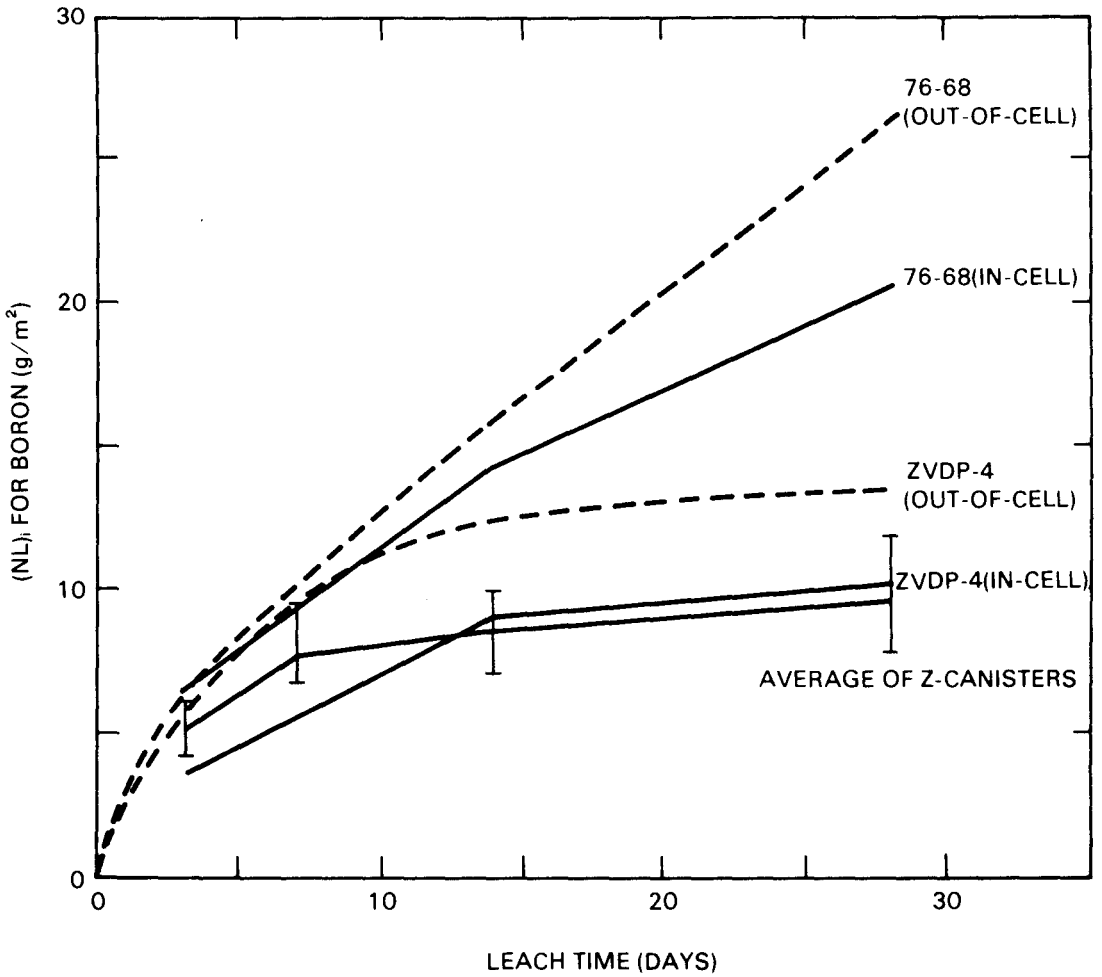


Figure 20. Comparison of normalized boron weight loss in radioactive vitrified zeolite, MCC 76-68 glass, and ZVDP-4 glass. (Bars represent the range of values for vitrified zeolite.)

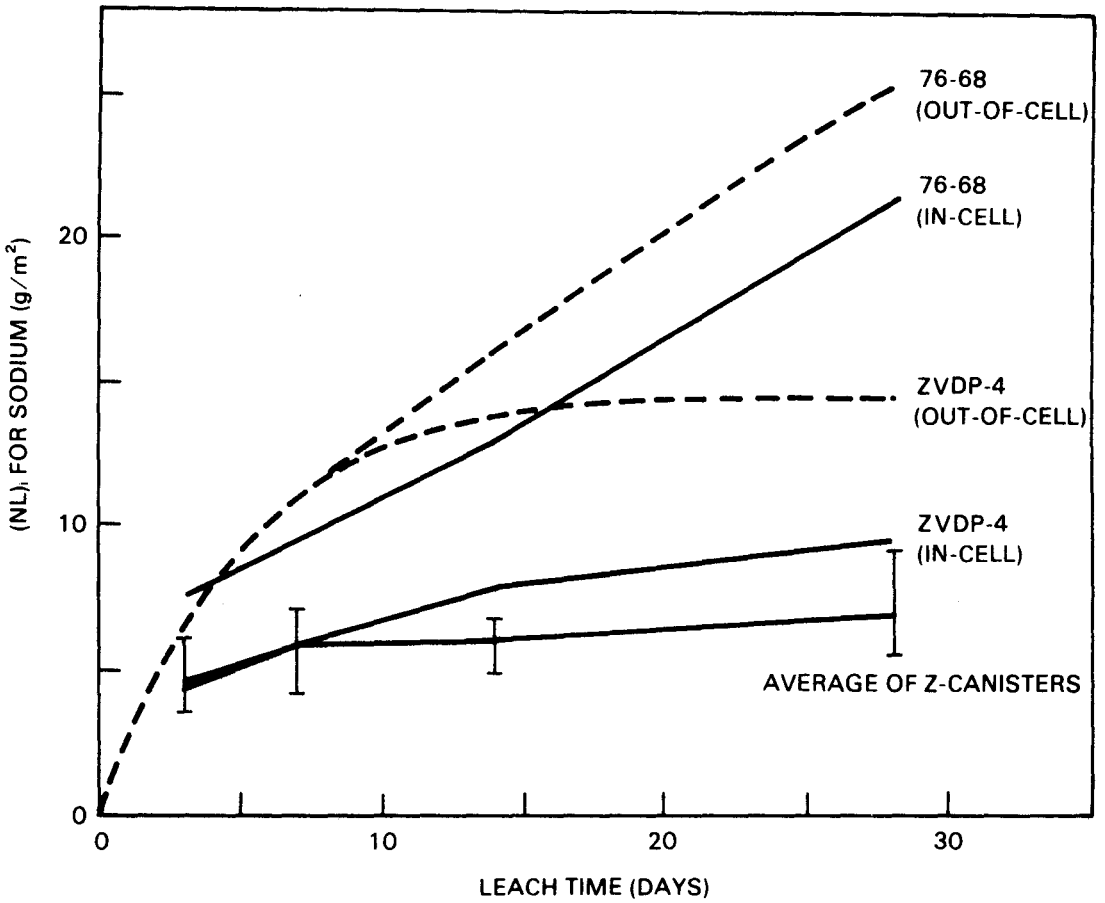


Figure 21. Comparison of normalized sodium weight loss in radioactive vitrified zeolite, MCC 76-68 glass, and ZVDP-4 glass. (Bars represent range of values for vitrified zeolite.)

