

RADIONUCLIDE MIGRATION EXPERIMENTS IN TUFF BLOCKS UNDER UNSATURATED AND SATURATED CONDITIONS AT A SCALE OF UP TO 1 METRE

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ABSTRACT

To complement migration experiments with non-radioactive tracers in the Busted Butte experimental facility (BBTF) at the Nevada Test Site, an exploratory migration experiment has been performed under unsaturated conditions in a $\sim 0.3\text{m} \times \sim 0.3\text{m} \times \sim 0.3\text{m}$ block of tuff. Longer term migration experiments, up to 600 days, under unsaturated and saturated conditions in $\sim 1\text{ m}^3$ blocks of tuff have recently been completed. Na-fluorescein, ^3H (as tritiated water), ^{22}Na , ^{60}Co , $^{95\text{m}}\text{Tc}$ and/or ^{99}Tc (as the pertechnetate anion), ^{137}Cs , and ^{237}Np were used as tracers in all three experiments. Under unsaturated conditions, Tc is transported slightly faster than ^3H , while under saturated conditions, the chemical conditions became highly reducing, leading to significant retardation of Tc along the flow field. If chemically reducing conditions can be demonstrated to exist in the saturated zone downstream from the proposed repository, the geological formations underlying the proposed repository horizon can potentially act as a geological barrier to the transport of some multivalent radionuclides.

INTRODUCTION

The host rock selected for the proposed US spent fuel repository is the Topopah Springs tuff formation in Yucca Mountain. This formation is underlain by the non-welded Calico Hills formation (1). Transport of any radionuclides leached from the emplaced wastes is expected to be vertically downward from the Topopah Springs formation through the unsaturated zone in the Calico Hills and, below the water table, horizontally in the saturated zone to a discharge area. An understanding of the transport behavior of radionuclides through the geological formations underlying the repository horizon under unsaturated and saturated zones is essential to assess the environmental impact of the disposal of high-level nuclear wastes. Although field migration experiments have been performed under unsaturated conditions in non-welded tuff at the Busted Butte Test Facility (BBTF) at the Nevada Test Site, only chemical analogs of radionuclides could be used in these experiments (1). The transport behavior of radionuclides under these conditions must therefore be inferred from results obtained in these field experiments and a comparison between the sorptive behavior of chemical analogs and radionuclides and from laboratory studies performed on relatively small scales in various national laboratories. The availability of suitable laboratory facilities and supporting infrastructure at the Atomic Energy of Canada's Whiteshell Laboratories in Pinawa, Manitoba presented a unique opportunity to study the migration of selected radionuclides in tuff from the geological formations of interest to the US DOE nuclear waste disposal program.

EXPERIMENTAL

Excavation

The tuff blocks were excavated from Vitric Zone 1 of the Topopah Spring Tuff formation (Ttpv1) and from the Calico Hills formation (Tac) in the BBTF in 1999. Busted Butte itself is a small (2.5 km x 1 km), north-trending mountain block primarily made up of thick ignimbrite deposits of the Paintbrush Group and is located to the southeast of Yucca Mountain, on the western part of the Nevada Test Site (1). The tuff blocks were excavated from the rear wall of the main adit of the BBTF and transported to the Whiteshell Laboratories.

The excavation technique involved the removal of extraneous tuff to form a pillar with a square cross section of ~30 x 30 cm for the trial block and ~120 x ~150 cm for the two larger blocks. To stabilize the friable geological material, the tuff was encased in epoxy prior to its removal from the formations. The pillars were wrapped with layers of commercially available plastic shrink-wrap, similar to SaranWrap™, which was then gently heated with a hot air gun. Four-sided plywood enclosures were built around the pillars to serve as a form for the epoxy and leaving a gap between the plywood and the tuff. The bottom of the plywood enclosure was sealed to the shrink-wrap using a silicone sealant, RTV-108, and allowed to cure. One-litre batches of a low heat generation epoxy, Hysol™ were added to the gap between the plywood and the tuff and allowed to cure.

The excavation of the small, trial block proceeded as follows: a horizontal cut was made through the tuff pillar, below the plywood frame, and a steel plate, wrapped in polyethylene sheathing, was inserted into the saw cut. This plate was bolted to flanges on the plywood frame. A second horizontal cut was made directly above the plywood to sever the connection between the tuff block and the remainder of the pillar, allowing the blocks to be removed from the pillar. A cover was sealed and screwed to the top flange of the plywood frame to prevent loss of moisture during storage and transit. The method used to excavate the large blocks was similar. The blocks were then transported by truck to the Whiteshell Laboratories.

