

NNWSI WASTE FORM TEST METHOD FOR UNSATURATED DISPOSAL CONDITIONS

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ABSTRACT

A test method has been developed to measure the release of radionuclides from the waste package under simulated NNWSI repository conditions, and to provide information concerning materials interactions that may occur in the repository. Data are presented from Unsaturated testing of simulated Savannah River Laboratory 165 glass completed through 26 weeks. The relationship between these results and those from parametric and analog testing are described. The data indicate that the waste form test is capable of producing consistent, reproducible results that will be useful in evaluating the role of the waste package in the long-term performance of the repository.

INTRODUCTION

The Nevada Nuclear Waste Storage Investigations Project (NNWSI) is investigating the volcanic tuff beds of Yucca Mountain, Nevada, as a potential location for a high-level radioactive waste repository. One of the topics to be addressed during this investigation is the performance of the waste package components in the repository at the termination of the containment period, i.e., following the 300-1000 year period during which containment of high level waste within the waste package will be substantially complete.¹

The containment period is followed by the isolation period during which the potential for canister breach and subsequent groundwater/waste form contact is significant. Radionuclide release from the waste package would then be possible, although in accord with Nuclear Regulatory Commission regulations¹ the total release rate must be less than one part in 10^5 per year of the total repository inventory of a particular radionuclide after the 1000 year period.

To measure this radionuclide release, a test method is required that incorporates the interactions between the waste package components and the groundwater under realistic repository conditions. Release results obtained in this fashion could then be used as source term data in modeling the long-term behavior of the repository. NNWSI is developing such materials interaction tests and using them to produce waste form release data.

The first test is a rock cup, total submersion test² while the second test, termed Unsaturated Test, is a more realistic simulation of conditions expected in the unsaturated zone in tuff. This report summarizes the Unsaturated Waste Form Test procedure. It also describes parametric and analog tests that provide support data, and then discusses the relationship between the data from the three types of tests.

NNWSI UNSATURATED TEST

Purpose and Description

The purpose of the NNWSI Unsaturated Test is to obtain data on the release of waste components from the waste package in the NNWSI repository environment as it is expected to be after the currently envisioned 300/1000 year containment period has elapsed. Specifically, the test will provide information to be used by the NNWSI project in licensing the NNWSI repository site. The test procedure will (1) provide data that describe the release of radionuclides from a specifically designed glass/container assemblage under strictly controlled test conditions, and (2) provide information concerning synergistic effects that may occur between waste package components. Inherent in this goal is that a specific and reasonable description of the waste package and repository at this time period is available, so that aging effects can be incorporated into the test procedure.

The utility of the data depends on the selection and restriction of the repository conditions and on the materials interactions that are incorporated into the test. The rationale used for selecting test conditions and variables was to address those materials interactions that may exist in a repository during the isolation period. The materials consist of the waste form and the canister overpack. No packing material is included because it is not part of the reference waste package.² The interactions are promoted by contact between water and air and the waste package components. The test provides for several interactions to occur and allows the experimenter to assess the importance of each. During actual repository disposal, each waste package may not be subjected to all possible materials interactions. The experimental design simulates interactions, but does not attempt to model the current waste package conceptual design. A complete description of the development of the test and of its uses and limitations is presented elsewhere.³

Apparatus

The test apparatus is shown diagrammatically in Fig. 1. The components are the test vessel, which provides for collection and containment of liquid and support of the waste package; the waste package