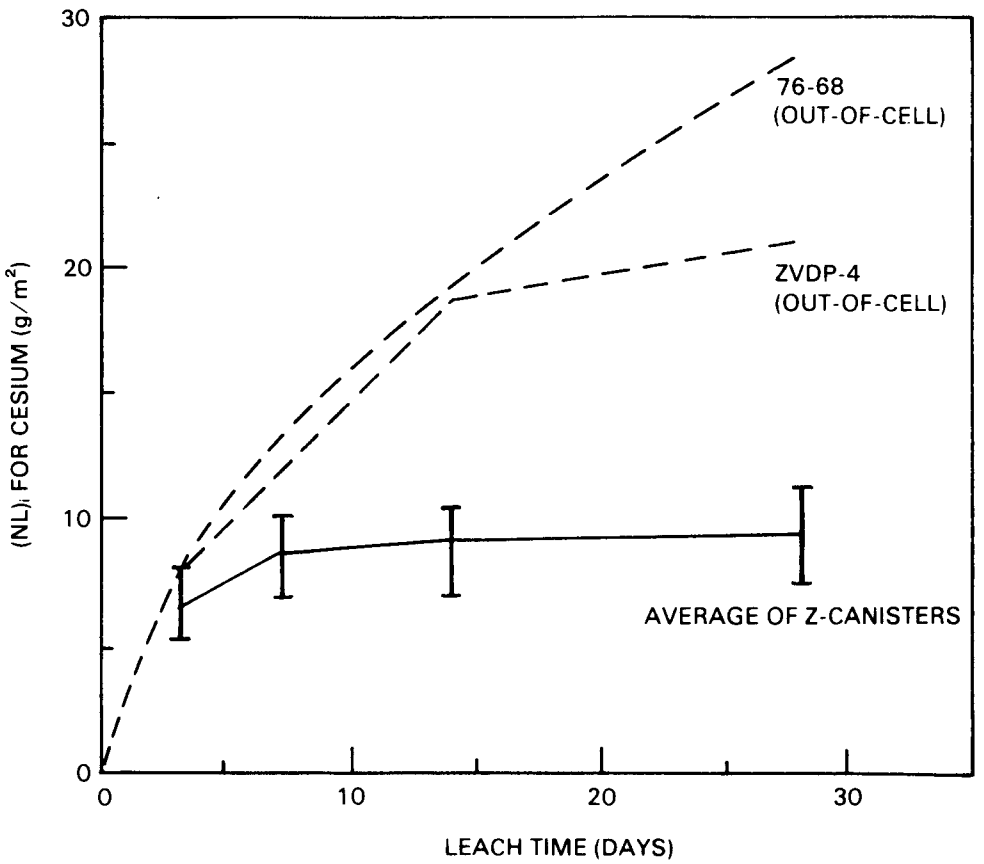


Figure 22. Comparison of normalized ^{137}Cs activity in radioactive vitrified zeolite, MCC 76-68 glass and ZVDP-4 glass. (Bars represent range of values for vitrified zeolite.)

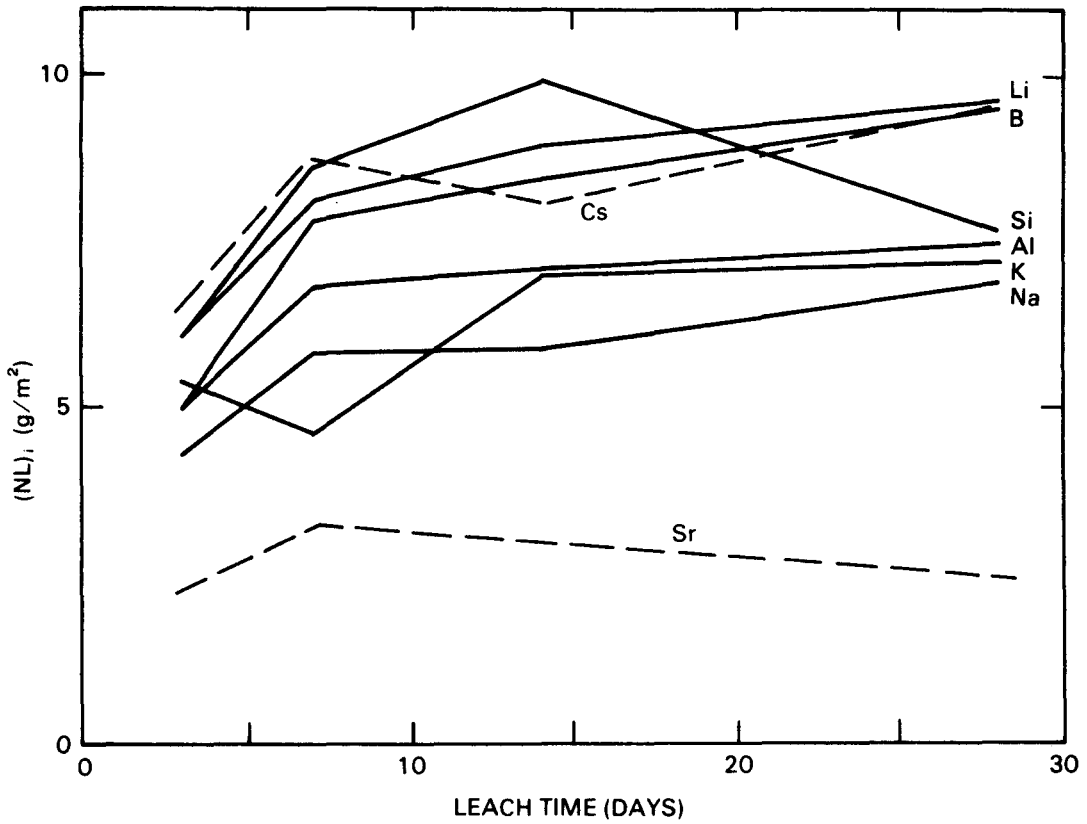


Figure 23. Comparison of leach behavior of glass-formers and fission products in radioactive vitrified zeolite.

observed in the out-of-reactor tests.[2] The lower leach rate for strontium probably results from the effect of increasing pH, as has been observed in tests on nonradioactive glass.

The lack of generally higher leach results for all of the specimens leached in-cell, compared to out-of-cell measurements on ZVDP-4 and MCC 76-68 glasses, indicates that the radiation field did not enhance the leach rates of the glasses that were tested.

CONCLUSIONS

The normalized mass loss of cesium and the glass-former elements from the radioactive vitrified zeolite after the 28-day leach test was significantly lower (a factor of 2 or more) than that from the nonradioactive reference glass MCC 76-68.

The average leach rate for the radioactive vitrified zeolite was slightly lower than the rate for the nonradioactive vitrified zeolite, for all elements reported and all leach times greater than seven days.

The leach behavior of radioactive vitrified zeolite from all three canisters was essentially the same.

There was no significant systematic difference in leach behavior relative to location of the radioactive vitrified zeolite (top, middle, bottom) in the canisters tested.

The microstructures of the radioactive material were consistent with those observed in the nonradioactive material, and no second phase components were detected.

The radiation field of the hot cell apparently did not adversely affect the leaching behavior of the reference MCC 76-68 glass and the nonradioactive simulated vitrified zeolite, and, therefore, should have had little effect upon the leaching of the radioactive vitrified zeolite.

REFERENCES

1. G. H. Bryan et al., Summary of Radioactive Operations for Zeolite Vitrification Demonstration Program, PNL-4768, Pacific Northwest Laboratory, June 1983.

2. J. L. Daniel, Zeolite Vitrification Demonstration Program - Characterization of Nonradioactive Demonstration Product, PNL-4271 (GEND-025), Pacific Northwest Laboratory, April 1982.

3. DOE/TIC-11400, Nuclear Waste Materials Handbook Waste Form Test Methods, prepared by Materials Characterization Center, Pacific Northwest Laboratory, Richland, Washington, December 1980.

APPENDIX A

EQUIPMENT

APPENDIX A

EQUIPMENT

Activity	Equipment
Specimen weight measurement	Mettler AC 100 balance
Leach container weight measurement	Ohaus #7503 balance
pH measurement	Markson Model 88 meter with Model 830 probe
Temperature measurement	Type K thermocouples
Temperature recording	Fluke data logger, Model 2240 B
^{137}Cs analysis	EG&G Ortec 7040 Series spectrometer
Chemical analysis	Applied Research Laboratories Model 3400 ICP spectrometer

APPENDIX B

CALIBRATION PROCEDURE

APPENDIX B

CALIBRATION PROCEDURE

Out-of-cell calibrations were performed on each oven, during which the temperatures in the specimen cavities were measured and correlated with an average oven body temperature. This procedure permited cavity temperatures to be inferred from hot-cell oven temperature measurements. The temperatures in the specimen cavities were not measured directly during leach testing.

