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A GROUNDWATER STREAM EXPERIMENT FOR THE WASTE ISOLATION PILOT PLANT

by

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ABSTRACT

This project was conducted to evaluate the practicality of using laboratory groundwater stream experiments to model a hydraulic breach of a nuclear waste repository located deep in a bedded salt environment. A test plan is included in this report that gives details of the apparatus, rocks, solutions, and analyses to be used in a groundwater stream experiment.

Preliminary experiments revealed the essential impermeability of halite; only a small concentration of water (about 75 ppm) moved in halite by diffusion, with a coefficient of 2.0×10^{-7} cm²/s. From work completed in this program, groundwater stream experiments appear to be a practical method of establishing the chemical interactions that would occur in a breached repository in bedded salt.

I. INTRODUCTION

The potential for radionuclide migration by groundwater flow from a breached nuclear-waste repository depends on the leaching process and on chemical changes that might occur as the radionuclide moves away from the repository. Migration involves the interactions of leached species with (1) the waste and canister, (2) the engineered barrier, and (3) the geologic materials surrounding the repository. Some of these interactions would occur in the radiation and thermal gradients centered on the solidified waste. Rather than trying to identify the important interactions and then study them individually, we attempt to consider all potential interactions, using experiments that integrate repository components in order to simulate a groundwater stream that infiltrates a breached repository. This report describes potential experimental work by the groundwater stream simulation method.

This work, initiated using program development funds of the Argonne National Laboratory, was extended with support from Sandia Laboratories, Albuquerque, NM, for the Waste Isolation Pilot Plant (WIPP) program. Described are (1) a test plan for a stream-simulation experiment, (2) apparatus developed to implement the test plan, and (3) preliminary experiments performed to examine experimental aspects suggested for the stream-simulation approach.

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II. PRIOR WORK

The stream-simulation experiments are an elaboration of the leach migration experiments conducted for the Waste Isolation Safety Assessment Program (WISAP) in which solid waste at elevated temperature and geologic media were combined into one groundwater stream [SEITZ-1979A, SEITZ-1979B, SEITZ-1979C, SEITZ-1980A]. The approach of combining the repository components in one groundwater stream has recently been discussed for the Waste Rock Interactions Program [SEITZ-1980B, COLES]. In the approach used to date, naturally jointed samples of impermeable rock [SEITZ-1981] were used, as well as permeable rock.

In other studies done prior to the leach-migration experiments, a part of a nuclear waste repository (geologic media, engineered barrier, canister, or waste form) was selected for individual scrutiny. It was hoped that the findings could be integrated by computer model with findings pertaining to other parts of the repository. Although some integration can be expected with this approach, there is little hope of identifying the synergisms that are known to occur when repository components are allowed to interact. For example, it was observed in leach-migration experiments using a simulated waste glass and rock cores that the migration of cesium in the rock depended upon the quantity of cesium leached from the waste glass [SEITZ-1979A].

III. THE WIPP REPOSITORY

The Waste Isolation Pilot Plant (WIPP), to be located in salt beds 42 km east of Carlsbad, New Mexico, is to be used primarily for the geologic isolation of defense transuranic (TRU) waste. This use constitutes the basis of the experimental design for this program.

If WIPP is a geologic repository for TRU waste, an important consideration for laboratory integration experiments is the effect on the leaching and migration process of short range, heavily ionizing radiation (i.e., alpha decay). Highly penetrating radiation (gamma radiation) is not a major consideration with TRU waste and need not be considered in the experiments. Thermal gradients are small, in comparison to those expected for a high-level-waste repository. The temperature at the surface (below diurnal and seasonal variation) is about 28°C and increases to about 40°C at the 663-m (2175-ft) depth of the repository [POWERS]. The TRU waste disposed of in the WIPP repository may be incinerator ash, wastes solidified in glass, or slags made by melt-refined metals [GERDING].

IV. TEST PLAN

A test plan was developed in this program for a stream-simulation experiment that would physically model a hydraulic breach of a nuclear waste repository located deep in a bedded salt environment. The stream-simulation experiment could be performed to determine the utility of physical models in assessing the safety of the proposed WIPP. The test plan is presented in the supplement of this report.

The test plan is written for an experiment in which a brine solution flows at elevated temperature over components of a waste repository (canister metal, engineered barrier, and radioactive waste form), it being assumed that radionuclides can be leached from the waste form and carried downstream. The brine, containing leached radionuclides, is then passed through rocks of the strata that overlie the repository horizon. Our ability to create a flow of brine through repository components and through rocks associated with the bedded salt was examined in several preliminary experiments that were run before the test plan was formulated.

V. EXPERIMENTAL APPARATUS CONSTRUCTION

The apparatus needed for the groundwater-stream experiment is described in Appendix B of the test plan. To give it corrosion resistance to brine, this apparatus is made primarily of the alloy, Hastelloy-C-276, and of the fluoride resin, Teflon.

Only the pressure transducers (not made of Hastelloy) are susceptible to corrosion by brine. To isolate the brine from corrodible parts of the pressure transducers, we filled the transducers with silicon grease. The transducers' outputs were not affected by the grease, as indicated in tests of the transducers using calibrated dial gauges. Holders were constructed to hold rock cores in inert materials and to allow the core to be removed after it was used in an experiment. The holder design is shown in Fig. S-5 of the supplement to this report. Rock core holders were made for two sizes of rock core, 2.20-cm diameter by 8.89 cm long and 6.82-cm diameter by 15.25-cm long. The holders for the smaller cores were made of Monel-400 alloy with Teflon sleeves.

VI. PRELIMINARY EXPERIMENTS

A. Materials

Rock cores used in the experiments were machined from blocks of salt obtained from the Mississippi Chemical Corporation potash mine located about 15 miles northwest of the proposed WIPP site. The two halves of the rock salt fissure were cut from two different cylindrical rock cores so that the halves fitted to form a cylinder with no material missing that would have represented the width of a saw blade.

Hectorite clay for the experiments was obtained from Hector, California, through Wards Natural Science Establishment.

B. Permeability of Solid Rock Salt and Hectorite - Preliminary Experiment 1

To examine the ability to create a flow of groundwater through rock salt, we applied water at 4.5 MPa (650 psi) to the end of a right circular cylinder of McNutt halite (6.83-cm diameter by 15.2 cm long) in a Hastelloy rock core holder (Fig. S-5). The outside confining pressure was maintained in excess

of 12.4 MPa (1800 psi) to prevent water from bypassing the core. In 8 days, no water was observed to have been transmitted through the core (if less than 2 mL of water had been transmitted, it might not have been detected). Based on observations in this experiment, the permeability was calculated to be less than 29 microdarcy.

The end pressure was raised in excess of the confining pressure, and flow around the rock core began to occur. The confining pressure dropped but was raised periodically using the manual pump. In 6 days, 197 mL of water passed around the core, and the weight loss of the core was 80 g from an original weight of 1.21 kg. The Teflon sleeve was severely distorted by the confining fluid and conformed to a flat side of the core that had formed by dissolution.

Hectorite clay, nominally

has been considered as a possible engineered barrier in the WIPP repository [NOWAK-1980B]. To test our ability to establish flow through hectorite, we pressurized one end of a 15-cm-long, 1-cm-dia column of water-saturated hectorite with water at 4.5 MPa (650 psi). The hectorite was held in the column with a stainless steel metal frit. We observed no flow of water in two weeks, indicating that the packed hectorite is essentially impermeable to water. The ability of clays to form practically impervious barriers makes them potentially useful in repository construction [PUSCH].

A potential failure mode for a clay barrier is washing out of the clay, allowing the water to bypass the barrier. This failure mode will be adopted for modeling in stream-simulation experiments since it has been found difficult to cause flow directly through the clay.

C. Fissured Rock Salt Permeability - Preliminary Experiment 2

Because of the low permeability of McNutt salt, a second infiltration experiment was performed, using two halves of right circular cylinders of the salt. In the experiment, water that had passed through a 1.0-m-long column of crushed McNutt salt (passing through the salt for ten days) was applied to the bottom of the two core halves confined in a rock-core holder, as depicted in Fig. 1. Confining pressures on the salt core for the first week were between 26.4 MPa (3.83×10^3 psi) and 30.5 MPa (4.43×10^3 psi, corresponding to the lithostatic pressure at 1150 m for rock of density 2.70 g/cm^3). The pressures in the second and third weeks ranged from 14.8 MPa (2.15×10^3 psi) to 18.9 MPa (2.75×10^3 psi). The water was pumped with a Varian Model 8500 syringe pump at 1.0 mL/h until the pressure on the top of the rock salt core reached the preset limiting value of 5.2 MPa (751 psi, corresponding to a hydrostatic head of 520 m) and ranged between 4.96 MPa (719 psi) and 6.71 MPa (974 psi) during the three weeks. No brine was observed to have passed through the fissured salt core.