Transport Solution

Synthetic Busted Butte pore water was used as the transport solution in all migration experiments. The target chemical composition (1) is shown in Table I.

Table I. Chemical Composition of Synthetic Busted Butted Pore Water

| Constituent | Conc. (mg/L) |
|------------------|--------------|
| Ca | 26 |
| Mg | 3.8 |
| Na | 21 |
| K | 3.2 |
| Si | 338 |
| Cl | 18 |
| NO ₃ | 233 |
| F | 1.7 |
| HCO ₃ | 51 |

Migration Experiments

The experimental arrangement used in the migration experiments under unsaturated conditions is shown in Figure 1. The steel plate underneath the two tuff blocks was replaced with a porous polyethylene sheet with 35 μm pores. A collection system was installed underneath this membrane with 9 collection ports (for the trial block) and 36 ports (for the large block). An acrylic plenum was attached to the top of the plywood enclosures and a dynamic partial vacuum applied to the collection system to impose a slight pressure differential across the polyethylene membrane to maintain a vertical unsaturated flow through the blocks.

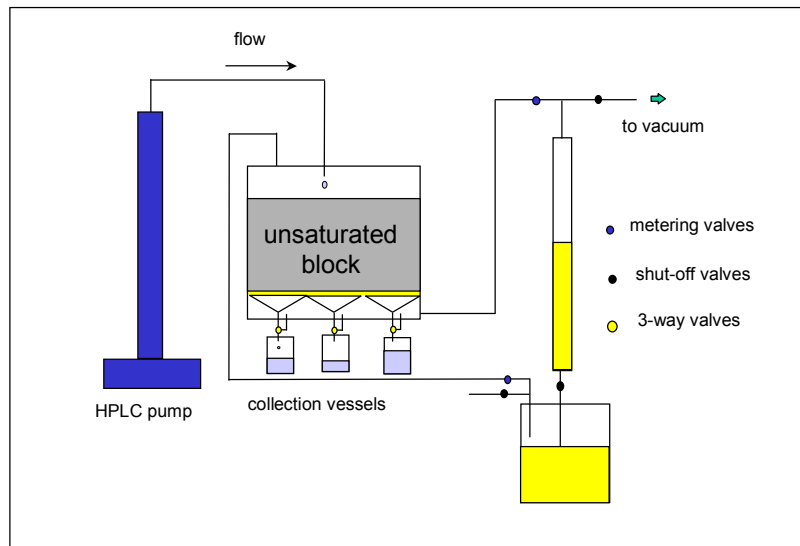


Fig. 1. Schematic used in migration experiment under unsaturated conditions.

For the experiment under saturated conditions, the second large block was encased in a stainless steel shroud to avoid potential leaks. Two 38-mm diameter boreholes were drilled vertically in opposite corners of the block to a depth of 50 cm to serve as injection and withdrawal boreholes and to give a flow path of ~ 100 cm. Five 20-mm diameter vertical boreholes were drilled between the injection and withdrawal boreholes to a depth of 75 cm and equipped with Teflon™ inserts, each with five sampling ports at

10 cm intervals. Four additional vertical boreholes with a diameter of 2.5 cm were drilled in each of the four corners of the block to flood the block slowly with synthetic Busted Butte pore water. After the block had been saturated, an acrylic cover was fitted to the top of the block. The schematic for this experiment is shown in Figure 2.

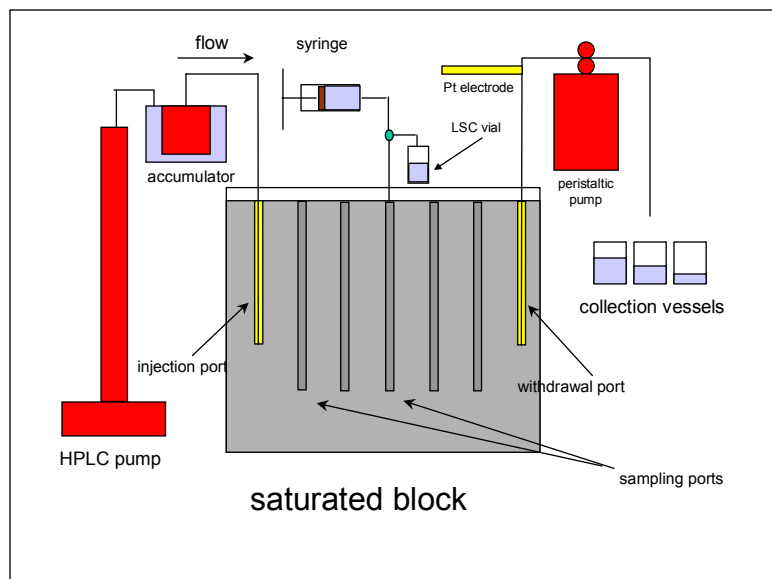


Fig. 2. Schematic used in migration experiment under saturated conditions.