During the out-of-cell calibrations, each oven was heated to and controlled at approximately 90°C. The temperature of the oven body was measured with thermocouples in the same manner as in the hot-cell tests. In addition, the temperature of each specimen cavity was measured with a thermocouple while empty. Based on an average for the oven body temperatures and using the temperature in each specimen cavity, correlation factors were computed for each specimen cavity which, when applied to the average oven body temperature for the hot-cell tests, provided an inferred temperature for each specimen cavity and thus for each specimen. Both ovens were calibrated without argon flowing through the ovens. Before calibration, extensive tests were run with and without argon in the ovens. The absence or presence of argon, either flowing or stagnant, did not noticeably affect the oven temperatures.

Each oven was calibrated separately at approximately 90°C over a 24-hour calibration period with temperature data taken every 15 minutes. The arrangement for the out-of-cell calibration of Oven 1 is shown in Figure 3. The arrangement was the same for Oven 2. All thermocouple outputs were recorded on the Fluke data logger paper tape except for the control thermocouple, which was connected to the LOVE controller.

Oven body thermocouples TC-1 and TC-2 were averaged and the standard deviaton calculated for each 15-minute interval over the 24-hour calibration period. The standard deviatons varied from 0 to 0.28. Thermocouple TC-3 was

not included in the average because, for some unexplained reason, the temperature at this location was about 3°C below the average of TC-1 and TC-2. However, the temperature in cavity 9 was generally less than 1°C below the average of the nine thermocouples in the cavities. Both ovens displayed the same effect.

The average of oven body thermocouples TC-1 and TC-2 and the time-average of the average of these thermocouples are shown in Figure B-1 for Oven 1 and in Figure B-2 for Oven 2. The time-average of the average of the two thermocouples was obtained from pairs of readings taken at 15-minute intervals. In the same manner, a time-average plot for each thermocouple in each cavity was also generated. Cavity temperature corrections for each specimen cavity were computed by subtracting the specimen cavity time-average temperature from the oven body time-average temperature at each time increment, and averaging overall time increments. These corrections were applied to the oven body temperatures taken during leach testing in the hot cell to provide inferred specimen cavity temperatures. The additive corrections for the specimen cavities are listed in Table B-1.

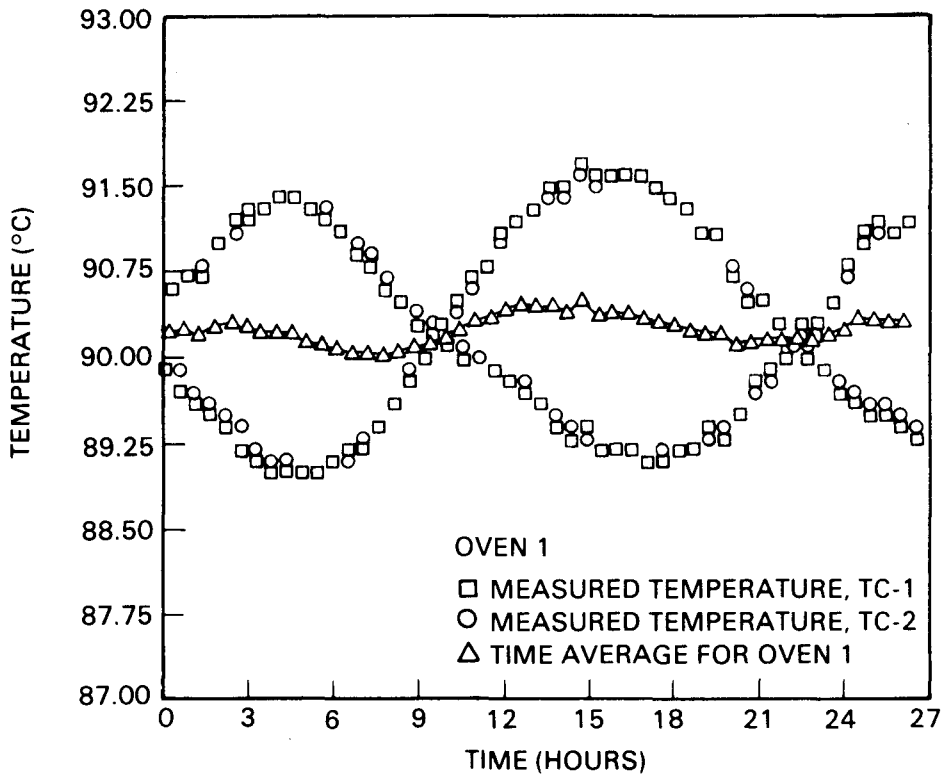


Figure B-1. Calibration data for Oven 1. (April 1983, Time 0 = 12:14:37:22)

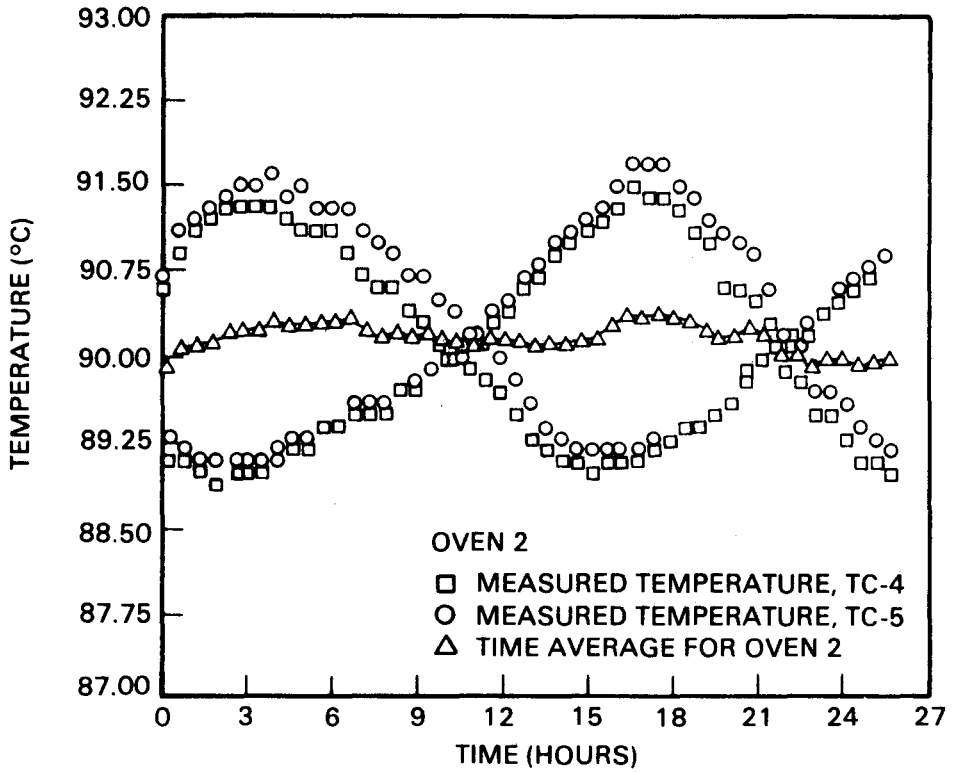


Figure B-2. Calibration data for Oven 2. (April 1983, Time 0 = 7:13:49:49)

TABLE B-1. TEMPERATURE CORRECTIONS FOR SPECIMEN CAVITIES

Specimen Cavity Identification	Correction (additive) (°C)
1	0.44
2	0.31
3	0.25
4	0.27
5	0.20
6	0.09
7	0.43
8	0.41
9	0.52
10	0.39
11	0.30
12	0.38
13	0.26
14	0.20
15	0.18
16	0.31
17	0.32
18	0.46