Test Vessel

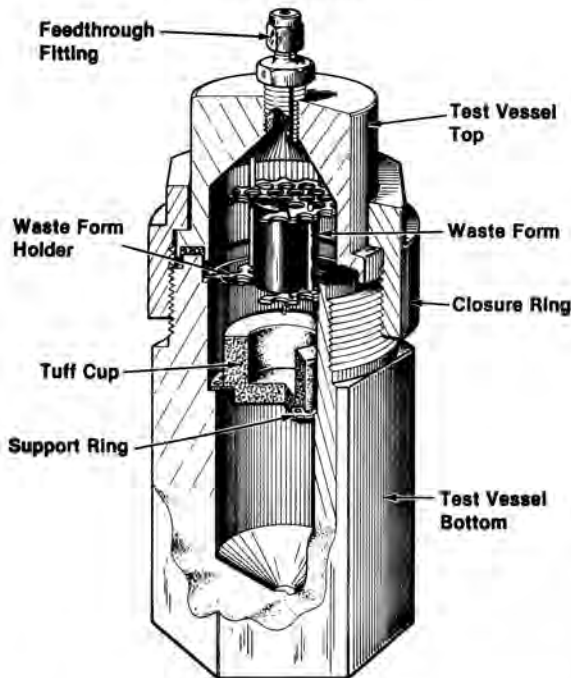


Fig. 1. Test apparatus used for NNWSI Unsaturated testing. Groundwater is introduced through tubing (not shown) that enters the vessel through the feedthrough fitting.

assembly, which consists of the waste form and metallic components representing the canister; the tuff cup, which acts to collect the solution that drips directly from the waste package assembly and which interacts with vapor; the tuff cup supporting ring, which separates the tuff cup from the test vessel; and a solution feed system to inject test water.

The waste package assembly is contacted intermittently by dripping repository water, that has been preequilibrated with tuff at 90°C (EJ-13). The nature and degree of radionuclide release from the assembly is determined by collection and analysis of the water that has contacted the package and by surface analysis of the assembly components. Materials interactions are noted, and secondary alteration products, which influence the nuclide release from the assembly, are identified.

The test schedule incorporates batch and continuous testing. In the batch mode, tests are terminated at selected 13 week time intervals. The test apparatus is disassembled, and analyses of both the solution and components are performed. In the continuous mode the waste package assembly (including liquid associated with the assembly) is transferred to a new test vessel, and the test is continued. Analyses can be done on the solution in the old vessel. Using the continuous testing matrix, replication of solution analysis can be achieved, investigation of the test components is possible at the termination points, yet the test can continue for an unspecified number of test periods or until information most useful to repository evaluation is obtained. A detailed description of the apparatus and test procedure is provided by Bates.³

INTERIM UNSATURATED TEST RESULTS

A series of tests using the Unsaturated procedure are in progress. These tests are being done at 90°C with SRL 165 frit borosilicate glass that is doped with uranium, cesium, and strontium. The glass composition is given in Table I. These tests should provide an indication of the degree of reaction expected and the type of interactions that are important, but since no transuranic elements have been incorporated, this series of tests cannot provide data on the behavior of these important radionuclides. The tests have been completed through the 26 week period using the batch testing mode and through five 6.5 week sampling periods using the continuous testing mode.

It is useful to assess the test results based on a) general observations as to what types of interactions occur, 2) what trends can be established from solution analyses and what release can be attributed to the waste form, and 3) the degree of degradation of waste package components as determined by surface analytical techniques.

General Observations

1. *A priori* it can be judged that the extent of the water/glass contact will greatly affect the amount of release that occurs. Any aspect of the test design that influences this contact is, therefore, a critical test parameter. The appearance of the test components at both the termination and sampling points provides an indication as to how and where interactions occur. When the vessel is opened, the top surface of the test assembly is usually covered by a thin layer of water which evaporates within 30 seconds of opening the test vessel. The sides of the glass are watermarked and there is a considerable amount of standing water around the bottom of the assembly. From these observations, the method of contact between the water and the waste form can be ascertained. After initial contact with the test assembly, the water spreads rapidly over the top surface and down the sides of the glass. Little standing water remains on the top surface because flow is promoted by the tight fit that exists between the perforated 304 L stainless steel (ss) metal section and the glass (Fig. 1). However, at the

TABLE I

Composition of SRL 165 Glass Used in the Unsaturated Test

Component	wt. %
SiO ₂	53.3
Fe ₂ O ₃	12.1
Na ₂ O	10.1
B ₂ O ₃	6.7
Li ₂ O	4.6
Al ₂ O ₃	4.0
MnO ₂	2.9
CaO	1.5
NiO	0.9
MgO	0.8
ZrO ₂	1.2
U ₃ O ₈	1.2
Cs ₂ O	0.17
SrO ₂	0.15
F	0.06
Cl	0.05
Pb	0.05

Analysis by dissolution.

bottom interface more water collects and some interaction occurs. Thus, to some degree the test incorporates water reaction with the test components via both thin film and standing water interactions. These types of interactions are expected to exist during the isolation period.