Fig. 1. Schematic of an Experiment for Conducting Flow through Fissured Rock Salt that is Subjected to Confining Pressure

Upon disassembly after the three-week-long experiment, the core was found to be in one piece; the two halves had been recemented together. The Teflon sleeve was closely examined and was found to have held the rock core very well, as indicated by impressions of the core and end plugs on the sleeve. No brine was present in the distilled water that confined the Teflon sleeve around the core. From this data (and assuming a viscosity of salt water of 1.05 cP), we have calculated the permeability of the split rock salt core to be below 10 microdarcy.

D. Rock Salt Infiltration by Water - Preliminary Experiment 3

An experiment was set up to investigate the penetration of water into rock salt. Because of the low permeability of the salt, we used tritiated water that could be monitored radiochemically to provide us with a sensitive measure of the penetration of water into the salt.

1. Water Infiltration

Four cores of rock salt (8.89 cm long and 2.20-cm diameter from the McNutt formation) were obtained from blocks of salt taken from the Mississippi bed of the New Mexico evaporite structure. The cores exhibited heterogeneities in mineral constituents, with the third core showing the largest amount of rust-colored mineralization on its surface.

The experiment consisted of pressurizing the four cores (each 8.89 cm long and 2.20-cm dia) of the McNutt rock salt with tritiated brine. Four rock salt cores were assembled in holders, and pressure (14 MPa, 2000 psi) was applied to the Teflon core sleeves with a Haskel MS 188 pump. This pressure was used to confine the Teflon sleeve around the sides of the core, making a nearly perfect seal. A 2-mL tritiated brine solution (80% saturated McNutt salt solution, 20% distilled water, and 20 μCi tritium as water per milliliter of solution) was added to the top of each core. This solution had a measured specific activity of 2.27×10^7 d/min per milliliter. The atmosphere above the solution was purged with argon, and then argon was applied at a pressure of 760 kPa (110 psi). The experimental setup is depicted in Fig. 2. The experiment was performed at an ambient temperature of 21°C.

Fig. 2.

Schematic of the Experimental Setup to Study Water Penetration into McNutt Salt Cores

The sleeve confining pressure and the argon pressure were monitored by pressure transducers and were recorded on a strip chart for the 137-day duration of the experiment. The argon pressure was stable or varied slowly [ranging from 710 kPa (103 psi) to 785 kPa (114 psi)] during the experiment.

The sleeve confining pressure varied considerably during the experiment, as depicted in Fig. 3. The pressure oscillated at first, with a period of 20 to 30 h. No correlation was found between variations in the confining pressure and (1) ambient temperature, (2) time of day, or (3) the line air pressure that maintained the confining pressure. The four cores were pressurized for durations of 31, 61, 103, and 137 days. After removal of the second core from the pressurized lines (after 61 days), the confining pressure became considerably more stable, suggesting that the oscillations had been caused by a peculiarity of the second core holder.

Fig. 3. Sleeve Confining Pressure during the Water-Penetration Experiment. Water infiltration of rock salt

2. Experimental Results

A rock core was removed from the pressurized lines by isolating the core holder with valves, releasing the argon pressure, then removing the tritiated water from above the rock core. The confining pressure was then lowered, and the core was removed from the core holder. The bottom exit tube was inspected for any water, then rinsed to produce a sample for tritium analysis. Tritium detected in the rinses of the first two rock cores was attributed to solution that had not been completely removed from above the rock core but had passed along the outside of the core after the sleeve confining pressure was lowered. The bottom of the third and fourth rock cores was rinsed before the sleeve confining pressure was removed, and these rinses contained no detectable tritium.

The rock cores were sectioned by the use of a diamond saw with Freon coolant; then the sections were each dissolved in water, and aliquots of each solution were analyzed for tritium by scintillation detection. The sectioning of all cores was very similar to that for the first core (depicted in Fig. 4). The tritium analyses of the various rock segments are listed in Table 1. Specific activities of rock segments are plotted in Fig. 5 against depth of the segments within the core.

Fig. 4.

Sectioning Diagram for the First Rock Salt Core. The width of the saw cut (the kerf) is about 0.4 mm.

Fig. 5. Specific Activity of the Segments of the Rock Core Relative to their Depths in the Core. The smooth curves are solutions to Fick's law of diffusion.

Table 1. Weights, Activities, and Lengths of Segments for the Four Rock Cores

Core Number	Segment Number	Segment Weight, g	Tritium Activity, d/min·g	Calculated Segment Length, ^a cm
1	1 ^b	1.24	1.4×10^4	0.30
1	2 ^b	2.33	2.0×10^3	0.56
1	3 ^b	4.10	<10	1.00
1	4 ^b	3.97	<10	0.96
1	5 ^c	7.39	<10	0.89
2	1 ^b	1.41	6.4×10^4	0.34
2	2 ^b	2.52	2.1×10^3	0.61
2	3 ^b	3.91	1.7×10^3	0.94
2	4 ^b	4.11	1.3×10^3	0.99
2	5 ^c	9.75	2.2×10^3	1.18
2	6 ^c	8.80	510	1.96
2	7 ^c	11.43	430	1.38
2	8 ^c	17.51	360	2.11
3	1 ^b	1.49	1.2×10^5	0.36
3	2 ^b	2.39	6.3×10^3	0.58
3	3 ^b	2.69	1.7×10^4	0.65
3	4 ^b	4.34	6.0×10^3	1.05
3	5	15.54	510	1.88
3	6 ^c	7.33	160	0.88
3	7 ^c	10.36	26	1.25
3	8 ^c	14.18	145	1.71
4	1 ^b	1.58	2.2×10^4	0.38
4	2 ^b	1.97	7.1×10^3	0.48
4	3 ^b	3.21	3.4×10^3	0.77
4	4 ^b	7.03	1.8×10^3	1.70
4	5	4.71	1.3×10^3	0.58
4	6	6.28	670	0.76
4	7	7.49	410	0.90
4	8	9.41	310	1.14
4	9	12.89	320	1.56

^aThese segment lengths were calculated from the segment weights, using a rock salt density of 2.18 g/cm³.

^bThese segments each consisted of one-half of the circular shape of a cylinder. Other segments consisted of cylinders sampled along the length of a rock core.

^cThese segments of the cores were sectioned and analyzed at the same time (after 137 days) as the segments of core No. 4.

3. Mass Balance Calculations

A mass balance was calculated for tritium and for the weight of the first core before and after cutting. The about 2 mL of solution recovered from the top of the first core had a specific activity of 2.19×10^7 d/(min·mL). This activity is 3.5% less than the activity of the original solution; the difference could have been caused by an increase in volume due to even slight dissolution of salt from the core. Also, since the analytical error was 1-2%, the difference is small, perhaps nonexistent. The tritium activities of rinses of the bottom plate and the outlet tube of the rock core holder for the first core were 2.48×10^3 and <10 d/min, respectively. The amount of tritium in the segments (Table 1) and in the rinses, therefore, is seen to be an insignificant fraction of the tritium introduced at the top of the first rock core.

The calculated and measured lengths of four segments of the first rock core are given in Table 2. Because some segments had irregular fracture surfaces, the weights of these segments were used to calculate their lengths.

Table 2. Measured and Calculated (from weight) Lengths of Four Segments of the First Rock Core

Segment Number	Measured Length, cm	Calculated Length, cm
1	0.32	0.30
2	0.64	0.56
3	0.95	1.00
4 ^a	0.95	0.96

^aThis segment had an irregular fracture surface.

4. Experimental Interpretation

The results listed in Table 1 and illustrated in Fig. 5 indicate that although the water had penetrated to a depth of only 1 cm in the first 31 days, the water penetrated the complete length of the 8.85-cm-long core in 61 days. Nonetheless, the concentrations of tritiated water are always lower at the bottom than at the top of the cores, and the gradient established in core No. 2 in 61 days remained fixed for longer core pressurization, as indicated by the similar tritium distributions in cores 3 and 4. These results suggest that after 61 days, a steady-state transfer process was established in which water moved from the reservoir at the top of the rock core down through the core to the bottom, where it evaporated.

Using this interpretation, we fitted the tritium data in Fig. 5 with a solution to Fick's law of diffusion. For a steady-state condition, with a source of water at one end of the core and a sink at the other (evaporation), the concentration of water will vary linearly along the length of the core. This distribution, fitted to the data in the center of the rock core, is plotted as a continuous line in Fig. 5. (Also drawn is the steeper curve of tritium distribution after 31 days, calculated using the diffusion coefficient determined below.) The steady-state distribution is consistent with the data from segments of cores 2, 3, and 4, excepting the top surface segments. A probable cause of the high tritium concentration of the top segments is recrystallization, in which tritium is incorporated as the hydrated fraction of newly formed minerals. This recrystallization effect is superimposed on, and would have no effect on, the tritium distribution expected from diffusion alone.

The results shown in Fig. 5 are not consistent with water permeation caused by the hydraulic gradient imposed on the rock cores in which the water would be expected to saturate the cores uniformly from top to bottom.