APPENDIX C

CHEMICAL ANALYSES, EQUIVALENT OXIDE, AND f_i FOR CONTROL SAMPLES

TABLE C-1. CHEMICAL ANALYSES, EQUIVALENT OXIDE, AND f_i FOR AS-CAST (NONLEACHED) SAMPLES

Element	Weight Oxide/ Weight Cations	Average Analyses - Elemental[a] (wt%)			Equivalent Oxide (wt%)			f_i (elemental mass fraction)		
		CL-1 (Z4-3)	CH11 (Z2-3)	CH21 (Z5-3)	CL-1 (Z4-3)	CH11 (Z2-3)	CH21 (Z5-3)	CL-1 (Z4-3)	CH11 (Z2-3)	CH27 (Z5-3)
Al (Al_2O_3)	1.89	8.15	7.78	8.51	15.40	14.70	16.08	0.075	0.073	0.080
B (B_2O_3)	3.22	3.25	3.18	2.51	10.47	10.24	8.08	0.030	0.030	0.024
Ba (BaO)	1.12	0.25	0.24	0.23	0.28	0.27	0.26	0.0023	0.0023	0.0022
Ca (CaO)	1.40	1.49	1.36	1.59	2.09	1.90	2.23	0.014	0.013	0.015
Ce (Ce_2O_3)	1.17	0.10	0.04	--	0.12	0.05	--	0.0009	0.0004	--
Cr (Cr_2O_3)	1.46	0.09	0.06	--	0.13	0.09	--	0.0008	0.0006	--
Fe (Fe_2O_3)	1.43	1.76	1.33	1.24	2.52	1.90	1.77	0.016	0.013	0.012
K (K_2O)	1.20	1.26	0.61	1.34	1.51	0.73	1.61	0.012	0.006	0.013
Li (Li_2O)	2.15	2.23	2.19	1.87	4.79	4.71	4.02	0.020	0.021	0.018
Mg (MgO)	1.66	0.49	0.42	0.46	0.81	0.70	0.76	0.0045	0.0040	0.0043
Mn (MnO_2)	1.58	0.08	0.07	0.08	0.13	0.11	0.13	0.0007	0.0007	0.0008
Na (Na_2O)	1.35	13.7	13.6	12.5	18.50	18.36	16.88	0.126	0.128	0.118
Nd (Nd_2O_3)	1.17	0.12	0.04	--	0.14	0.05	--	0.0011	0.0004	--
Ni (NiO)	1.27	0.03	0.01	--	0.04	0.01	--	0.0003	0.0001	--
Rh (Rh_2O_3)	1.23	0.07	0.03	--	0.08	0.04	--	0.0006	0.0003	--
Si (SiO_2)	2.14	21.05	21.30	22.50	45.05	45.58	48.15	0.193	0.201	0.212
Sr (SrO)	1.18	0.08	0.08	0.08	0.09	0.09	0.09	0.0007	0.0008	0.0008
Ti (TiO_2)	1.67	4.01	3.82	3.53	6.70	6.38	5.90	0.037	0.036	0.033
Zn (ZnO)	1.24	0.03	0.01	--	0.04	0.01	--	0.0003	0.0001	--
Zr (ZrO_2)	1.35	0.01	--	--	0.01	--	--	0.0001	--	--
Cs (Cs_2O)	1.06	0.06	0.05	0.05	0.05	0.05	0.05	0.0006	0.0005	0.0005
Totals					108.95	105.97	106.01			

a. Average of two ICP analyses on the as-cast specimens; one from fusion in a nickel crucible, the other in a zirconium crucible.

TABLE C-2. ^{137}Cs ACTIVITY AS-CAST SAMPLES

Sample Number	Control for Canister	Method of Determination[a]	Activity (μCi/mg)
SH1M1	Z2	Solid glass	222
CH11		Fusion - Zr	146
CH11		Fusion - Ni	129
Average	Z2		166
SLM1	Z4	Solid glass	212
CL-1		Fusion - Zr	173
CL-1		Fusion - Ni	185
Average	Z4		190
SH2M1	Z5	Solid glass	222
CH21		Fusion - Zr	123
CH21		Fusion - Ni	157
Average	Z5		167

a. Solid glass = γ-counting of ~10 mg sample of vitrified zeolite.
Fusion - Zr = γ-counting of solution prepared from material fused in NA_2O_2 in a zirconium crucible.
Fusion - Ni = γ-counting of solution prepared from material fused in KOH in a nickel crucible.

TABLE C-3. CHEMICAL ANALYSES, EQUIVALENT OXIDE, AND f_i FOR ZVDP-4 and MCC 76-68 REFERENCE GLASSES

Element	Weight Oxide/ Weight Cations	Elemental (wt%)		Equivalent Oxide (wt%)		f_i (elemental mass fraction)	
		ZVDP-4	76-68	ZVDP-4	76-68	ZVDP-4	76-68
Al	1.89	7.51	0.3	14.20	0.6	0.079	0.0032
B	3.22	1.55	2.7	4.98	8.8	0.016	0.028
Ba	1.12	0.05	0.5	0.06	0.5	0.0005	0.005
Ca	1.40	0.64	1.7	0.89	2.4	0.007	0.018
Cr	1.46	0.02	0.1	0.03	0.2	0.0002	0.0011
Cs	1.06	0.40	0.9	0.42	1.0	0.0042	0.010
Cu				0.05			
Fe	1.43	1.21	6.6	1.73	9.4	0.013	0.069
Li	2.15	2.35	--	5.05	--	0.025	--
Mg	1.66	0.20	0.1	0.33	0.2	0.0021	0.0011
Mn	1.58	0.02	0.1	0.03	0.1	0.0002	0.0011
Na	1.35	11.08	8.5	14.96	11.5	0.117	0.089
Si	2.14	21.21	19.1	45.38	40.8	0.224	0.201
Sr	1.18	0.03	0.3	0.03	0.4	0.0003	0.0032
Ti	1.67	4.23	1.8	7.06	3.0	0.045	0.019
Zn	1.24	0.04	3.79	0.05	4.7	0.0004	0.040
Zr	1.35	0.01	1.3	0.02	1.7	0.0001	0.014
Rare earths	--	--	--	--	9.9		
Totals				94.92	95.2		

APPENDIX D

INDIVIDUAL TEST RESULTS

TABLE D-1. SUMMARY OF 3-DAY TESTS FROM 6/7/83 TO 6/10/83[a]

	Specimen Number/Source								
	1(3) Z4-3	2(3) Z2-3	SH2M3 Z5-3	4(3) ZVDP-4	5(3) Blank	6(3) 76-68	7(3) Z5-5	8(3) Blank	9(3) Z5-1
Weight leachant (g)	30.40	29.10	30.55	29.35	29.60	29.70	30.80	30.20	30.40
Experiment initial weight (g)	103.00	98.20	95.60	93.95	102.90	102.80	100.90	93.40	99.40
Experiment final weight (g)	101.60	98.20	95.55	92.40	--	102.45	100.00	93.45	99.45
Leachant loss (g)	1.40	0.00	0.05	1.55	All	0.35	0.90	+0.05	+0.05
Initial pH at 20°C	7.00	7.00	7.00	7.00	7.00	7.00	7.00	7.00	7.00
Final pH at 20°C	8.25	8.91	8.75	8.93	--	7.15	8.55	7.26	8.56
Initial specimen weight (g)	0.3738	0.3831	0.3847	0.4039	--	0.7209	0.3830	--	0.3952
Final specimen weight (g)	0.3724	0.3819	0.3833	0.4035	--	0.7203	0.3827	--	0.3948
Specimen weight loss (g) [%]	0.0014 [0.37]	0.0012 [0.31]	0.0014 [0.36]	0.0004 [0.10]	-- --	0.0006 [0.08]	0.0003 [0.08]	-- --	0.0004 [0.10]
Specimen weight loss (g/m^2)	4.7	4.0	4.6	1.3	--	2.0	1.0	--	1.3

a. Test temperature was 90.4 ± 2.3°C.