2. There is considerable reaction between the glass and the bottom ss section as evidenced by discoloration of the glass and the metal. The same type of reaction was also seen in earlier tests.⁴ This reaction is focused mainly, but not exclusively, around the weld-affected area of the metal. A considerable amount of rust colored flakes are present in the solution. A more complete description of the alteration of the glass and metal is presented later.

3. When tuff is present in the tests, standing water is found inside the tuff cup. This is important because it demonstrates that some water is being removed from the waste form/ss assemblage by a dripping process, as opposed to an evaporative process. It is likely that both processes will be operative in the repository. This also offers an opportunity to analyze the interaction between standing water and tuff.

The following sections on solution and solid components analyses expand on these general observations and are presented, not as detailed explanations of the reaction processes, but as overviews to illustrate important aspects of the test.

Solution Analyses

At the termination of a test (13 and 26 weeks), the samples are rinsed with deionized water so that all of the liquid that has contacted the glass/ss assemblage can be analyzed. For the 6.5 week continuous test series only the liquid that is collected in the test vessel is analyzed. Thus, a difference in measured elemental release is expected between the two test types, but the effect of this difference should be minimized in the longer test periods.

Another difference between the test types is that in the terminated 13- and 26-week tests the liquid has contacted the tuff cup, while in the continuous tests no tuff is present. The tuff cup allows the interaction and migration of nuclides with the tuff to be studied, but complicates the interpretation of waste form dissolution data because the tuff, itself, interacts with the liquid and only selected elements can be identified as having been released uniquely from the glass. The total release of important radionuclides can be determined in tests which contain tuff by completely dissolving the rock, if necessary. However, the total radionuclide release can be obtained more easily from the continuous test series, where no tuff is present. One key in analyzing the experiments is to establish the relationship between the two types of tests.

Selected results for both the continuous and terminated tests are shown in Tables II and III. The data in Table II show the amount of each element that was detected in solution, corrected for the contribution due to E-J13 water added during the test. The amount for most elements in solution has increased with time. Exceptions to this are Na and Si in the continuous tests, which are actually being extracted from solution. In no case does the sum of the short continuous tests equal that of the longer term, terminated tests. The ramifications of this, and the

interpretation of these results, are discussed later in relation to the analyses of the solid components of the test.

Table III presents the concentration of elements in the solutions that were collected in the tuff cup at the end of the 26-week test period. The water collects in the tuff as it drips from the glass/ss waste assemblage. The amount that remains in the tuff cup depends on the porosity of the tuff. As this is not constant, so the amount of water collected varies between tests. The concentration of elements in solution depends on what is released from the waste assemblage and what interactions occur with the tuff. While this interplay requires more data for a complete interpretation, the elemental concentrations in the solution are very similar, which is indicative that the same reactions and interactions are occurring in each test.

Solid Component Analyses

Those components available for study at the termination of a test include the glass waste form, the top and bottom sections of the perforated ss waste form holder, and the tuff cup. No component analyses can be done on the continuous test series until the test has been terminated.

Weight change measurement provide an initial indication as to the degree of reaction that has occurred. These data for the glass waste form and both sections of perforated ss are given in Table IV, together with the normalized weight loss, $(NL)_{wt}$, for the glass. This value was calculated using standard methods⁵ and utilized the entire surface area of the glass ($\sim 13 \text{ cm}^2$) in the calculation. This was done because, although the degree of reaction associated with each type of surface, e.g., side, top, or bottom, may be different, the entire surface was contacted by water. The consequences of this approach are being studied in ongoing parametric tests.

The weight loss for replicate glass samples is quite consistent with the total weight loss increasing $\sim 72\%$ between 13 and 26 weeks. The weight changes in the perforated ss sections are less constant, with the observed changes being close to the instrument precision, but indicate the top section has a tendency to lose weight while the bottom section gains weight. These observations correspond well to what is observed when the ss sections are examined optically or with the scanning electron microscopy (SEM).