To establish a stable, unsaturated flow through the trial block, synthetic pore water was added to a central location at the top of the block and collected from the nine collection points at the bottom of the block. After a stable vertical flow of 20 mL/hr had been established through the trial block under unsaturated conditions, an 800 mL volume of pore water containing $^3\text{H}_2\text{O}$, ^{22}Na , ^{60}Co , $^{95\text{m}}\text{Tc}$, ^{99}Tc , ^{137}Cs and ^{237}Np was added to the top of the block over a 40-hour period. This tracer addition was followed by adding tracer-free synthetic pore water for a period of 86 days until elution profiles had been obtained for the fluorescein anion, ^3H , $^{95\text{m}}\text{Tc}$ and ^{237}Np .

Addition of the synthetic Busted Butte pore water at a rate of 10 mL/h at each of two locations on the upper surface of the large block was started in October 2000 and the first indication of water exiting the block at the bottom was observed in March 2001. By April 2001, the flow through the block had stabilized sufficiently to start the addition of the tracer solutions. The same suite of radionuclides as in the trial block was used with the exception that $^{95\text{m}}\text{Tc}$ was not included, as the planned duration of this migration experiment was long compared to the 61-day half life of this isotope. In contrast to the migration experiment in the trial block, the radionuclide tracer injection was performed as a step function with continuous injection of the tracer solution. The migration experiment was terminated in November 2002. The duration of the migration phase of this experiment was 589 days. Water samples were collected from the 36 sampling ports for the duration of the experiment and analyzed spectrophotometrically for Na-fluorescein and by liquid scintillation counting for ^3H and ^{99}Tc . Analysis of selected samples by gamma spectrometry did not detect any gamma-emitting radionuclides in the eluent.

For the experiment under saturated conditions, a flow of 10 mL/h of synthetic Busted Butte pore water was established in March 2001 between the injection and withdrawal boreholes of the block. The injection of the suite of radionuclide tracers, identical to that used in the large unsaturated block, was also started in March 2001 and continued until November 2002, for a duration of 602 days. A Pt electrode was installed in the line leading from the withdrawal port to monitor the redox conditions in the water

exiting the block. Samples of the eluted water were analyzed spectrophotometrically for Na-fluorescein, by liquid scintillation counting for ^3H and ^{99}Tc , and by gamma spectrometry for the other radionuclides. Small volumes of water were also extracted along the flow field in the block through the sampling ports in the five vertical boreholes located between the injection and withdrawal boreholes.

Post Migration Experiment Analysis

At the termination of the migration experiment in the trial block, successive layers of tuff were removed from the top of the block to a depth of 5 cm, to obtain information on the spatial distribution of the radionuclides retained by the block near the injection point. Thirteen 2.5-cm diameter holes were drilled vertically into the block to obtain additional samples. The samples were weighed and submitted for radiometric analysis and then dried to constant weight to obtain a measure of the degree of saturation of the rock matrix.

Supporting Sorption Experiments

Static batch sorption experiments were performed in support of the radionuclide migration experiments using the method by Weaver (2). These experiments were carried out in triplicate for each of two types of geological material, Topopah Spring tuff, crystal-poor, vitric zone 1, and Calico Hills Formation tuff, and from two synthetic groundwaters, synthetic Busted Butte pore water and synthetic J-13 groundwater. Two liquid volume to solid mass ratios (V/m) (nominally 4:1 and 20:1) and five individual radioactive tracers (^{22}Na , ^{60}Co , $^{95\text{m}}\text{Tc}$, ^{99}Tc , ^{137}Cs and ^{237}Np) as well as Na-fluorescein were used. The tracers were used individually and in mixtures to determine the effect on sorption by competing ions. In all cases, a nominal 20 mL volume of the appropriate tracer solution was added to the conditioned solid material. The amount of conditioning solution retained with the solid resulted in slightly higher V/m ratios than the nominal values cited above. In all cases, triplicate blanks (centrifuge tubes containing the tracer solution but no geological material) were used to determine the extent of tracer interaction with the reaction vessel.