TABLE D-2. SUMMARY OF 7-DAY TESTS FROM 7/25/83 TO 8/1/83 AND FFROM 8/1/83 TO 8/8/83[a]

	Specimen Number/Source								
	12a(14) Z4-3	12b(14) Z4-5	15a(14) Z2-3	15b(14) Z4-1	16a(14) Z5-3	16b(14) Z2-1	17a(14) Z2-5	17b(14) Z5-1	18a(14) Z5-5
Weight leachant (g)	29.80	30.00	29.80	30.25	29.05	30.00	29.10	29.90	29.20
Experiment initial weight (g)	87.40	101.25	103.65	93.70	118.35	103.45	115.60	106.80	97.00
Experiment final weight (g)	87.25	98.95	103.40	93.50	116.75	88.25	115.25	106.15	96.15
Leachant loss (g)	0.15	2.30	0.25	0.20	1.60	All[b]	0.35	0.65	0.85
Initial pH at 20 °C	7.00	7.00	7.00	7.00	7.00	7.00	7.00	7.00	7.00
Final pH at 20°C	8.43	8.80	8.77	8.55	8.70	--	8.76	8.59	8.71
Initial specimen weight (g)	0.4143	0.4016	0.4084	0.4055	0.4063	0.3573	0.3683	0.3714	0.3725
Final specimen weight (g)	0.4120	0.3994	0.4063	0.4036	0.4062	0.3566	0.3670	0.3698	0.3704
Specimen weight loss (g) [%]	0.00023 [0.56]	0.0022 [0.55]	0.0021 [0.52]	0.0019 [0.47]	0.0001 [0.02]	0.0007 [0.20]	0.0013 [0.35]	0.0016 [0.43]	0.0021 [0.56]
Specimen weight loss (g/m^2)	7.54	7.16	6.93	6.32	0.33	2.38	2.34	5.30	7.00

a. Test temperature was 91.6 ± 2.4°C.
b. Remaining leachant lost during weighing/sampling.

TABLE D-3. SUMMARY OF 14-DAY TESTS FROM 7/25/83 TO 8/8/83[a]

	Specimen Number/Source						
	1(14) Z4-5	2(14) Z4-3	3(14) Z4-1	4(14) 76-68	5(14) Blank	6(14) Z5-5	7(14) Z2-5
Weight leachant (g)	29.65	29.35	28.70	29.80	29.10	29.85	29.90
Experiment initial weight (g)	93.95	119.15	120.50	101.95	116.55	89.20	117.85
Experiment final weight (g)	93.20	118.65	119.75	101.00	116.25	65.80	117.50
Leachant loss (g)	0.70	0.50	0.75	0.95	0.30	23.40	0.35
Initial pH at 20°C	7.00	7.00	7.00	7.00	7.00	7.00	7.00
Final pH at 20°C	8.07	7.98	8.46	8.51	8.07	--	8.42
Initial specimen weight (g)	0.3769	0.4123	0.4140	0.7345	--	0.3647	0.3910
Final specimen weight (g)	0.3749	0.4102	0.4111	0.7325	--	0.3647	0.3889
Specimen weight loss (g) [%]	0.0020 [0.53]	0.0021 [0.51]	0.0029 [0.70]	0.0020 [0.27]	-- --	-- --	0.0020 [0.51]
Specimen weight loss (g/m^2)	6.64	6.91	9.55	6.54	--	--	6.66

TABLE D-3 (continued)

	Specimen Number/Source					
	8(14) Z2-3	9(14) Z2-1	10(14) Z5-3	11(14) Z5-1	13(14) ZVDP-4	14(14) Blank
Weight leachant (g)	29.50	29.65	29.80	29.70	29.45	29.25
Experiment initial weight (g)	112.40	119.20	90.90	95.05	93.30	95.95
Experiment final weight (g)	110.00	118.80	89.20	89.95	93.25	94.60
Leachant loss (g)	2.40	0.40	1.70	5.10	0.05	1.35
Initial pH at 20°C	7.00	7.00	7.00	7.00	7.00	7.00
Final pH at 20°C	8.54	8.63	8.16	8.40	8.96	7.25
Initial specimen weight (g)	0.3922	0.4144	0.3853	0.3684	0.4098	--
Final specimen weight (g)	0.3911	0.4131	0.3844	0.3663	0.4075	--
Specimen weight loss (g) [%]	0.0011 [0.38]	0.0013 [0.31]	0.0009 [0.23]	0.0021 [0.57]	0.0023 [0.56]	-- --
Specimen weight loss (g/m^2)	3.65	4.19	2.97	6.98	7.38	--

a. Test temperature was 91.6 ± 2.4°C.

TABLE D-4. SUMMARY OF 28-DAY TESTS FROM 6/15/83 TO 7/13/83[a]

	Specimen Number/Source								
	1(28) Z2-3	2(28) Z2-3	3(28) Z2-3	4(28) 76-68	5(28) Blank	6(28) Z4-3	7(28) Z5-3	8(28) Z5-3	9(28) Z5-3
Weight leachant (g)	29.45	29.50	29.65	29.70	29.45	30.05	29.60	28.55	29.50
Experiment initial weight (g)	85.50	100.00	99.30	99.55	90.80	106.10	101.30	106.05	94.15
Experiment final weight (g)	77.50	78.70	--	99.20	90.85	105.45	--	105.05	75.55
Leachant loss (g)	8.00	21.30	All	0.35	+0.05	0.65	All	2.00	18.60
Initial pH at 20°C	7.00	7.00	7.00	7.00	7.00	7.00	7.00	7.00	7.00
Final pH at 20°C	8.48	--	--	8.54	7.95	7.93	--	8.12	8.38
Initial specimen weight (g)	0.3580	0.3559	0.3681	0.7276	--	0.3742	0.3596	0.3878	0.4071
Final specimen weight (g)	0.3556	0.3537	0.3678	0.7249	--	0.3717	0.3587	0.3875	0.4050
Specimen weight loss (g) [%]	0.0024 [0.67]	0.0022 [0.62]	0.0003 [0.08]	0.0027 [0.37]	--	0.0025 [0.67]	0.0009 [0.25]	0.0003 [0.08]	0.0021 [0.52]
Specimen weight loss (g/m^2)	7.89	7.38	1.00	8.83	--	8.39	2.98	0.99	6.89

TABLE D-4. (continued)

	Specimen Number/Source								
	10(28) Z4-5	11(28) Z4-1	12(28) Z2-5	13(28) ZVDP-4	14(28) Blank	15(28) Z4-3	16(28) Z2-1	17(28) Z5-5	18(28) Z5-1
Weight leachant (g)	29.10	29.90	29.35	29.35	29.60	29.30	28.75	29.85	29.50
Experiment initial weight (g)	100.35	99.30	94.60	97.15	92.75	96.50	96.70	91.35	96.65
Experiment final weight (g)	100.00	98.25	91.25	96.00	--	79.10	94.40	--	96.65
Leachant loss (g)	0.35	1.05	3.35	1.15	All	17.40	2.30	All	0.00
Initial pH at 20°C	7.00	7.00	7.00	7.00	7.00	7.00	7.00	7.00	7.00
Final pH at 20°C	7.73	8.30	8.54	9.01	--	8.24	8.59	--	8.43
Initial specimen weight (g)	0.4176	0.3699	0.3850	0.4100	--	0.4220	0.3871	0.3695	0.3648
Final specimen weight (g)	0.4154	0.3681	0.3830	0.4078	--	0.4202	0.3850	0.3691	0.3630
Specimen weight loss (g) [%]	0.0002 [0.53]	0.0018 [0.49]	0.0020 [0.52]	0.0022 [0.54]	--	0.0018 [0.43]	0.0021 [0.54]	0.0004 [0.11]	0.0018 [0.49]
Specimen weight loss (g/m^2)	7.26	6.06	6.70	7.05	--	5.89	6.81	1.33	5.94

a. Test temperature was 90.4 ± 1.1°C.