Optically the top section of the ss appears unreacted, but with the SEM (Fig. 2) a general, Si-rich, coverage is observed on that section of the ss that was in contact with the glass.

In isolated areas, additional reactions have occurred resulting in discrete products containing Fe/Si, Na/Si/Ca, and Si. The distribution of these products are random and limited, and to date they have not been identified. The region of the top ss section where there has been glass contact appears unreacted after 13 weeks, but some Fe/Si products are evident after 26 weeks.

Visual observation reveals that the bottom ss section has reacted. The bulk of the reaction occurs in the weld-affected area where the vertical support rods are attached (Fig. 1). The weld-affected area is not uniform between samples and likely introduces some variance to the results. Reaction in this region results in extensive coverage of iron and

TABLE II
Solution Analyses^a for the NNWSI Unsaturated Test

Test Type ^d	Element ^{b,c}							
	B	Ca	Li	Mg	Na	Si	Sr	U
13 week, terminated	3.9	21.5	5.9	4.4	(8.0)	(13.8)	0.4	0.1
13 week, terminated	5.9	30.3	6.2	5.8	27.7	27.0	0.6	0.3
Sum of two 6.5 week continuous	3.3	3.8	10.0	1.2	(31.7)	(27.7)	<0.3	<0.05
26 week, terminated	15.1	35.5	10.9	7.4	46.9	107.1	0.8	1.1
26 week, terminated	15.5	35.7	11.1	7.5	61.6	112.5	0.7	0.7
Sum of four 6.5 week continuous	4.4	7.3	16.4	2.1	(60.2)	(50.4)	<0.3	0.1

^aSolutions were analyzed by ICP except for U which was done by fluorescence.

^bThe results are presented as the total of each element (μg) analyzed in solution.

^cThe results have been corrected for the elemental contribution due to E-J13 water added during the test, () indicates lesser of an element is in solution than originally added.

^dDuplicate tests were terminated at 13 and 26 weeks. Duplicate tests were also run in the 6.5-week continuous test series, and an average of two tests is reported.

TABLE III
Concentration of Water Contained in the Tuff Cup

Solution Type	Element (ppm)							
	B	Ca	Li	Mg	Na	Si	Sr	U
E-J13	0.18	4.50	0.06	0.39	46.60	35.30	0.03	0.004
26 week terminated	4.2	9.9	2.3	1.5	50.3	76.2	0.17	0.07
26 week terminated	4.2	8.8	2.3	1.5	50.3	76.6	0.15	0.06

TABLE IV
Component Weight Changes in NNWSI Unsaturated Tests

Test #	Test Description	Weight Change, ^a μgm			(NL) _{wt} , g/m^2
		Top ss	Bottom ss	Glass	Glass
1	13 week terminated	(160)	360	(350)	0.26
2	13 week terminated	40	20	(320)	0.25
3	26 week terminated	(50)	80	(630)	0.47
4	26 week terminated	(20)	30	(530)	0.40

^a(Values) indicate weight loss, estimated precision $\pm 20 \mu\text{gm}$.



Fig. 2. Dual photomicrograph of the alteration that occurs on the top ss section that has contacted the glass. Magnification 1000 and 10,000X.

chromium silicate nodules (Fig. 3). However, SEM examination reveals that some degree of reaction is widespread outside the weld-affected regions. Here, the surface is continuously covered with small Si-rich deposits (Fig. 4), and intermittently covered with other reaction products. The ss that has not contacted the glass is covered by reaction products to a much lesser degree.

Visual observation also reveals that the bottom of the glass is reacted, especially in regions where there is corresponding reaction on the ss. Examples of the type of reaction products observed are shown in Fig. 5, where Si-rich strands are shown covering a region of iron silicate nodules. There is widespread appearance of a uranium-containing phase (Fig. 6). This phase contains Ca, P, U, and Si, as determined by energy dispersive X-ray analysis (EDS), but to date, X-ray diffraction has not yielded a pattern, and the phase is unidentified. Figure 7 shows the