From the steady-state diffusion curve (Fig. 5), we estimated that the concentration of water diffusing through rock salt in contact with water is 7.5×10^{-5} g of migrating water per gram of rock salt or 75 ppm. This estimate was obtained by using the intercept of (1) the postulated steady-state diffusion curve [1.4×10^3 tritium d/(min·g)] and (2) the activity of the brine [1.89×10^7 d/(min·g)]. The activity of the brine was calculated from its activity by volume [2.27×10^7 d/(min·mL)] at an estimated density of the brine of 1.2 g/mL.

The average concentrations of water incorporated by recrystallization at the top of the rock cores were estimated (by a calculation similar to that described above) to be 0.07, 0.33, 0.63, and 0.11% by weight for cores 1, 2, 3, and 4, respectively. We would expect that most of the recrystallized water would occur very near the surface of the rock cores so that the concentrations at the surface would be greater than the calculated average values for the entire segments.

The amount of water in recrystallized minerals appears to be readily modified. Prior to sectioning and analysis, rock core 4 was exposed to lighting for about 10 min while it was photographed. The small amount of recrystallized water on the surface of this core in comparison to cores 2 and 3 (Table 1) may have been due to evaporation losses.

Again discussing the diffusion-controlled process, we can estimate the diffusion rate of water in the rock salt from the tritium profile seen in the first rock salt core. The amount of water entering the rock cores would increase with time, then remain constant once steady state is reached.

At steady state, the amount of water, M_{∞} , entering the core is given by the formula

$$M_{\infty} = 1/2 l C_0$$

where C_0 is the concentration at the upper surface, and l is the length of the rock core [CRANK, p. 50]. Based on the data reported here, $C_0 = 7.5 \times 10^{-5}$ g/g and $l = 8.89$ cm, so that

$$M_\infty = 3.33 \times 10^{-4} \text{ cm}$$

or 7.27×10^{-4} g/(cm² of rock surface), assuming the density of rock salt to be 2.18 g/cm³.

The diffusion rate of water through the rock salt can be estimated from the amount of water that penetrated rock core 1 in the first 31 days of the experiment. This value is not equal to the total amount of water determined to have entered the first core since a significant fraction of this water is water of hydration. However, the amount of water subject to diffusion that enters each square centimeter of the top of rock core 1 in 31 days can be calculated approximately by

$$M_{31 \text{ days}} = 2 C_0 (W_{1+2}) / A$$

where C_0 is the maximum concentration of migrating water (equal to 7.50×10^{-5} g/g at the top surfaces of the rock cores), W_{1+2} is the weight of the two top segments of rock core 1, and A is the cross-sectional area of the core (equal to 3.80 cm²). Using this formula,

$$M_{31 \text{ days}} = 1.4 \times 10^{-4} \text{ g water/cm}^2$$

The ratio of water that entered the core in 31 days to the amount that entered at steady state is

$$M_{31 \text{ days}} / M_\infty = 0.194$$

This ratio can be used to calculate the diffusion coefficient for water in the rock salt core. From [CRANK],

$$\frac{M_t}{M_\infty} = 1 - \frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp \left\{ -D(2n+1)^2 \pi^2 t / l^2 \right\}$$

where D is the diffusion coefficient, t is the duration of water diffusion, and l is the length of the core. The ratio can be approximated by

$$\frac{M_t}{M_\infty} = 1 - \frac{8}{\pi^2} \exp \left\{ -D\pi^2 t / l^2 \right\} - \frac{8}{9\pi^2} \exp \left\{ -9D\pi^2 t / l^2 \right\}$$

Using $M_t / M_\infty = 0.194$, $t = 2.68 \times 10^6$ s (31 days), and $l = 8.89$ cm, we find

$$D = 2.0 \times 10^{-7} \text{ cm}^2/\text{s}$$

Now, the transport rate of water through the rock salt cores, F , can readily be calculated by Fick's law of diffusion

$$F = -D \frac{dC}{dx}$$

where C is the concentration of diffusing water along the length of the core. At the top of the core ($x = 0$), C is equal to C_0 . Here

$$\begin{aligned} C_0 &= 7.50 \times 10^{-5} \text{ g/g} \times 2.18 \text{ g/cm}^3 \\ &= 1.64 \times 10^{-4} \text{ g/cm}^3 \end{aligned}$$

$$\text{then } \frac{dC}{dx} = C_0/l = \frac{1.64 \times 10^{-4}}{8.89} = 1.84 \times 10^{-5} \text{ (g/cm}^4\text{)}$$

$$\begin{aligned} F &= 2.0 \times 10^{-7} \text{ (cm}^2\text{/s)} \times 1.84 \times 10^{-5} \text{ (g/cm}^4\text{)} \\ &= 3.68 \times 10^{-12} \text{ g/cm}^2\cdot\text{s} \end{aligned}$$

Using the cross-sectional area of a core (3.80 cm^2), we would expect that for the core that was pressurized the longest (137 days), less than $0.14 \text{ }\mu\text{L}$ of water would traverse the 8.89-cm-long rock core. Evaporation might easily have prevented detection of this volume of solution.

The curve in Fig. 5 for the data of the first core was plotted, using the surface concentration ($1.4 \times 10^3 \text{ d/min}\cdot\text{g}$) and the concentration (7.1% of the surface concentration) 0.89 cm from the surface as calculated for a diffusion coefficient of $2 \times 10^{-7} \text{ cm}^2\text{/s}$ and a diffusion time of 31 days. The curve is consistent with the data from the first core. However, if the same diffusion parameters are used in the second core as were used in the first core, a steeper gradient of diffusing water would be expected (after 61 days) than was observed. The data from the second core suggest a diffusion coefficient higher than $2 \times 10^{-7} \text{ cm}^2\text{/s}$ —perhaps higher by a factor of two. This discrepancy may be due to heterogeneities in the rock cores. Alternatively, the discrepancy may be due to some changing nature of diffusion in the cores, possibly brought on by a slow adjustment of the rock cores to the new stress state they experience in the rock core holders.

5. Conclusions Based on the Rock Salt Infiltration Experiments

Tritiated water appears to be incorporated into rock salt cores (1) by recrystallization (including hydration) at the top core surface that is in contact with brine and (2) by permeation due to diffusion and evaporative loss at the core surface that is in contact with air. Water is incorporated by recrystallization at concentrations up to 0.7% by weight but recrystallized water is readily removed by evaporation into air. Water subject to migration by diffusion reaches its maximum concentration, $7.5 \times 10^{-5} \text{ g/g}$ (75 ppm), in the salt that is in contact with aqueous brine solution. Although a discrepancy was noted between the profiles in the first and second cores, the observed behavior of the water is generally consistent with its having a diffusion coefficient around $2.0 \times 10^{-7} \text{ cm}^2\text{/s}$. On the basis of the strong

and sustained gradient in tritiated water concentration that was observed experimentally, there does not appear to be significant permeation of the water through the rock as a result of the imposed hydraulic gradient. This interpretation of results could be tested in additional experiments to study fluid flow through rock salt.

The lack of any permeation of the rock salt due to the hydraulic gradient imposed on the rock cores, along with the very low diffusion coefficient for water in the rock salt, necessitate that groundwater stream experiments involving the flow of brine through rock salt be done with fissured (jointed) units of rock salt.

E. Analytical Development

1. Radiochemical Separation of Neptunium and Plutonium from Brine

The ability to analyze for neptunium and plutonium in brine solutions will be necessary for the completion of the proposed stream simulation experiment. A literature search for suitable radioanalytical techniques and procedures produced what appeared to be an ideal candidate for the analytical method [REGO].

The procedure was set up and run using neptunium and plutonium tracers (^{239}Np and ^{242}Pu) in a solution of McNutt rock salt (consisting of 200 mg salt per milliliter of water). The method was followed as described [REGO], and no problems were observed with the $\text{La}(\text{OH})_3$ precipitation, dissolution of $\text{La}(\text{OH})_3$, reduction of neptunium and plutonium, and subsequent neptunium extraction into thenoyltrifluoroacetone (TTA). Because of the relatively short half life of ^{239}Np (2.35 days), back-extraction of the TTA fraction and electrodeposition were carried out first. The electroplated planchet was then counted on a GE(Li) detector to determine the amount of gamma-emitting ^{239}Np .

The plutonium fraction underwent additional purification by $\text{LaF}_3\text{-PuF}_3$ precipitation. Because of the large amounts of reductant chemicals present in this solution, the $\text{LaF}_3\text{-PuF}_3$ precipitate tended to carry chemicals that made the precipitate insoluble in HCl. The addition of concentrated HNO_3 and heating to dryness prior to precipitation removed I^- and destroyed the hydroxylamine, but the precipitate still would not completely dissolve in the HCl. Repeated addition of HNO_3 and evaporation helped, but total dissolution of the residue in HCl could not be achieved. The cloudy solution was placed on a 9M HCl preconditioned AGIX8 ion-exchange column, rinsed with 9M HCl, and converted to a NO_3^- column with 8M HNO_3 . The plutonium was then eluted with 0.5M HNO_3 , with subsequent electrodeposition.