TABLE D-5. CHEMICAL ANALYSIS RESULTS[a] AND $(NL)_i$[b] FOR 3-DAY TESTS

	Specimen Number/Source																
	1(3) Z4-3		2(3) Z2-3		3(3) Z5-3		4(3) ZVDP-4		6(3) 76-68		7(3) Z5-5		9(3) Z5-1		8(3) Blank	Average of Acceptable Canisters[c]	Standard Deviation
Element	ppm	$(NL)_i$	ppm	$(NL)_i$	ppm	$(NL)_i$	ppm	$(NL)_i$	ppm	$(NL)_i$	ppm	$(NL)_i$	ppm	$(NL)_i$	ppm	$(NL)_i$	
Al	4.8	6.2	4.5	6.0	3.7	4.7	4.2	4.8	(0.11)	3.3	3.3	4.1	3.4	4.1		5.0	0.9
B	1.87	6.0	1.86	6.0	1.08	4.5	0.626	3.5	1.87	6.5	0.990	4.1	1.07	4.3		5.0	0.8
Ba	0.009	--	(0.004)	--	(0.004)	--	(0.002)	--	0.046	0.7	(0.007)	--	0.007	--	0.010	--	
Ca	0.477	3.3	0.323	2.4	0.342	2.3	(0.109)	1.4	0.631	3.4	0.306	2.0	0.370	2.4		2.5	0.4
Ce							(0.08)	--	0.08	--	(0.12)	--	(0.04)	--		--	
Cr																	
Dy																	
Fe		--	(0.004)	--		--		--	0.034	--	0.022	0.2	(0.042)	0.3	(0.003)	0.1	
K	(0.6)	4.8	(0.7)	11.4	(0.3)	2.3	(0.5)	--	(0.3)	--	(0.4)	3.1	(0.7)	5.2		5.4	3.2
La									(0.010)	--							
Li	1.44	7.0	1.41	6.5	1.08	6.0	1.31	4.7			0.969	5.4	0.995	5.4		6.1	0.6
Mg	0.098	2.1	0.057	1.4	0.086	2.0	0.008	0.3	0.059	5.2	0.088	2.0	0.090	2.0		1.9	0.3
Mn																	
Mo									0.889	--							
Na	6.8	5.2	6.8	5.1	4.8	4.1	6.1	4.6	6.9	7.5	4.3	3.6	4.3	3.5	(0.05)	4.3	0.7
Nd		--		--		--		--	0.02	--	(0.04)	--	(0.05)	--	(0.03)	--	
Ni																	
Rh							0.034	--			0.071	--	(0.02)	--		--	
Ru		--		--		--		--		--		--		--	(0.02)	--	
Si	15.6	6.8	17.5	7.4	14.1	5.7	15.8	5.4	25.7	11.4	13.4	5.3	14.0	5.4	2.16	6.1	0.8
Sr	0.024	3.3	0.018	2.2	0.015	1.9			0.120	3.7	0.013	1.7	0.014	1.7		2.2	0.6
Te													(0.03)	--		--	
Ti							(0.002)	--			(0.002)	--					
Zn																--	
Zr																	
^{137}Cs		6.8		8.2		6.5		--		--		5.4		5.3		6.4	1.1

a. ppm by weight; blank indicates element not detected; parentheses ( ) indicate results near limit of detection.

b. For all elements except ^{137}Cs, $(NL)_i = \frac{(\text{ppm-blank})(\text{grams leachate})}{(f_i)\ (\text{SA in meters})\ (10^6)}$.

For ^{137}Cs, $(NL)_i = \frac{(\text{activity/mL})\ (\text{mL of leachate})}{(\text{activity of control sample/mg})\ (\text{mg of specimen})} \cdot \frac{(\text{g of specimen})}{(\text{SA in meters}^2)}$.

Dash (--) indicates either $(NL)_i$ <0.05, was negative after correction for blank, or analyses of original material not available to calculate f_i.

c. See Table 11 for basis of acceptable data.

TABLE D-6. CHEMICAL ANALYSIS RESULTS[a] AND $(NL)_i$[b] FOR 7-DAY TESTS

	Specimen Number/Source									
	12a(14) Z4-3		12b(14) Z4-5		15a(14) Z2-3		15b(14) Z4-1		16a(14) Z5-3	
Element	ppm	$(NL)_i$	ppm	$(NL)_i$	ppm	$(NL)_i$	ppm	$(NL)_i$	ppm	$(NL)_i$
Al	5.71	7.4	6.31	7.6	5.89	7.9	5.03	6.7	6.16	0.2
B	2.52	7.9	2.85	8.6	2.97	9.4	2.32	7.7	0.425	1.3
Ba	(0.009)	0.3	0.011	0.4	(0.004)	0.1	(0.009)	0.3	0.010	0.3
Ca	0.331	2.0	0.352	2.0	0.324	2.1	0.379	2.4	0.212	1.0
Ce										
Cr	0.013	1.1	0.029	3.3	0.023	3.1	(0.018)	2.3		
Dy										
Fe	0.021	--	0.046	--	0.039	--	0.054	0.3	0.069	0.1
K	(0.4)	3.2	0.9	6.8	(0.4)	6.5	(0.4)	3.3		
La			(0.013)	--						
Li	1.76	8.6	1.94	8.8	2.00	9.3	1.60	8.0	0.417	2.1
Mg	0.068	1.0	0.051	0.6	0.051	0.7	0.124	2.3	0.068	0.2
Mn									(0.005)	0.1
Mo			(0.011)	--	(0.007)	--	(0.007)	--		
Na	19.1	5.4	9.82	7.0	20.6	6.5	9.05	7.1	13.5	1.1
Nd			(0.03)	0.3						
Ni										
Rh										
Ru										
Si	21.0	8.1	22.5	8.2	23.9	9.2	27.5	11.7	5.32	0.1
Sr	0.028	3.6	0.028	3.6	0.026	2.9	0.025	3.6	0.009	0.8
Te			(0.08)							
Ti	(0.004)	--	0.013	--	(0.005)	--	0.008	--		
Zn			0.010	3.0					0.013	--
Zr										
^{137}Cs		8.9		9.1		10.3		6.9		1.6

TABLE D-6. (continued)

Element	Specimen Number/Source 17a(14) Z2-5 ppm	$(NL)_i$	17b(14) Z5-1 ppm	$(NL)_i$	18a(14) Z5-5 ppm	$(NL)_i$	Created Blanks a-series	b-series	Average of Acceptable Canisters[c] $(NL)_i$	Standard Deviation
Al	5.00	6.6	4.75	5.8	5.00	5.9			6.8	0.8
B	2.35	7.3	1.79	7.2	1.81	6.8	0.086		7.8	0.8
B	0.010	0.3	0.021	0.8	0.013	0.5	0.002	0.002	0.4	0.2
Ca	0.370	2.4	0.419	2.5	0.407	2.3	0.040	0.040	2.2	0.2
Ce										
Cr	0.020	2.6	0.015	--	0.013	--	0.004		[2.5]	0.8
Dy										
Fe	0.062	0.1	0.082	0.6	0.044	--	0.050	0.007	[0.3]	0.2
K	(0.4)	6.4	(0.4)	3.0	(0.4)	2.9			4.6	1.7
La										
Li	1.59	7.3	1.45	7.8	1.45	7.6			8.2	0.7
Mg	0.068	1.1	0.100	1.8	0.111	2.0	0.021	0.021	1.4	0.6
Mn										
Mo			(0.006)	--	(0.005)	--				
Na	18.4	4.7	6.85	5.6	17.2	4.1	12.1	0.05	5.8	1.1
Nd										
Ni										
Rh										
Ru										
Si	23.7	9.0	21.4	7.5	18.9	6.2	5.0	5.0	8.6	1.6
Sr	0.025	2.8	0.024	2.9	0.027	3.0	0.02		3.2	0.4
Te										
Ti	(0.002)	--	0.012	--	(0.003)	--	0.009			
Zn										
Zr										
^{137}Cs		9.5		7.8		7.8			8.6	1.1

a. ppm by weight; blank indicates element not detected; parentheses ( ) indicate result near limit of detection.

b. For all elements except ^{137}Cs, $(NL)_i = \frac{(\text{ppm-blank})(\text{grams leachate})}{(f_i)\ (\text{SA in meters}^2)\ (10^6)}$.

For ^{137}Cs, $(NL)_i = \frac{(\text{activity/mL})\ (\text{mL of leachate})}{(\text{activity of control sample/mg})\ (\text{mg of specimen})} \cdot \frac{(\text{g of specimen})}{(\text{SA in meters}^2)}$.

Dash (--) indicates either $(NL)_i$ <0.05, was negative after correction for blank, or analyses of original material not available to calculate f_i.

c. See Table 11 for basis of acceptable data. Brackets [ ] indicate that the average was calculated using those specimens for which a value is shown.