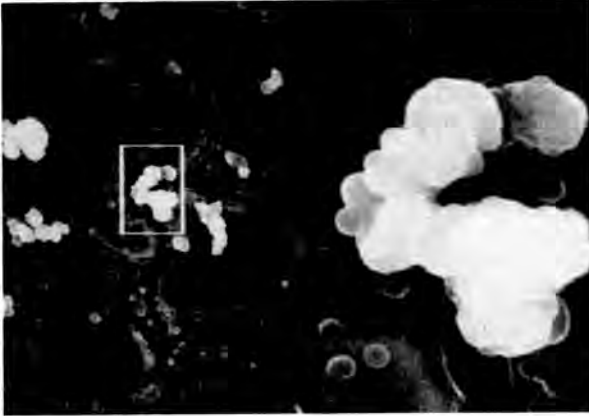


Fig. 3. Nodules that form on the weld-affected region of the bottom ss section. Magnification 2500, 12,500X.

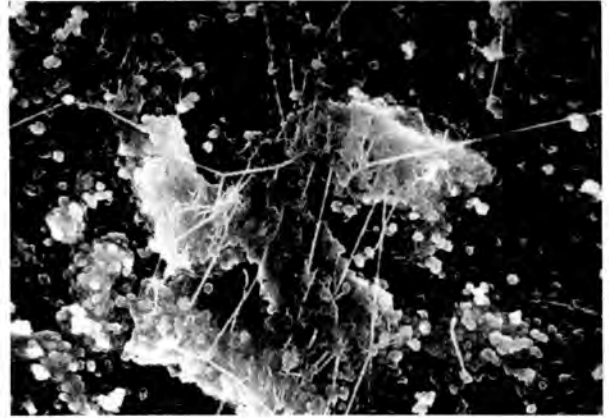


Fig. 5. A photomicrograph of alteration that occurs on the bottom surface of glass that has been in contact with the weld-affected ss. Magnification 3000X.

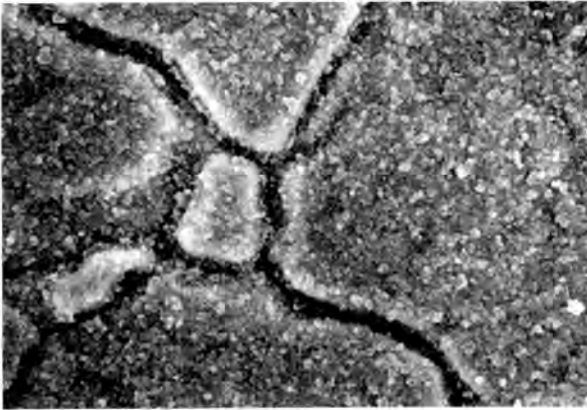


Fig. 4. General coverage on the bottom ss section that was in contact with the glass. Magnification 5000X.



Fig. 6. A photomicrograph of a uranium-containing phase surface of glass. Magnification 10000X.

surface in a region not affected by any localized reaction. The surface is enriched in Fe, Mg, and Al and depleted in Na and Si. The circular forms are an outgrowth and have the same composition as the surface.

The top surface of the glass generally shows less evidence of localized reaction and alteration product formation than the bottom and has the appearance shown in Fig. 7. The identification of all the reaction products is an important part in understanding the alteration process that will likely control the eventual release of radionuclides. A more complete description of these phases is being prepared.⁶

Discussions

Testing in this matrix has been completed through 26 weeks so only a preliminary interpretation of the results is warranted. However, several important issues can be addressed and other issues that merit close attention can be identified.

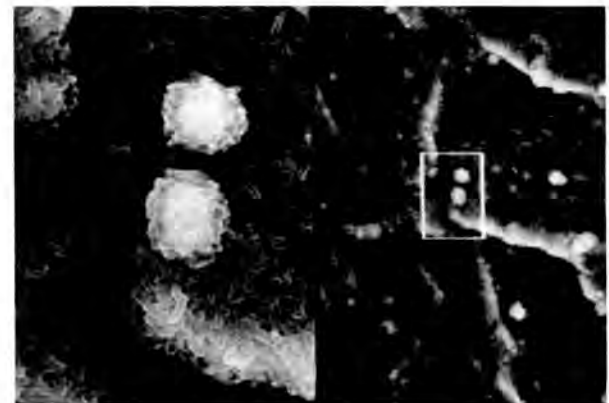


Fig. 7. A photomicrograph of the general surface of reacted glass. Magnification 2000X.