After several attempts and failures caused by the above problems, a standard was processed through the entire procedure. The neptunium planchet was counted on a GE(Li) detector, and the ^{239}Np recovery was calculated to be about 25%. The plutonium planchet was analyzed on a surface barrier silicon detector for the amount of ^{249}Pu , and about 10% yield was calculated.

The alpha spectra of plutonium and neptunium planchets showed little or no cross-contamination, i.e., no ^{242}Pu was present on the neptunium planchet and vice versa.

The yield results were far from ideal, but the important separation of neptunium from plutonium was achieved. This separation is necessary because ^{237}Np and ^{242}Pu have essentially the same alpha decay energies (about 4.8 MeV), causing the two isotopes to be indistinguishable if analysis is performed by alpha spectrometry. Even with the poor yields, the method is still sensitive and will be adequate at the concentration levels expected in the experiments. Improvements will be sought in percent yield possibly by reducing the amounts of reductant chemicals in the method without sacrificing the separation of neptunium from plutonium needed for meaningful results.

2. Investigation of Quenching of Scintillation by Salt

Counting tritium directly in solutions of natural salts would be considerably easier than separating the tritium into distilled water before counting. To determine the effect of salt on tritium counting, 20 g of natural rock salt (from McNutt formation) was dissolved in 100 mL of water (a small amount of insoluble material settled to the bottom of the flask). Aliquots of this salt solution were used for a study of the quenching of tritium scintillation by various concentrations of salt. The vials and their contents (listed in Table 3) were prepared by adding 0.01 mL tritiated water standard (making the total activity of each 0.1 mL mixture 1845 d/min) and 15 mL Insta-Gel scintillation medium.

Table 3. Vials Prepared to Study the Quenching of Tritium Scintillation by Salt

Vial	Salt Solution, mL	H ₂ O, mL	Salt in Solution, mg
1	0	1	0
2	0.5	0.5	100
3	0.75	0.25	150
4	1	0	200

Vials 3 and 4 were cloudy after mixing, and vial 4 had a precipitate after standing, but repeated shaking of the vials turned the solution clear. The vials were placed in a liquid scintillation counter (Packard Model 2660), and each was counted several times. The results are listed in Table 4.

Table 4. Counting Efficiencies for Solutions of Various Salt Concentrations

Vial Number	Avg. Eff. Factor, %
1	43.1
2	42.3
3	42.3
4	42.6

With no real difference in the efficiency factors, we concluded that the tritium content can be readily analyzed with rock salt dissolved in the water. The detection limit for a 1 g solution is about 4 d/(min·g); therefore, the detection limit for salt dissolved at 20 mg/mL would be about 20 d/(min·g).

3. Conclusions of Analytical Development

As a result of the developments described above, the radiochemical analyses needed to conduct a groundwater stream experiment are now available. The ability to analyze tritium without the dissolved natural salt having quenching effects considerably lessens the need for laboratory purification steps. The radiochemical separation of neptunium and plutonium from salt solutions is adequate for the experimental requirements, but improved recovery of the radioelements during separation would benefit the analyses.

F. Conclusions from Preliminary Experiments

Based on the result of the preliminary experiments performed in this program, water flow cannot be established through packed clay of the type that is considered for an engineered barrier in a waste repository. This suggests that groundwater stream experiments should be performed by percolating water through loose clay or flowing water over a bed of clay. Similarly, water flow could not be established in cores of fissured but confined rock salt.

From experiments, flow by diffusion in rock salt was seen to be too low to be of importance in stream simulation experiments. Flow will have to be through fissures of rock salt that are held open by spacers. Practical analytical methods are available for the repeated analyses needed for the stream experiments.

Groundwater stream experiments are feasible, provided they are designed by considering the physical properties of the repository materials. With the constraints imposed by the materials, groundwater stream experiments appear to be a practical method of establishing the chemical interactions that would occur in a breached repository in bedded salt.

SUPPLEMENT. TEST PLAN. DEVELOPMENT OF DYNAMIC STREAM SIMULATION METHODOLOGY

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ABSTRACT

This test plan describes a proposed experiment developed by personnel at Argonne National Laboratory and Sandia National Laboratories as part of a joint program to develop a methodology for studying radiochemical transport processes. The experiment performed by this procedure is designed to evaluate the feasibility and practicality of using a complex laboratory experiment to explore the chemical interactions of repository components in the event of a hydraulic breach of a nuclear waste repository mined in bedded salt. The results of this experiment will show whether existing empirical data collected in simple batch tests can be applied to a dynamic model of a repository failure or whether yet more complex experimental methodologies of the type developed here are required.

I. INTRODUCTION

This test plan describes a laboratory experiment designed to evaluate the feasibility and practicality of using an elaborate system to produce chemical interactions that would occur from a hydraulic breach of a nuclear waste repository located deep in a bedded salt environment.

Simplistic transport models have been used to depict (1) the movement of groundwaters contaminated with radiochemicals dissolved from buried nuclear wastes and (2) the sorption of these radiochemicals in an aquifer. Of necessity, these models assume that chemical processes occurring near the repository do not affect the concentrations of radiochemicals released to the aquifer, that the various radiochemicals released to the aquifer do not interact with one another or compete for available sorption sites, that the brine carrying the radiochemicals through soluble evaporites is not significantly altered by dilution with groundwaters or reaction with rocks in the aquifer, and that the carrier brine, in turn, does not significantly alter the rocks or the hydrology in the aquifer. In general, there are insufficient experimental data to validate these assumptions or to provide a technically sound alternative to their use.

Previous experiments have generally addressed one or another aspect of the radiochemical migration process with the assumption that the aspect selected is the dominant influence in migration. It is the hope of many experimenters that interactions between different components that affect migration (canister, engineered barrier, fluid composition) can be modeled

by computer. The laboratory experiment described in this test plan was designed to consider most of the potential complications to migration by integrating all of the components of a breached waste repository in one fluid stream. This test plan describes the experiment in detail, outlines the proposed operation and sampling schedules, and defines the actions needed to implement the experiment. The experiment was designed using the details of a scenario describing a hypothetical hydrologic breach of a nuclear waste repository, as well as the experimental realities revealed in the preliminary experiments (described in the first section of this report).

II. OBJECTIVE

A credible experimental methodology needs to be developed for studying complex geohydrologic/radiochemical reactions in a mode that is parallel to the scenarios used in evaluating the geologic isolation of nuclear waste. The experiment described in this plan was developed to provide a method for reproducing interactions expected because of flow through a repository and into an aquifer. However, the purpose of this experiment is not to provide data for models used in consequence assessments, but to provide experience in conducting complex, dynamic stream-simulation experiments, and in transferring the conceptual details of a hypothetical failure into a credible experimental framework for technical evaluation.

III. DESCRIPTION OF THE EXPERIMENT

This experiment will simulate the flow of a brine solution through a nuclear waste repository mined in bedded salt. Ideally, site-specific details of a proposed repository and its geologic setting would be experimentally modeled. However, the intent of this experiment is better served by identifying a self-consistent set of details and postulates that describe a repository, a waste-form system, an environment near the repository, and an environment far from the repository. The experimental objective is then to simulate a hydraulic breach that conducts a solution through the repository, leaches radiochemicals from the waste, and carries these radiochemicals through the near- and far-geologic environments defined for study. Based on the ability to understand or interpret observations and measurements made in this experiment, the feasibility of using a stream-simulation methodology to assess the long-term performance of a specific repository in bedded salt can be evaluated.

A. Scenario

Figure S-1 depicts the hydraulic breach of the hypothetical repository to be modeled by this experiment. The repository contains high-level nuclear waste entrained in a borosilicate glass, canistered in stainless steel, and placed in a hole backfilled with bentonite and sand. The repository is situated in a rock salt formation with thick interbeds of anhydrite and polyhalite lying above the repository horizon. Because of the extremely low permeability of the rock salt (as verified in preliminary experiments described above) and the other evaporite rocks in this formation, porous flow through

Fig. S-1. Hypothetical Repository Failure to be Modeled

these rocks is assumed to be insignificant. In addition, as also seen in the preliminary experiments conducted in this program, water flow through the compacted clay that has been proposed as an engineered barrier in the Waste Isolation Pilot Plant (WIPP) repository cannot be sustained. That is, the only consequential water movement through the repository that credibly can release radiochemicals to an aquifer is through open conduits--e.g., open boreholes or open, connected fractures produced by some catastrophic event.

The model used in this experiment postulates a hydraulic communication--an open borehole--that permits flow from an aquifer deep below the repository into a dolomite aquifer overlying the rock salt formation hosting the repository. It is further postulated that the groundwater flowing from below dissolves the rock salt and is nearly saturated with salt by the time it reaches the repository. This dissolution below the repository causes collapse at the repository horizon, mechanically breaching the backfill and canister and exposing the waste glass to the brine solution. Dissolution and collapse also fracture overlying strata. The brine, which is contaminated with radiochemicals leached from the waste, flows through the fractured evaporite rocks overlying the repository and up into the subsiding dolomite aquifer.