TABLE D-7. CHEMICAL ANALYSIS RESULTS[a] AND $(NL)_i$[b] FOR 14-DAY TESTS

	Specimen Number/Source											
	1(14) Z4-5		2(14) Z4-3		3(14) Z4-1		4(14) 76-68		7(14) Z2-5		8(14) Z2-3	
Element	ppm	$(NL)_i$	ppm	$(NL)_i$	ppm	$(NL)_i$	ppm	$(NL)_i$	ppm	$(NL)_i$	ppm	$(NL)_i$
Al	5.50	7.2	5.05	6.4	6.30	7.7	(0.03)	0.9	5.60	7.6	6.00	7.4
B	2.46	7.8	2.33	7.1	2.92	8.7	4.30	14.3	2.58	8.2	3.03	8.8
Ba	0.012	0.4	0.009	0.3	(0.006)	0.2	0.015	0.3	0.008	0.3	(0.004)	0.1
Ca	0.384	2.3	0.422	2.5	0.362	2.0	0.384	2.0	0.418	2.8	0.344	2.0
Ce												
Cr	0.029	3.1	0.024	2.8	0.021	2.0	(0.011)	0.6	0.024	3.3	0.031	4.2
Dy												
Fe	0.32	--	0.041	--	0.019	--	0.060	--	0.031	--	0.015	--
K	0.9	7.4	(0.7)	5.6	0.8	6.1			(0.6)	9.8	(0.7)	10.5
La	(0.010)	--										
Li	1.75	8.6	1.68	8.0	2.06	9.5			1.80	8.4	2.06	8.8
Mg	0.051	0.4	0.126	2.0	0.045	0.3	0.020	--	0.038	0.1	0.038	0.1
Mn	(0.003)	0.4										
Mo	0.007	--			(0.006)	--	1.81	--	(0.007)	--	(0.007)	--
Na	18.8	5.2	18.6	4.9	20.9	6.4	24.3	13.0	19.3	5.5	21.6	6.7
Nd	(0.04)	3.6										
Ni												
Rh												
Ru												
Si	31.5	11.8	34.8	13.1	29.0	9.9	59.5	24.2	27.5	9.4	30.2	9.8
Sr	0.028	3.7	0.026	3.3	0.027	3.3	0.083	2.4	0.026	3.0	0.024	2.5
Te	(0.07)	--	(0.05)	--			(0.003)	--				
Ti	0.011	--	0.005	--	(0.005)	--	0.034	0.1			(0.002)	--
Zn												
Zr	0.002	--										
^{137}Cs		8.3		6.9		8.9				10.5		10.1

TABLE D-7. (continued)

Element	Specimen Number/Source 9(14) Z2-1 ppm	(NL)$_i$	10(14) Z5-3 ppm	(NL)$_i$	11(14) Z5-1 ppm	(NL)$_i$	13(14) ZVDP-4 ppm	(NL)$_i$	5(14) Blank ppm	14(14) Blank ppm	Blank Average ppm	Average of Acceptable Canisters[c] (NL)$_i$	Standard Deviation
Al	5.83	7.5	5.77	6.7	6.33	6.5	7.56	9.0				7.1	0.5
B	3.24	9.9	2.23	8.3	2.43	8.0	1.62	9.0	0.100	0.072	0.086	8.4	0.8
Ba	(0.007)	0.2	0.025	1.0	0.031	1.1	0.004	0.4		(0.003)	0.002	0.5	0.4
Ca	0.294	1.8	0.416	2.3	0.508	2.5	0.162	1.5		0.102	0.051	2.3	0.3
Ce													
Cr	0.027	3.6	(0.015)	--	(0.007)	--				(0.008)	0.004	[3.2]	0.7
Dy													
Fe	0.051	--	0.025	--	0.008	--	0.022	--	0.59	0.40	0.50		
K	(0.6)	9.4	(0.6)	4.3	(0.5)	3.1						7.0	2.5
La													
Li	2.17	9.7	1.76	9.1	2.01	9.1	2.40	9.1				8.9	0.5
Mg	0.045	0.3	0.082	1.1	0.064	0.6	0.014	--	0.045	0.020	0.033	0.6	0.6
Mn													
Mo			(0.06)	--			(0.005)	--		(0.007)			
Na	20.4	6.1	18.6	5.1	21.8	6.7	21.8	7.8	11.1	13.1	12.1	5.8	0.7
Nd													
Ni													
Rh													
Ru													
Si	32.4	11.3	24.3	7.0	26.9	7.2	33.4	10.5	6.90	9.68	8.29	9.9	2.0
Sr	0.020	2.1	0.029	3.1	0.028	2.7	0.005	0.9	(0.001)	(0.002)	0.002	3.0	0.5
Te													
Ti	(0.001)	--	(0.002)	--	(0.002)	--			0.012	(0.005)	0.009		
Zn										0.426			
Zr													
^{137}Cs		10.2		8.8		8.9						9.1	1.1

a. ppm by weight; blank indicates element not detected; parentheses ( ) indicates result near limit of detection.

b. For all elements except ^{137}Cs, $(NL)_i = \frac{(\text{ppm-blank})\ (\text{grams leachate})}{(f_i)\ (\text{SA in meters}^2)\ (10^6)}$.

For ^{137}Cs, $(NL)_i = \frac{(\text{activity/mL (mL of leachate)}}{(\text{activity of control sample/mg (mg of specimen)}} \cdot \frac{(\text{g of specimen})}{(\text{SA in meters}^2)}$

Dash (--) indicates either $(NL)_i$ <0.05, was negative after correction for blank, or analyses of original material not available to calculate f_i.

c. See Table 11 for basis of acceptable data. Brackets [ ] indicate that the average was calculated using only those specimens for which a value is shown.

TABLE D-8. CHEMICAL ANALYSIS RESULTS[a] AND $(NL)_i$[b] FOR 28-DAY TESTS

	Specimen Number/Source													
	1(28) Z2-3		2(28) Z2-3		4(28) 76-68		6(28) Z4-3		8(28) Z5-3		9(28) Z5-3		10(28) Z4-5	
Element	ppm	$(NL)_i$	ppm	$(NL)_i$	ppm	$(NL)_i$	ppm	$(NL)_i$	ppm	$(NL)_i$	ppm	$(NL)_i$	ppm	$(NL)_i$
Al	10.2	9.9	17.9	6.8			5.95	7.8	5.59	6.1	12.1	5.4	5.46	6.9
B	5.06	11.9	10.5	9.6	6.14	21.1	3.05	10.0	2.43	8.9	5.48	8.2	2.65	8.4
Ba	0.12	3.6	(0.012)	0.1	0.019	0.3	0.016	0.6	0.018	0.6	0.018	0.2	(0.016)	0.5
Ca	0.118	0.2	0.061	--	0.153	0.4	0.366	2.1	0.362	1.7	0.128	0.1	0.197	0.8
Ce	0.110	19.4	(0.06)	4.1					0.38	--			(0.24)	2.4
Cr	0.063	7.4	0.140	6.4			0.036	4.4	0.047	--	0.041	--	(0.046)	5.5
Dy	0.013	--	(0.008)	--					0.032	--			(0.020)	--
Fe	0.027	--	0.078	0.1	0.081	0.1	0.029	--	0.052	0.2	(0.040)	--	(0.036)	0.1
K	1.32	15.5	2.05	9.4			(0.6)	4.9	1.23	8.3	1.45	4.0	(0.8)	6.3
La	0.029	--	(0.015)	--			(0.014)	--	0.048	--	(0.009)	--	(0.034)	--
Li	3.54	11.9	7.64	10.0			1.94	9.6	1.93	9.4	4.44	8.8	1.88	8.9
Mg	0.019	--	0.022	--	0.027	--	0.046	--	0.085	0.7	0.032	--	0.040	--
Mn			(0.002)	0.1					(0.003)	0.3			(0.003)	0.4
Mo	(0.015)	--	(0.025)	--	2.69	--	(0.011)	--	0.024	--	(0.011)	--	(0.020)	--
Na	16.6	9.2	33.7	7.2	20.2	21.8	8.80	6.9	7.87	5.9	18.4	5.6	8.59	6.5
Nd	0.16	28.2	(0.13)	8.9			(0.11)	9.9	0.25	--	(0.06)	--	(0.23)	19.8
Ni	0.022	15.5	(0.019)	5.2			(0.010)	3.3	0.042	--	(0.006)	--	(0.03)	9.5
Rh	0.13	30.6	(0.08)	7.3			(0.04)	6.6	0.29	--			(0.23)	36.4
Ru	(0.05)	--	(0.02)	--					(0.08)	--			(0.05)	--
Si	42.9	7.4	68.4	6.4	106	40.2	32.2	5.3	45.2	9.9	67.3	7.7	39.0	8.5
Sr	0.018	1.6	0.010	0.3	0.036	--	0.024	3.4	0.023	2.5	0.015	0.7	0.022	3.0
Te	0.19	--	(0.13)	--			(0.13)	--	0.23	--	(0.11)	--	(0.17)	--
Ti	0.029	0.1	(0.03)	--			(0.015)	--	0.033	0.1	0.018	--	(0.028)	0.1
Zn								--		--		--		--
Zr	0.011	--	(0.012)				(0.006)	5.9	0.022	--	(0.006)	--	(0.018)	--
^{137}Cs		11.3						9.1		7.5				9.0