Samples of material from the two geological formations were disaggregated by hand. Portions of the material were weighed into tared 30-mL polycarbonate centrifuge tubes, which were used as the reaction vessels. The geological materials were conditioned for a period of 7 d with synthetic pore water or groundwater prior to initiating the sorption step. The conditioning solution was removed following centrifugation at 14000 rpm for 2 hours using a Beckman Model J2-HC centrifuge with a Beckman JA-17 fixed angle rotor. The amount of conditioning solution retained with the geological material was determined by weight. The appropriate tracer solution was added and the sorption reaction was allowed to proceed for 21 d. Following the sorption period, the separation of the solid and liquid phases was ensured by three successive centrifugations, as described above, prior to removing aliquots of the aqueous phase for analysis. The radionuclide tracer concentrations were determined by gamma spectroscopy and the Na-fluorescein concentrations using an HP 8542A Diode Array Spectrophotometer.

Supporting Microbial Investigations

To determine the presence and viability of microbes, samples of the tuff block, used in the migration experiment under saturated conditions, were obtained using a sterile drill. These, and samples of unconsolidated tuff from the Calico Hills and Topopah Springs formations and samples of synthetic Busted Butte pore water were analyzed for the presence of microbes using a number of microbial procedures. (3)

RESULTS AND DISCUSSION

Migration Experiments

The elution profiles for Na-fluorescein, $^3\text{H}_2\text{O}$, $^{95\text{m}}\text{Tc}$, and ^{237}Np in water collected from the trial block are shown in Figure 3. These results have been averaged for all nine sampling ports. The transport of the fluorescein and the pertechnetate anions was $\sim 17\%$ faster than that of tritiated water. This accelerated transport may be explained by anion exclusion by which the negatively charged mineral surfaces cause a slight repulsion of the anionic tracers. The ^{237}Np was retarded by a factor of 3.2. These results agree quantitatively with those predicted from results obtained in static sorption experiments at the Whiteshell Laboratories using crushed tuff from the same geological formation.

Radiochemical analysis of the samples obtained in the post-migration experiment analysis showed that ^{60}Co was most strongly retained near the injection point, followed by ^{137}Cs . The front of the ^{22}Na plume had migrated approximately 20 cm through the block. These results are consistent with measured sorption coefficients obtained under static conditions obtained at the Whiteshell Laboratories.

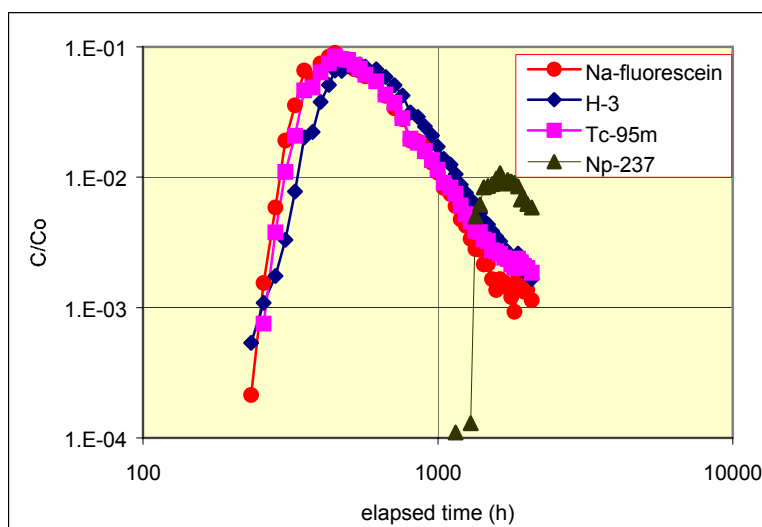


Fig. 3. Elution profiles for Na-fluorescein, TcO_4^- , NpO_2^+ , and $^3\text{H}_2\text{O}$ from all sample ports plotted as a log-log plot.