This assumed process may not be realistic because the dissolution of rock salt underlying the repository would require a prolonged flow of fresh water to and from the face of the rock salt, a situation that does not now exist. The process is postulated for the purpose of this experiment because some of its general features were used in an environmental impact statement written for the WIPP.

B. Equipment and Materials

The diagram in Fig. S-2 shows the apparatus to be used for experimentally modeling the hypothetical repository breach described above. The scenario describes a pathway leading from a source of groundwater, through the repository and near-repository environment, and into an aquifer that hypothetically discharges contaminated water into the biosphere. In the experimental model, a brine solution representing the groundwater/rock solution originating below the repository will be pumped through a simulated, breached waste package, through a column of fractured rocks from overlying strata, and then through a section of dolomite representing the flowing aquifer above the repository.

The descriptions in this section and in Appendix B of this plan include equipment presently available at Argonne National Laboratory (ANL) or being designed and manufactured at the request of ANL. Other materials (solutions,

Fig. S-2. Schematic of Apparatus Used in the Stream-Simulation Experiment. "T" refers to points of temperature measurement, "S" to points where solutions are sampled for analyses, "F" to points where flow is measured, "P" to points where pressures are measured, and R, is a gamma activity recorder.

sample vials, etc.) will also be supplied by ANL unless Sandia National Laboratory (SNL) is specified as the supplier for this experiment. Physically, this experiment will be located in the Chemical Engineering Building (Bldg. 205) at ANL.

The brine feed stock will be WIPP Brine A. This solution will be prepared by the standard SNL recipe (Appendix A), deaerated by sparging with pure, water-saturated nitrogen, and filtered to remove insolubles. Samples of this stock solution and any precipitate will be analyzed for bulk chemical composition to determine true liquid-phase composition. For reference, Table S-1 lists the nominal composition of standard Brine A.

Table S-1. Representative Brine and Solution
for WIPP Experimentation

Ion	Brine "A", mg/L ($\pm 3\%$)	Solution "C", mg/L ($\pm 3\%$)
Na^+	42,000	100
K^+	30,000	5
Mg^{2+}	35,000	200
Ca^{2+}	600	600
Fe^{3+}	2	1
Sr^{2+}	5	15
Li^+	20	--
Rb^+	20	1
Cs^+	1	1
Cl^-	190,000	200
SO_4^{2-}	3,500	1,750
$\text{B} (\text{BO}_3^{3-})$	1,200	--
HCO_3^-	700	100
NO_3^-	--	20
Br^-	400	--
I^-	10	--
pH (adjusted)	6.5	7.5
Specific Gravity	1.2	1.0

The deaerated brine will be pumped through the rest of the system by use of a solution-metering pump having ANL-specified wetted parts of Hastelloy-C276 alloy. This pump has been designed so that it is not corroded by brine. The minimum flow rate of the pump is reported to be 0.9 mL/h (by the manufacturer) at pressures to 27 MPa (4000 psi). Alternatively, a proportioning pump (illustrated in Fig. S-3) can be used to pump brine through the system at a flow rate one-tenth the flow of a primary driving fluid. The components in contact with the brine are made of Hastelloy-C276 with seals of Teflon. A Varian fluid-metering pump which is made of stainless steel and has a minimum flow rate of 1.0 mL/h can produce a flow of the primary fluid equal to 0.1 mL/h.

Fig. S-3. Proportioning Pump

The brine solution will be pumped into two reaction vessels contained in an oven. The first vessel is a settling chamber (trap) included in this system to collect precipitates that may form from the brine as it is heated. As in the case of the proportioning pump, all feed lines, fittings, and reactors are made of Hastelloy-C276 unless specified otherwise.

The other vessel (waste reactor) in the oven is the repository surrogate. This "repository" vessel contains a backfill/getter material, coupons of stainless steel, and a borosilicate waste glass doped with actinides. The detailed assembly of the precipitation and repository vessels is illustrated in Fig. S-4. The backfill/getter material in the repository vessel will be a bentonite:sand mixture with a ratio of 30:70 such as that proposed by E. J. Nowak at SNL [NOWAK-1980A, -1980B]. Crimped and/or preheated and/or

Fig. S-4. Details of the Precipitation Vessel and the "Repository" Vessel

irradiated coupons of stainless steel (SS 304L) will be included in this reactor to simulate canister material. (Note that no overpacks or overcoats are considered at this time.) The activated borosilicate glass will be obtained from an ANL waste-form development program. Nominally, this glass contains 4.2 wt % UO_2 and about 0.4 wt % each of NpO_2 and PuO_2 . A detailed analysis of the waste glass is given in Appendix D. The operating temperature of the oven that contains the precipitation and repository vessels will be 100°C .

After passing through the vessels in the first oven, the contaminated brine will flow through an insulated feed line and into a second oven containing rocks representing the strata overlying the repository. Nominally, this oven will be operated at 40°C . The rocks will be contained in a specially designed holder (see Fig. S-5.). This holder will provide a radial confining pressure on the column to prevent fluid from flowing around the outside of the column. The Teflon sleeve surrounding the column is known to deform and flow into cracks and voids on the surface of rock, providing a seal. The small void spaces at the top and bottom of the rock column will provide some volume for the collection of precipitates (formed as recrystallization products in the brine).

Figure S-6 shows a multicomponent rock column representing fractured strata overlying the failed repository. Of particular interest are the anhydrite and polyhalite beds. Both anhydrite and polyhalite strongly sorb

Fig. S-5. Hastelloy Rock Column Holder with Pressurized Teflon Jacket

Fig. S-6.
Multicomponent Column

actinides from brine solutions and are expected to be more sorbing than halite [DOSCH and DUPREE]. In addition, these rocks are far less soluble (in brine mixed with fresh water) than rock salt is. Therefore, their presence near the repository may contribute in a major way to the retention of radionuclides. Cores of anhydrite and polyhalite have been obtained from marker beds in the Salado Formation from a location near the proposed WIPP site [FORTNEY-1981, p. 63]. The sampling activities are described in detail in Appendix E. Fractured column sections of polyhalite and anhydrite will be made from this core for use in this experiment.

The sections of rock salt will also be prepared from bulk samples obtained from a potash mine near the WIPP site. The rock salt sections will be fractured or perforated to permit fluid flow. Although fracturing may be preferred, saw cuts or drill holes may be made to insure sufficient flow of the brine through these sections.

The rock sections will be stacked into the Teflon sleeve and inserted in a column holder. A radial confining pressure will be applied and maintained, using a manual hydraulic pump connected to the jacket of the holder. A confining pressure of 11 to 12 MPa (1600 to 1800 psi) will seal the Teflon around the rock cores. The operating pressure will be decided from the results of early screening studies, using the multicomponent column. A detailed chemical analysis and a physical description of each rock will be made prior to brine contact to establish baseline characteristics of all components. Post-test analyses are discussed below.

The effluent from the multicomponent column will flow into a second rock column holder containing dolomite. Although similar to the holder shown in Fig. S-5, the metal components will be Monel and the inside diameter of the Teflon sleeve will be only 2.20 cm (0.88 in.). The total length of the rock column will be 9.5 cm (3.7 in.). The rock will be obtained from the more permeable zones in the Culebra member--a dolomite aquifer--of the Rustler Formation, which overlies the proposed WIPP repository. For this experiment, the dolomite will be fractured parallel to the axis of the column. The radial confining pressure necessary to prevent flow between the column surface and the Teflon sleeve will be determined. Again, before the dolomite is contacted with brine, bulk chemical and petrographic analyses of the dolomite will be made. The effluent from the dolomite column will be collected for radiochemical analysis and chemical analysis of the bulk solution.

An experimental option is feeding groundwater into the dolomite column. WIPP Solution C (refer to Table S-1) or another Culebra water composition may be used to represent the aquifer's groundwater. Because of the less corrosive nature of this potable water, a standard Varian metering pump and proportioning pump can be used for flow control.

C. Operation/Sample Schedule

Locations of flow meters, thermocouples, and pressure transducers are indicated in Fig. S-2 by F_i , T_i , and P_i , respectively; numbers substituted for the subscript, i , identify the specific locations. Standard metering and strip-chart recording equipment will be calibrated in accordance with

manufacturers' instructions and used to continuously monitor and record the parameters at the points indicated. Routine calibrations will be made, and records will be kept at ANL of all equipment settings and modifications required.

Figure S-2 also shows the locations for sampling the flow streams, indicated by S_1 . In some cases, the sample point will also serve as an injection point for tracer tests; e.g., tritiated water will be injected on different occasions to measure fluid dispersion through the rock columns. In general, sampling valves and lines will be purged before required samples are collected or tracers injected. All methods developed for sampling and all apparatus modifications made during experiments will be documented, and this information will be retained by the project leader at ANL; copies will be sent to SNL for information and retention.