The reproducibility of the reaction process as determined from the physical appearance of the test components, the weight loss of the waste form, and the solution analyses of both the terminated and continuous tests is good enough so that credence can be given to the data, and that observed trends may be interpretable. Trends would provide some measure of the extent of reaction with time, so that it can be determined whether the data can be fit into models that will establish the behavior of the repository for long time periods.

Equally important are that 1) the interactions between the test components (glass, ss, water, tuff) appear to affect and control the reactions that occur and 2) that a relationship between what is found in solution and what is observed on the test components can be established. For example, the extent of reaction of the glass can be estimated by the $(NL)_{wt}$. Yet a value obtained in this fashion must be tempered by the possibility that reaction products may form that incorporate components from the ss and the water, and these products could increase the final weight.

It would be useful to identify a marker element that is not incorporated into alteration products and that does not interact with the tuff. One possibility is Li. The amount of Li found in the water in both the continuous and terminated tests correlates well with what would be expected for an element that does not interact. Li levels found in the continuous tests are greater than that found in the terminated tests because ~1 mL of water is retained in the tuff and remains unanalyzed. However, for the other elements the evidence is that they may be retained on the glass/ss waste assemblage or come from the tuff. It will be necessary to correlate the results of surface science analysis of the reacted surfaces with the solution results to adequately assess the degree of

waste form reaction. However, based on these preliminary data the release from the waste assemblages after 26 weeks is 0.43 g/m² based on weight loss, 0.58 g/m² based on Li release, and 0.08 g/m² based on U release.

Parametric Testing

The NNWSI Unsaturated Test requires that a rigid set of controls be placed on the test conditions. Yet, there are many aspects of the test that need to be further studied, including the effect of removing the perforated ss sections, varying the ratios of the top and bottom surface area that of the sides, or varying the rate and amount of water that contacts the waste form. Changes in these process parameters are being studied in parametric tests.⁴

The first set of parametric tests⁴ is investigating the release from a glass waste form only. The goal is to establish a known release from the glass with no contact with perforated ss sections. The test is being done in a Teflon[®] vessel, and the waste form is positioned on a Teflon[®] stand so that the release of all elements can be monitored. Otherwise, the test parameters are identical to the Unsaturated Test. This test has been completed through 39 weeks, and selected results are given in Table V.

The striking points in these data are that the waste form is gaining weight, likely due to the extraction of Ca, Mg, and Si from the J13 water, and that except for Fe, Na, and Si, the total amount of elemental release is similar in the continuous and terminated tests.

The first point is confirmed by visual examination of the top glass surface which has a white, crusty appearance, and with SEM where large quantities of Ca-, Mg-, and Si-containing deposits are observed. These deposits form because without any ss present the water is able to remain on the top surface longer, and since the Teflon[®] vessels do not maintain a leak-tight atmosphere, may be subject to evaporative forces.

These observations help interpret data from the Unsaturated Test where the elemental release in the continuous and terminated tests do not match. It appears that Na, Si, and Fe may be forming phases on the glass that are washed off during the rinsing process. In fact, the remnant of an unstable Na/Si phase has been observed on the glass surface with the SEM. This would suggest that in the Unsaturated Test the variances for the other elements are caused by interaction with the tuff.

ANALOG EXPERIMENT

In order to establish the relationship between the Unsaturated Test and the repository, an analog test is being performed. The analog test attempts to more closely simulate anticipated repository conditions than the Unsaturated Test and is necessary because some of the constraints required in the controlled Unsaturated Test procedure may not exist in a repository. If these test constraints cause a significant deviation between the analog and Unsaturated Test results, then the data obtained by use of the Unsaturated Test may not be a reasonable repository simulation and should be questioned if used in repository modeling.

The analog test³ utilizes a tuff rock core the shape of a right circular cylinder, 5" in length and 2-1/2" in diameter, as a reaction vessel. Each core is cut diametrically to give two sections. A cavity is machined in the larger section to accommodate a test assembly similar to that used in the Unsaturated test. The dimensions of the cavity are similar to the inside of the Unsaturated test vessel, except that the upper section of tuff, which acts as the vessel lid, had a flat lapped surface.