The distribution of the water collected in the 36 sampling port in the large block used in the migration experiment under unsaturated conditions fluctuated more than in the trial block. This may simply be due to unstable flow at this scale. A representative elution profile from one of the sampling ports is shown in Figure 4 and shows that the elution profiles of the Na-fluorescein, $^3\text{H}_2\text{O}$ and ^{99}Tc are very similar and that no evidence of accelerated transport of the anionic species is observed. The average residence time of the non-sorbing tracer in the block is estimated to be ~ 600 d. This corresponds to a linear flow velocity of the transport solution of ~ 1.7 mm/d. As expected from the results obtained in the trial block and the measured sorption coefficients, no evidence of the other tracers has been observed in the eluent from this second block.

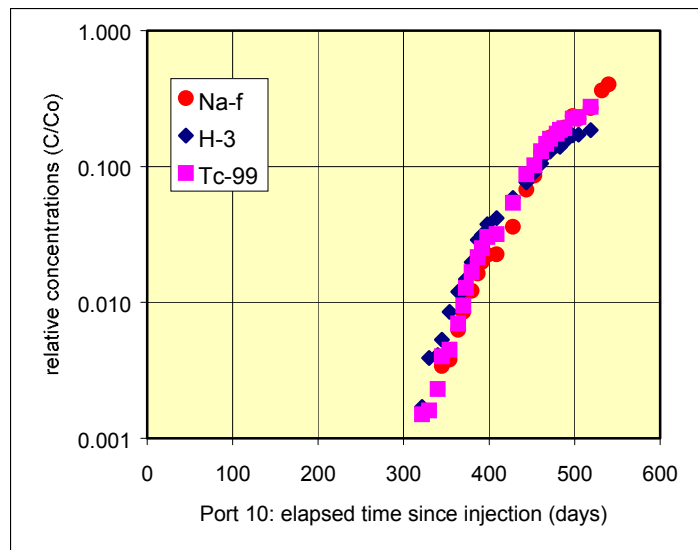


Fig. 4. Elution profiles for Na-fluorescein, $^3\text{H}_2\text{O}$ and ^{99}Tc for water collected from sampling port 10.

The results obtained from the migration experiment performed under saturated conditions show a much different behavior of the ^{99}Tc . Although some ^{99}Tc was detected in the water collected from the withdrawal borehole in the block, its concentration decreased after a few months and fell below the detection limit after ~12 months. Samples obtained along the flow field also show this decrease with time and distance from the injection point and indicate that the ^{99}Tc is removed from solution in the block.

One plausible explanation for this behavior is that the injected Tc(VII) is reduced to a sparingly soluble lower oxidation species with, possibly, a higher affinity for the geological material (4,5). This is consistent with very low measured Eh values (-500 mV, uncorrected) obtained with the in-line Pt electrode. The cause of the low observed redox conditions is not yet clear but could be microbially induced and may be accelerated by the presence of the fluorescein anion that was used as the dye tracer in these experiments..

These results suggest that any Tc released from the waste repository in Yucca Mountain may initially migrate vertically downward through the unsaturated zone with little or no retardation as long as chemically oxidizing conditions prevail and Tc is present as Tc(VII). However, if chemically reducing conditions exist in the saturated zone, the bulk of the Tc is expected to be reduced to a lower oxidation state and precipitate or sorb onto the geological material. The Tc concentration in the groundwater will then be solubility controlled, resulting in a very low flux of this radioelement.

First arrival of ^{237}Np at sampling points 25 cm from the injection borehole was detected ~400 days after tracer injection was initiated. This is slower than would be expected from its sorption coefficient and may also be the results of chemically reducing conditions in the block.

Sorption Experiments

The sorption values, R_d , were calculated from the results of the batch sorption experiment using the equation

$$R_d = \frac{V}{m} \times \frac{C_0 - C_f}{C_f}, \quad (\text{Eq. 1})$$

where V is the volume of contacting solution
 m is the mass of geological material
 C_0 is the initial tracer concentration, and
 C_f is the final tracer concentration following the reaction period.

A summary of the sorption values obtained is given in Table II.

Table II. Summary of Static Batch Sorption Results

| Tracer | R_d (mL/g) (single tracer) | R_d (mL/g) (mixture of tracers) |
|----------------|---------------------------------|--------------------------------------|
| Na-fluorescein | -0.13 ± 0.26 | 0.45 ± 0.27 |
| Tc | -0.06 ± 0.20 | -0.06 ± 0.18 |
| Np | 0.73 ± 0.34 | 3.66 ± 6.17 |
| Na | 6.35 ± 0.33 | 2.44 ± 0.17 |
| Cs | 1360 ± 600 | 250 ± 0.14 |
| Co | 2050 ± 600 | 440 ± 60 |