Before the final experimental setup is completed, the dolomite core will be saturated with brine and then infiltrated with tritiated water to measure the volume of core that is subject to fluid flow. This volume will be only a fraction of the total volume subject to fluid flow measured in the preconditioning sequence of the experiment. Independently of the experimental setup, a sample of the brine will be held at 100°C for 40 h (the residence time of the brine in the 100°C oven), then separated from any precipitate; both the brine and precipitate obtained will be analyzed. (A precipitate of magnesium hydroxide and/or magnesium sulfate may be expected.) A similar precipitate would be expected to form during a stream simulation experiment. Accumulation in the precipitation vessel (Fig. S-4) would be allowable.

Table S-2 details the proposed operation/sample schedule for preconditioning the system. This experiment will be started using the brine A solution. Flow through the assembled system--excluding the solid waste--will continue for 31 days to precondition the rock columns. During this time, the flow stream will be sampled for analyses as indicated by the days and sequence of locations in Table S-2; nominal sample volumes are included for reference. (Analyses are discussed in the following section.) During the 31-day period, the brine flow rate will be maintained at 1.0 mL/h by setting the solution-metering pump at its lowest operating level. Flow rates will be measured on the basis of (1) volumes of solution pumped from the brine reservoir and (2) the volumes of solution fractions collected at the exit of the dolomite core; records of volume readings at defined intervals will be maintained. Sample collection periods will be determined by flow rates and required sample volumes. In general, two full days will be required to collect each sample, and a sample-collection period will be followed by a 5-day period of undisturbed flow. Following the day-24 to day-25 sampling period, the system will be operated undisturbed for only four days, whereupon sampling will be repeated.

On day-31 the surrogate waste will be inserted in the flow stream, and flow will be reestablished and maintained undisturbed for three days. The simulated flow stream will be operated with the surrogate waste in place for a proposed period of 60 days. The sample schedule in Table S-3 indicates the locations and volumes to be collected during this loaded-waste period.

Table S-2. Operation/Sampling Schedule for Preconditioning the System

Day	Description of Operation	Sample Location (see Fig. S-2)	Sample Volume, mL
0	Spurge and sample solution(s) in reservoir(s)	S ₁ , (S ₅)	10, (10)
1-2	Final checkout; establish solution flow rate(s)		
3-4	Collect stream samples (48 h)	S ₆ , S ₄	5, ^a 5
5-9	Reestablish and maintain flow		
10-11	Collect stream samples (48 h)	S ₆ , S ₄	5, 5
12-16	Inject tritium slug; reestablish and maintain flow	S ₄ (Inject)	
	Collect tritium discharge samples (4 h after injection)	S ₆	1
17-18	Collect stream samples (48 h)	S ₆ , S ₄	5, 5
24-25	Collect stream samples (48 h)	S ₆ , S ₄	5, 5
26-29	Reestablish and maintain flow		
30-31	Collect stream samples (48 h) Stop flow(s)	S ₆ , S ₄	5, 5

^aSample volumes at location S₆ do not include the volumes of the optional freshwater feeds. Discharge samples are taken daily at point S₆.

The actual duration of the experiment will be decided as the experiment progresses; continuous evaluation of the movement of contaminants through the rock columns will be based on analysis of the liquid phase concentrations of the species to be described (see below). Because this methodology is in a development phase, it is prudent not to restrict the operating period at this time; the actual period of operation may be shorter or longer than the 60 days proposed here.

Table S-3. Operation/Sampling Schedule: Waste in Place

Day	Description of Operation	Sample Location (see Fig. S-2)	Sample Volume, mL
31	Load waste into system, and check out; establish feed(s)		
32-34	Maintain flow; collect discharge	S ₆	7 ^a
35	Collect stream samples (24 h)	S ₃ , S ₄ , S ₆	1, 1, 5
36-38	Maintain flow; collect discharge	S ₆	7
39	Collect stream samples (24 h)	S ₃ , S ₄ , S ₆	1, 1, 4
40-42	Maintain flow; collect discharge	S ₆	7
43	Collect stream samples (24 h)	S ₃ , S ₄ , S ₆	1, 1, 5
44-46	Maintain flow; collect discharge	S ₆	7
47	Collect stream samples (24 h)	S ₃ , S ₄ , S ₆	1, 1, 4
48-63	--REPEAT CYCLE DESCRIBED FOR DAYS 32-47 ABOVE		
71	Collect stream samples (48 h)	S ₃ , S ₄ , S ₆	1, 1, 4
72-74	Inject tritium, maintain flow	S ₄ (Inject)	
	Collect tritium discharge samples (4 to 8 h after injection)	S ₄ , S ₆	1, remainder
75	Collect stream samples (24 h)	S ₃ , S ₄ , S ₆	1, 1, 5
76-91	--REPEAT CYCLE DESCRIBED FOR DAYS 36-43 ABOVE		
92	Stop flow; sample precipitates; dissect rock cores	TRAP	SOLIDS

^aSample volume at S₆. The volumes of the optional fresh groundwater feed are not included. Discharge samples at S₆ are taken daily except where otherwise specified.

D. Analyses

Detailed analyses of mobile liquid phases and stationary solid phases will be performed to improve understanding of the water/rock interactions, leaching reactions, and radiochemical sorption mechanisms, which are expected to be complex. Table S-4 lists standard analyses to be performed (1) on non-radioactive and radioactive solutions and (2) on rock columns before and after the operational period.

Table S-4. Qualitative and Quantitative Analyses

Sample Component	Type of Analysis
Rocks (uncontaminated)	X-ray diffraction SEM ^a Bulk chemical
Metals (uncontaminated)	Bulk chemical SEM
Getters/Backfill (uncontaminated)	Bulk chemical
Solutions (uncontaminated)	See Appendix C 21 major, minor, trace cations and anions, solution properties of total dissolved solids, pH
Rocks/Solids (contaminated)	Micro-autoradiography α , β , γ spectroscopy, as needed
Solutions (contaminated)	See Appendix C
Waste Glass	Bulk chemical SEM

^a SEM, scanning electron microscopy.

In general, SNL will perform pretest analyses on samples of all rocks to be used. ANL will perform (or contract with other laboratories to perform) post-test analyses on rocks, solutions, and solids contaminated with radiochemicals. ANL will also perform analyses to characterize borosilicate waste glass and to characterize the final states of contaminated metals, backfills, extraneous solids, and equipment components, as required in post-test evaluations.

Conclusions as to the practicality and feasibility of using this methodology to reproduce interaction expected in the hydraulic breach of a nuclear waste repository mined in bedded salt will be based on analytical results. Ideally, this method will permit the empirical characterization of corrosion, leaching, and sorption processes in a dynamic, flowing system. The extent to which the analytical data permit credible descriptions of these processes and potential synergistic reactions will be evaluated. In addition, the extent to which the outcome of this experiment can be predicted from data from simple system experiments (e.g., closed-system measurements of corrosion and leach rates, batch K_d 's, etc.) will be evaluated.

E. Responsibilities for Conducting the Experiment

Manpower and technical support for startup, operation, and post-test analyses are listed in Table S-5. Methods for data collection and handling, inspection of operations, designation of technical personnel responsibilities, and handling and storage of equipment and materials must be jointly approved by the project leaders. Project leaders will be responsible for reporting progress to their respective laboratories as required on a weekly, monthly, quarterly, and/or topical basis. The classification of this experiment, according to the SNL waste management three-level quality-assurance program, is the minor (Level III) quality classification. Therefore, the requirements in appropriate SNL WMP, and QAP documents will be met to ensure that the intent of the SNL QA program is satisfied.

Table S-5. Experimental Procurement Status

Item	Technical Support Procured
<u>Manpower</u>	
1.8 man year	No
<u>Materials</u>	
Solutions	Yes
Rock Samples	Yes
Waste Glass	Yes
Canister	Yes
Backfill/Getter	No
<u>Equipment</u>	
Equipment	Yes
Lab Space	No
<u>Operation</u>	
Stream Simulation Experiment	No
Analyses and Evaluation	No

The experiment described in this test plan is an attempt to satisfy the need to experimentally model the complex chemistry of waste-package degradation, waste-form leaching, and radiochemical transport in a manner consistent with hypothetical scenarios relied on to evaluate the long-term performance of a nuclear waste repository. The experiment described in this test plan integrates major systems that would interact with one another during the release and transport of radionuclides. It is not a simple experimental method, and the experiment will not be simple to perform. However, the results will have direct relevance to the evaluation of the safety of a repository.

This experiment will be evaluated on five points:

1. Is it possible to maintain and monitor the flow stream for a prolonged period without extraordinary expenditure of time or money?
2. Can experimental parameters be easily measured, correlated with field data, and/or used in transport models?
3. Can changes in solutions, rocks, and candidate waste-package components be predicted or interpreted by the use of existing data and models developed from simpler experimental methods?
4. Are expected synergisms identifiable and/or characterizable using the results of the analyses made?
5. Are the results of the experiment so critically dependent on the methodology that they cannot be scaled in size or time to the real world?

Future work in this subtask to develop experimental or other modeling techniques may be affected by the evaluation of this methodology. Stream-simulation experiments are obviously closer to reality than simple batch measurements. However, they will be of value only if they can be interpreted in a technically meaningful way and if they provide substantial new information leading to a better understanding of processes whereby radiochemicals might be released from buried nuclear waste and transported.