The test is initiated by placing the waste package in the cavity and placing both sections of J-13 saturated tuff into a Teflon[®] sleeve. The mating surfaces of both tuff sections have lapped faces which produce a good seal. The Teflon[®] sleeve is placed in a stainless steel holder and end caps secured over the top and bottom of the tuff core. The end caps are tapered so they only contact the tuff along the outside edges. The end caps are connected to inlet and outlet tubing. The Teflon[®] containment is sealed by water which pressurizes the Teflon[®] jacket to 1600 psi. This provides for flow through the tuff core and not at the Teflon[®]/tuff interface.

DWPF glass spiked with with ¹³³Ba, ¹³⁷Cs, and ¹⁵²Eu was used as the waste form. ¹³⁷Cs was used to represent highly mobile, easily leached elements, but is not expected to be a significant radionuclide during the isolation period. ¹³³Ba was used to represent the alkaline earth group of elements. Again few of these elements will be of concern during the isolation period. ¹⁵²Eu was used to represent fission products and actinide elements that are slowly removed from the glass and will be important during the isolation period.

TABLE V
Parametric Test Results

Test Type	Duration (weeks)	Δ wt (mgms)	Total Elemental Release (μ g) ^a							
			B	Ca	Fe	Li	Mg	Na	Sf	U
Terminated ^b	6	0.1	7.2	(8.8)	1.0	9.8	(1.2)	34.3	(11.3)	0.7
	13	0.7	24.0	(24.5)	1.3	23.9	(3.8)	84.5	(26.7)	1.8
	26	1.2	10.1	(49.6)	2.4	20.4	(7.9)	38.9	(28.5)	1.5
	39	1.5	21.5	(73.8)	2.1	27.0	(12.0)	77.0	(44.0)	2.9
Continuous ^c	6	-	8.6	(9.2)	2.0	2.7	(1.3)	(23.7)	(21.8)	0.3
	13	-	13.8	(20.6)	3.8	5.7	(2.7)	(46.9)	(49.2)	0.8
	26	-	22.1	(41.4)	6.6	16.4	(5.7)	(44.1)	(91.1)	1.8
	39	-	27.0	(62.3)	10.0	26.4	(9.3)	(10.0)	(110.8)	2.9

^aThe values have been corrected for the contribution due to J13 water, (value) indicates a net decrease in the amount in solution.

^bThe terminated tests represent the results of one test per sample period.

^cThe continuous tests represent the average of values from two tests, except for B which is one test.

Three tests were initiated, with one being terminated after 13 weeks. Detailed results of this test are presented elsewhere,^{3,4} but there was good agreement between both the analog and Unsaturated Test regarding the appearance of the waste assemblage and the extent of reaction. The test components were gamma-counted for long periods of time (10^6 sec) to determine the amount and directionality of radiotracer release. In relationship to the waste form weight loss the releases were: $(NL)_{wt} = 0.1 \text{ g/m}^2$, $(NL)_{Cs} = 0.1 \text{ g/m}^2$, $(NL)_{Ba} = 0.09 \text{ g/m}^2$, and $(NL)_{Eu} = 0.03 \text{ g/m}^2$. Most of the ^{133}Ba was associated with the top and bottom ss sections, while $\sim 30\%$ was found on the tuff. 98% of the ^{152}Eu was associated with the ss sections and $\sim 2\%$ with the bottom tuff, while the ^{137}Cs was found mostly associated with the bottom tuff. Very little of any radionuclide was detected in the rinse solution, and no radioactivity was found on the walls or top section of the tuff cavity.

CONCLUSIONS

Preliminary results of tests done using the NNWSI Unsaturated Test method indicate that consistent, reproducible results can be achieved. A close relationship between the laboratory environment of the Unsaturated method and a more realistic repository environment, as simulated in the analog tests, is also indicated. Combined with the results from parametric testing, these observations provide confidence that data acquired in longer term Unsaturated testing will establish trends that will be applicable to modeling long-term NNWSI waste package behavior. Adaptation of the Unsaturated Test method for use with spent fuel is in progress.

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