TABLE D-8. (continued)

Element	Specimen Number/Source: 11(28) Z4-1 ppm	11(28) Z4-1 $(NL)_i$	12(28) Z2-5 ppm	12(28) Z2-5 $(NL)_i$	13(28) ZVDP-4 ppm	13(28) ZVDP-4 $(NL)_i$	15(28) Z4-3 ppm	15(28) Z4-3 $(NL)_i$	16(28) Z2-1 ppm	16(28) Z2-1 $(NL)_i$	18(28) Z5-1 ppm	18(28) Z5-1 $(NL)_i$	5(28) Blank ppm	Average of Acceptable Canisters[c] $(NL)_i$	Standard Deviation
Al	5.97	7.7	6.42	7.7	8.23	9.4	10.9	5.7	6.67	7.8	5.05	6.2		7.5	1.1
Ba	2.90	9.4	3.09	90	1.81	10.2	5.85	7.6	3.74	10.7	1.94	7.9		9.5	1.2
Ba	0.012	0.4	0.037	1.3	0.012	1.6	0.028	0.4	0.011	0.3	0.12	0.4	(0.003)	1.0	1.0
Ca	0.141	0.5	0.209	0.9	0.033	--	0.291	0.6	0.116	0.3	0.303	1.5	0.073	1.0	0.7
Ce			(0.07)	15.3			(0.08)	3.5			(0.06)	--		[12.4]	7.2
Cr	0.031	3.8	0.048	7.0	0.022	9.9	0.058	2.8	0.043	6.2	0.026	--		[5.7]	1.3
Dy	(0.006)	--	(0.008)	--	(0.006)	--	(0.011)	--			(0.006)	--		--	
Fe	0.027	--	0.037	0.1	0.041	1.0	0.086	3.6	0.029	--	0.034	0.1	0.028	0.1	0.4
K	(0.4)	3.2	(0.6)	8.7	(0.4)	--	1.42	4.6	(0.5)	7.2	(0.5)	3.7		7.2	3.7
La	(0.011)	--	(0.014)	--	0.011	--	(0.017)	--	(0.005)	--	(0.016)	--		--	
Li	1.99	9.7	2.09	8.7	2.67	9.7	4.33	8.4	2.50	10.2	1.59	8.6		9.6	1.0
Mg	0.026	--	0.017	--	0.012	--	0.034	--	0.022	--	0.52	--	0.051	--	
Mn							(0.003)	0.2						--	
Mo	(0.012)	--	(0.014)	--	(0.013)	--	(0.015)	--	(0.014)	--	(0.010)	--		--	
Na	9.24	7.1	9.87	6.7	12.3	9.5	19.2	5.9	11.5	7.7	6.69	5.5		6.9	1.1
Nd	(0.11)	9.7	(0.15)	32.7	(0.09)	--	0.14	5.0	0.12	25.7	0.12	--		[21.0]	8.8
Ni	(0.010)	3.2	0.015	13.1	(0.009)	--	(0.021)	2.7	(0.011)	9.4	(0.015)	--		[9.0]	4.6
Rh	0.037	6.0	0.111	32.2	(0.06)	--	0.101	6.6	0.047	13.4	0.078	--		[20.9]	12.6
Ru			(0.02)	--			(0.03)	--						--	
Si	44.8	11.6	34.3	5.4	52.9	12.6	49.5	5.6	38.8	7.3	36.2	6.6	21.8	7.7	2.0
Sr	0.020	2.8	0.020	2.2	0.006	1.8	0.020	1.1	0.016	1.7	0.022	2.7		2.5	0.6
Te	(0.12)	--	(0.12)	--	(0.10)	--	(0.12)	--	(0.10)	--	(0.14)	--		--	
Ti	0.016	--	0.020	0.1	0.018	--	0.116	0.1	0.016	--	0.016	0.1		[0.1]	
Zn		--		--		--		--		--		--	0.013	--	
Zr	(0.006)	0.6	(0.009)	7.8	(0.007)	6.3	(0.010)	3.9	(0.005)	--	0.008	--		--	
^{137}Cs		10.2		10.3						10.3		8.2		9.4	1.2

a. ppm by weight; blank indicates element not detected; parentheses ( ) indicates result near limit of detection.

b. For all elements except ^{137}Cs, $(NL)_i = \frac{(\text{ppm-blank})\ (\text{grams leachate})}{(f_i)\ (\text{SA in meters}^2)\ (10^6)}$.

For ^{137}Cs, $(NL)_i = \frac{(\text{activity/mL})\ (\text{mL of leachate})}{[\text{activity of control sample/mg}\ (\text{mg of specimen})} \cdot \frac{(\text{g of specimen})}{(\text{SA in meters}^2)}$

Dash (--) indicates either $(NL)_i$ <0.05, was negative after correction for blank, or analyses of original material not available to calculate f_i.

c. See Table 11 for basis of acceptable data. Brackets [ ] indicate that the average was calculated using only those specimens for which a value is shown.

TABLE D-9. ^{137}Cs ACTIVITY RESULTS FOR RADIOACTIVE VITRIFIED ZEOLITE LEACH TESTS

Specimen Number	Canister Level	Activity (μCi/mL)	$(NL)_i$ (g/m^2)
1(3)	Z4-3	13.40	6.8
2(3)	Z2-3	13.97	6.5
3(3)	Z5-3	10.69	6.5
4(3)	ZVDP-4	$\sim 2 \times 10^{-5}$	--
6(3)	76-68	3.1×10^{-5}	--
7(3)	Z5-5	9.11	5.4
8(3)	Blank	$\sim 2.1 \times 10^{-5}$	--
9(3)	Z5-1	9.15	5.3
1(14)	Z4-5	16.33	8.3
2(14)	Z4-3	13.76	6.9
3(14)	Z4-1	18.38	8.9
4(14)	76-68	3.4×10^{-3}	--
5(14)	Blank	5.6×10^{-4}	--
7(14)	Z2-5	17.73	10.5
8(14)	Z2-3	18.62	10.1
9(14)	Z2-1	17.96	10.2
10(14)	Z5-3	15.78	8.8
11(14)	Z5-1	18.09	8.9
12a(14)	Z4-3	17.29	8.9
12b(14)	Z4-5	19.17	9.1
15a(14)	Z2-3	17.56	10.3
15b(14)	Z4-1	13.17	6.9
16a(14)	Z5-3	2.85	1.6
17a(14)	Z2-5	15.77	9.5
17b(14)	Z5-1	13.49	7.8
18a(14)	Z5-5	13.78	7.8
1(28)	Z2-3	26.83	11.4
4(28)	76-68	2.2×10^{-4}	--
5(28)	Blank	1.2×10^{-4}	--
6(28)	Z4-3	17.55	9.1
8(28)	Z5-3	14.21	7.5
10(28)	Z4-5	15.81	9.0
11(28)	Z4-1	17.49	10.2
12(28)	Z2-5	19.68	10.3
16(28)	Z2-1	20.04	10.3
18(28)	Z5-1	14.01	8.2