APPENDIX A. PREPARATION OF SOLUTIONS

This appendix contains the procedures for making standard WIPP brines and solutions. Brine A is made in a 20 L Nalgene carboy (Cat. No. 6422-0010). Solution C is made in a 4 L Nalgene bottle. Each formulation is filtered with a Gelman Pleated Filter Capsule having 0.2- μ m pore size media (Product No. 12106 or 12117) that had been dried at 110°C for 24 h and weighed before use. The weight of the undissolved solid (all retained by the filter) is then determined from the before-and-after weights of the filter capsule after drying at 110°C. The undissolved solids are then sampled for chemical analysis and stored in a Nalgene bottle under a nitrogen atmosphere.

Analytical reagents are used to prepare Brine A and Solution C.

Brine A - Sixteen-Liter Batch

Dissolve the following in water to make 14.4 L of solution (total volume of H₂O + salt)

NaCl	1601.6 g
Na ₂ SO ₄	82.8 g
Na ₂ B ₄ O ₇ ·10H ₂ O	31.20 g
NaHCO ₃	15.36 g
NaBr	8.32 g
KCl	915.20 g
MgCl ₂ ·6H ₂ O	4673.6 g
CaCl ₂	26.6 g

Add 40-mL aliquot from one liter of a solution containing:

CsCl	0.52 g
RbCl	10.87 g
LiCl	50.00 g
SrCl ₂ ·6H ₂ O	6.00 g
KI	5.20 g

Add a 40-mL aliquot from 250 mL of a solution containing:

FeCl ₃ ·6H ₂ O	1.25 g
--------------------------------------	--------

Dilute to 16 L, and sparge with carbon dioxide for 48 h while stirring with a Teflon-coated magnetic bar. The pH should be 6.5 to 6.6 and should need no adjustment. The density is expected to be about 1.20 g/cm³. Sparge with nitrogen gas, then filter to remove undissolved solids. Weigh the undissolved solids after drying, and analyze the undissolved solids. Store the solution under a nitrogen atmosphere.

Solution C - Two-Liter Batch

Dissolve the following in about 1800 mL of H₂O

Na ₂ SO ₄	0.34 g
MgSO ₄	1.98 g
CaSO ₄	3.36 g
CaCl ₂	0.574 g
NaHCO ₃	0.275 g

Add 100 mL from one liter of solution containing

NaNO ₃	0.54 g
KCl	0.19 g
FeCl ₃ ·6H ₂ O	0.10 g
SrCl ₂ ·6H ₂ O	0.903 g
RbCl	0.03 g
CsCl	0.0026 g

Dilute to 2000 mL, and sparge with carbon dioxide. Filter to remove undissolved solids (mainly iron compounds).

APPENDIX B. DESCRIPTION OF APPARATUS

The apparatus depicted in Fig. S-2 is described as follows:

Sparged Brine Reservoir

The brine reservoir consists of a 2 L Pyrex flask fitted with a suction tube (of Teflon tubing) and a glass frit for continuous sparging of the brine with nitrogen. The nitrogen gas is derived from liquid nitrogen and is purified to remove CO₂ and O₂ gases by the use of Ascarite (CaO on asbestos) and a chromous chloride solution. The gas is passed over a solid oxygen indicator to verify the absence of oxygen, then saturated with water prior to sparging of the solution. The apparatus is described in detail in [SEITZ-1979B].

Brine Pump

Laboratory Data Control Constametric II G Pump (acquisition number ANL-700851) giving flow rates of 0.03 to 10.0 mL/min at any pressure from 0 to 34 MPa (5000 psi). All wetted parts are inert plastics or Hastelloy C-276.

Alternatively, any solution-metering pump can be used in conjunction with the proportioning pump shown in Fig. S-3.

Pressure Transducers

Daytronic 502-3000 G (Serial 93.1 and Serial 84.1). Pressure range of 0 to 20.7 MPa (3000 psi). Made of stainless steel. Maximum analog output of 5 V.

Precipitation and Repository Vessels

Hastelloy-C276 body with Hastelloy-C276 gland. 1.32-cm ID by 12.7 cm long. Made by Autoclave Engineers, Inc. (see Fig. S-4).

Temperature Recorders

The Omega Dataplex 10-temperature measurement system (acquisition number ANL-700856) scans ten separate input channels. Uses thermocouple type T (copper vs. constantan; constantan is about 55% Ca, 45% Ni with $\geq 0.1\%$ Fe or Mn).

Ovens

Hotpack Oven Model 212032 (ANL-700910), capacity of 270 L and an ambient-temperature-compensated range up to 325°C. Operates at 208 V (4.4 kW).

Blue M Stabil Therm Model DV-12A. Capacity of 25.8 L and an ambient-temperature-compensated range up to 260°C. Operates at 120 V.

On-Stream Gamma Activity Recorder

Coiled Hastelloy-C276 tubing in a Bicron Model 3M3 NaI (Tl) crystal detector. Analyzer with continuous recording of the output.

Sleeve-Confining Column Holder

Holder for axially loading a 6.83-cm-dia by 15.2-cm-long cylindrical rock core to pressures of 40 MPa (6000 psi). The cylindrical sleeve is made of Teflon, and wetted metal parts are made of Hastelloy-C276 (see Fig. S-5).

Dolomite-Core Holder

Holder for axially loading a 2.20-cm-dia by 9.53-cm-long cylindrical rock core and applying sleeve-confining pressures up to 40 MPa (6000 psi). The cylindrical sleeve is made of Teflon, and wetted parts are made of Monel-400.

Hydraulic Pumps (not shown in Fig. S-2)

Enerpac 11-1000 (ANL-700698). Hand pumps to supply hydraulic pressure to core holders have a range of 0 to 69 MPa (10,000 psi). All wetted parts are made of stainless steel (SS). The pumps are connected to the SS jacket of the core holder with 0.16-cm-OD SS tubing.

Plumbing

Tubing is 0.32-cm-OD Hastelloy-C276 with an ID of 1.35 mm. Fittings are ferruled tube fittings (Speedbite, Swagelock).

Valves are Autoclave Engr. 10V-2071-HC 770-5582, made of Hastelloy-C276, rated to 75 MPa.

Tees are Autoclave Engr. ST-2220-HC 800-5670, made of Hastelloy-C276.

Elbows are Autoclave Engr. SL-2200-HC 800-5670, made of Hastelloy-C276.

Data Acquisition System

Data acquisition will initially be done by the use of strip-chart recorders and punched paper tape.

A digital processing unit and peripheral units have been purchased for data handling and storage on magnetic tape. The system includes Digital Equipment Corporation PDP 11/23 microcomputer with dual 10.4 megabyte disk drives. The system is designed to interconnect with a DEC VAX 11/780 computer system that is to be installed at the Chemical Engineering Division.

APPENDIX C. SOLUTION ANALYSES

Listed in Table S-6 are the major constituents and possible contaminants of WIPP Brine A, the planned methods of analysis, and the expected sensitivities of the methods. The analyses listed in the table, together with the analyses of total dissolved solids and pH of the solutions, constitute the solution analyses.

Table S-6. Expected Constituents of WIPP Brine A, Methods of Analysis, and Expected Method Sensitivity

Element or Compound	Initial (Approx.) Concentration, mg/mL	Method of Analysis ^a	Sensitivity of the Method, mg/L
Na	42	AAS FE	<500 <500
Cl	190	Direct Titration	<2000
SO ₄ ²⁻	3.5	IC	5
B	0.12	ICP	0.005 ± 50% 0.025 ± 10%
CO ₃ ²⁻	0.7	Evolution GC	<7
Br	0.2	IC	5 to 10
K	30	AAS FE	<300 <300
Mg	35	ICP	0.001 ± 50%
Ca	0.6	ICP	0.010 ± 50% 0.050 ± 10%
Cs	0.001	AAS FE or NA	0.050
Rb	0.018	AAS FE	1 1
Li	0.020	AAS FE	1 1
Sr	0.005	ICP	0.001 ± 50% 0.005 ± 10%
Fe	0.003	ICP	0.005 ± 50% 0.025 ± 10%

(contd)

Table S-6. (contd)

Element or Compound	Initial (Approx.) Concentration, mg/mL	Method of Analysis ^a	Sensitivity of the Method, mg/L
I	0.010	IC	
Pb	b	ICP	0.030 ± 50% 0.150 ± 10%
Ni	b	ICP	0.010 ± 50% 0.050 ± 10%
Co	b	ICP	0.005 ± 50% 0.025 ± 10%
Cd	b	ICP	0.002 ± 50% 0.010 ± 10%
Zn	b	ICP	0.001 ± 50% 0.005 ± 10%
U	b	SUA	$3 \times 10^{-5} \pm 50\%$

^aThe abbreviations correspond to the following methods of analysis:

AAS, atomic absorption spectrometry
 FE, flame emission spectrometry
 IC, ion chromatography
 ICP, inductively coupled plasma emission spectrometry
 GC, gas chromatography
 NA, neutron activation analysis
 SUA, scintrex uranium analyzer

^bThese elements were not specifically added to the Brine A solution but may be present as contaminants.

The types of analyses listed in Table S-6 are for nonradioactive solutions. However, most of the analyses listed in the table can also be performed on solutions containing leached radionuclides (except in some instances at high levels of activity). Only ICP analyses are explicitly unsuitable for radioactive solutions. Analyses by neutron activation (NA) may be necessary to obtain the sensitivity required for cesium. If NA analyses are performed, all elements for which the technique is sensitive will be determined. The radioactive solutions will be analyzed by gamma spectroscopy, alpha spectroscopy, and when needed by beta scintillation counting (e.g., for tritium analyses).

An investigation conducted at ANL (see above) showed that for salt concentrations up to 20 mg/mL of solution, the effect on scintillation counting of dissolved natural McNutt salt in solution with tritium was negligible. Therefore, tritium eluted through rock cores can be analyzed directly without the need to separate the water from the salt.

Neptunium and plutonium in solutions containing salt cannot be analyzed. Therefore, a procedure (described above) based on the work of [REGO] was developed to separate these actinide elements from the salt. The procedure, demonstrated using a solution of rock salt from the McNutt formation, will be employed for the analysis of actinide elements in this experiment.

APPENDIX D. COMPOSITION OF THE SOLID WASTE GLASS

The waste form to be used in this experiment is one of several glass beads supplied by D. J. Bradley of Battelle Northwest Laboratory. The glass, NFS (76-68), was formulated to simulate a solid that contains waste from the Nuclear Fuels Services plant in West Valley, New York. The glass contains the 33 metal oxides listed in Table S-7. The glass was activated by neutron irradiation and contains plutonium, neptunium, and uranium. The glass beads each have approximately the shape of a prolate spheroid with a major axis of 0.35 cm and a minor axis of 0.22 cm; each bead has a weight of about 0.4 g, a density of 3.23 g/cm³, and a surface area of about 1.2 cm² (as calculated for a prolate spheroid).

Table S-7. Chemical Composition of the NFS (76-68) Glass
(Analyses performed by Battelle Northwest)

Constituent	Conc, wt %	Constituent	Conc, wt %
SiO ₂	40.0	Pr ₆₀ 11	0.53
Na ₂ O	12.5	²³⁷ NpO ₂	0.46
Fe ₂ O ₃	9.6	P ₂ O ₅	0.46
B ₂ O ₃	9.5	Cr ₂ O ₃	0.40
ZnO	5.0	SrO	0.37
²³⁸ UO ₂	4.2	Sm ₂ O ₃	0.32
TiO ₂	3.0	TeO ₂	0.26
MoO ₃	2.2	Y ₂ O ₃	0.21
CaO	2.0	NiO	0.20
ZrO ₂	1.7	Rh ₂ O ₃	0.17
Nd ₂ O ₃	1.65	Rb ₂ O	0.13
CeO ₂	1.19	Eu ₂ O ₃	0.070
RuO ₂	1.07	Gd ₂ O ₃	0.050
Cs ₂ O	1.03	²³⁹ PuO ₂	0.46
BaO	0.56	CdO	0.33
PdO	0.53	Ag ₂ O	0.31
La ₂ O ₃	0.53		

APPENDIX E. DESCRIPTION OF POLYHALITE AND ANHYDRITE SAMPLING

Core samples have been obtained from marker beds 123 and 124 identified in ERDA drill hole 9 [POWERS, Vol. 1, Figs. 4.3-3A and 4.3-3B]. These core samples were taken from exposures in a Mississippi Chemical Corporation potash mine located about 15 miles north and west of the proposed WIPP site. The sampling is described in detail by [FORTNEY-1981]. Cores of 7.6-cm diameter (3 in.) and 10.1-cm diameter (4 in.) cores were cut from rocks tentatively identified as principally polyhalite or anhydrite. In all cases, the axis of the coring tool approximately paralleled any bedding planes in these units. To avoid dissolving solubles, all cores were cut with air; however, some moisture was carried through the trap on the compressor supplying the air and did contact the cores' surface. A 7.6-cm-dia coring tool with carbide cutters was used until one of the cutters broke loose from the core barrel. A 10.1-cm-dia tool with diamond cutters was then used to obtain the rest of the required core. Sections of each core were numbered as they were extracted; complete cores were wrapped in plastic and boxed in single-walled cardboard containers for transport. The in-mine operation took place from July 29 to August 1, 1980, and the cores were moved to Sandia National Laboratories on August 11, 1980.

At Sandia National Laboratories, the cores were inspected, tagged, and photographed to insure traceability. Because these marker beds were subject to blasting during the conventional development of the mine, the first 50 cm or so from the tunnel wall was highly fractured. In addition, some breaks in the core had to be made to extract it from the borehole. These fractures or breaks define the core sections shown in the photographs of the core (Figs. S-7 through S-10). For simplicity, the cores were labeled using the field identification provided by Richard Snyder (U.S. Geological Survey) as polyhalite (POLY) or anhydrite (ANHY), followed by the core diameter in inches (3 or 4). Unique identification of cores is ensured by the location and date included on each label card. The first section of core extracted from each borehole was numbered "1" and marked with an arrow to indicate the direction of coring; other sections are numbered in the sequence of extraction. After photography, two samples (each weighing 2 to 5 g) from selected

Fig. S-7. Three-Inch-Diameter (7.6-cm-dia) Core POLY 3 Identified by Powder X-ray Diffraction to be Predominantly Polyhalite [nominally $K_2MgCa_2(SO_4)_4 \cdot 2H_2O$] and Halite (NaCl)

Fig. S-8. Four-Inch-Diameter (10.1-cm-dia) Core POLY 4
Identified by X-ray Diffraction to be
Predominantly Polyhalite and Halite

Fig. S-9. Three-Inch-Diameter (7.6-cm-dia) Core ANHY 3
Identified as Predominantly Anhydrite (CaSO_4)
and Halite (NaCl) by Powder X-ray Diffraction

Fig. S-10. Four-Inch-Diameter (10.1-cm-dia) Core ANHY 4
Identified by Powder X-ray Diffraction to be
Predominantly Anhydrite and Halite

sections of each core were shipped and analyzed by powder X-ray diffraction. The analyses are described by [FORTNEY-1980]. The results of the analyses are given in Table S-8.

Table S-8. Listing and X-ray Analyses of Cores Taken from the Mississippi Chemical Corporation Potash Mine for Use in Radionuclide Transport Experiments

Core Label	Section	Mineral Content ^a
POLY 3	1	--
POLY 3	2	--
POLY 3	3	--
POLY 3	4	Polyhalite, halite
POLY 3	5	Polyhalite (major) with halite
POLY 3	6	--
POLY 4	1	--
POLY 4	2	--
POLY 4	3	--
POLY 4	4	--
POLY 4	5	Halite with polyhalite
POLY 4	6	Polyhalite (major) with halite
POLY 4	7	--
ANHY 3	1	--
ANHY 3	2	--
ANHY 3	3	--
ANHY 3	4	Anhydrite, halite
ANHY 3	5	--
ANHY 3	6	Halite (major) with anhydrite

^a Chips of the rock cores were analyzed by the Debye-Scherrer technique, using copper X-radiation. Estimated abundance (major) is qualitative. Polyhalite refers to the mineral of nominal composition, $K_2Ca_2Mg(SO_4)_4 \cdot 2H_2O$; halite refers to the mineral of composition NaCl; anhydrite refers to the mineral of composition $CaSO_4$. Analyst: D. M. Heinze, Sandia National Laboratories.

The core sections were then rewrapped in plastic and boxed for storage or distribution. The results of these analyses, full-color photographs of the core sections, and a physical description of each core are being permanently retained in WIPP Central Files (SWCF) in the nuclide migration category to provide traceability for samples used in experiments.

The joint ANL/SNL experiments require specially prepared rock cores. Because of the extremely low permeability of evaporite rocks in the Salado formation, it is assumed that there is no significant porous flow through these rocks in scenarios describing hypothetical repository breaches. That is, the only credible, consequential water movement through the Salado formation that can release radiochemicals to an aquifer is assumed to be through an open conduit--e.g., open boreholes, failed borehole plugs, or open, connected fractures. Therefore, fracture flow through evaporites from the Salado will be studied. Cylindrical rock columns will be made from the larger core samples; these columns will be fractured by the Brazil technique [JAEGER] and placed in a pressurized rock core holder that will permit simulated fracture flow.

Sections of the polyhalite and anhydrite cores described above have been selected for these experiments. All polyhalite sections were transferred to Argonne National Laboratory.

Some of the cores have been used to demonstrate the feasibility of machining and fracturing these rocks. Sections numbered 1 from both the 7.6-cm-dia (3-in.-dia) polyhalite and anhydrite cores were machined and fractured, using standard machining techniques and equipment, by H. Smulin of Sandia Laboratories.

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