Supporting Microbial Investigations

The enumeration results for all samples, i.e., aerobic and anaerobic heterotrophs, obtained on two media, R2A and R2A + nitrate are presented in Table III and show that there was evidence of microbial presence in the saturated block. The heterotroph concentrations are expressed as Colony Forming Units (CFU) per unit mass or volume. Table III also contains the data obtained for nitrate-reducing bacteria (NRB) and sulfate-reducing bacteria (SRB); these are expressed in terms of Most Probable Number (MPN) per unit mass or volume. The presence of nitrate is not expected to make a difference to the number of viable aerobes that can be cultured under aerobic conditions. The R2A medium is not N-limited (for assimilatory processes, i.e., the building of cells) and dissimilatory nitrate reduction (for energy production) occurs only under anaerobic conditions. Therefore, aerobic enumeration on R2A with and without nitrate added should give identical results. This is indeed the case for all samples enumerated in Table III, except for one sample from the saturated block, where the results from the two media vary by less than a factor of two. This close agreement between the results obtained from the two media for all aerobically enumerated samples is a good indication of the accuracy of the methods used and provides an extra degree of confidence in the results.

For anaerobically analyzed samples, it is expected that, if NRB are present (i.e., viable and culturable), R2A + nitrate medium would give higher results than R2A. Table III shows that in most cases (i.e., all the rock samples), anaerobic results on R2A + nitrate are in fact lower than on R2A, suggesting that none of these rock samples contained large amounts of viable and culturable NRB. The fact that SRB are not present either and that generally anaerobic heterotrophic enumeration results are low compared to aerobic results suggests that these rock samples came from very aerobic environments with either no NRB

population or, if NRB were present, they were not adapted to anaerobic conditions and nitrate reduction for energy.

Nitrate-reducing bacteria were also enumerated in a more specific manner. The results suggest that NRB were present only in liquid samples, i.e. 1.1×10^2 MPN/mL inflow and $>10^4$ to 5×10^6 MNP/mL outflow. Anaerobic enumerations on R2A and R2A + nitrate have not been counted yet for the outflow samples, so a comparison cannot be made except for the inflow sample where anaerobic heterotrophs on R2A with nitrate yielded somewhat higher results than in most rock samples. Therefore, although the rock samples did not contain significant amounts of viable and culturable NRB, the inflow sample did contain them, and the outflow samples contained large populations of NRB. This suggests that an anaerobic environment was created in the saturated block that induced the proliferation of NRB. Their presence would explain the reduction in nitrate levels in the outflow compared to the inflow water nitrate concentrations.

Table III. Microbial Analysis Results

| <u>Block</u> | <u>Sample and Sample Treatment</u> | <u>Aerobic Heterotrophs</u> CFU/g dry wt or mL water R = R2A medium RN = R2A & nitrate | <u>Anaerobic Heterotrophs</u> CFU/g dry wt or mL water R = R2A medium RN = R2A & nitrate | <u>NRB</u> MPN/g dry wt or mL water | <u>SRB</u> MPN/g dry wt or mL water |
|-------------------|---|---|---|---|--|
| Saturated Block | Synthetic pore water for Saturated Block | R $(2.23 \pm .14) \times 10^5$ RN $(2.29 \pm .2) \times 10^5$ | R $(2.86 \pm .23) \times 10^2$ RN $(7.13 \pm .4) \times 10^1$ | 110 UL = 470 LL = 76 | < 0.41 |
| Saturated Block | Ground tuff from inlet port | R $(5.29 \pm .64) \times 10^4$ RN $(5.48 \pm .84) \times 10^4$ | R $(3.86 \pm 6.7) \times 10^0$ RN $(3.86 \pm 6.7) \times 10^0$ | <3.48 | < 4.7 |
| Saturated Block | Ground tuff from outlet port | R $(1.19 \pm .03) \times 10^6$ RN $(1.16 \pm .02) \times 10^6$ | R $(1.29 \pm .4) \times 10^2$ RN $(3.58 \pm 6.2) \times 10^0$ | < 3.22 | < 4.4 |
| Saturated Block | Ground tuff from sample port | R $(1.95 \pm .04) \times 10^6$ RN $(2.04 \pm .1) \times 10^6$ | R $(4.85 \pm 2.4) \times 10^1$ RN $(4.57 \pm .45) \times 10^2$ | < 3.12 | < 4.2 |
| Saturated Block | Water eluted from saturated block; sample E-0-5 | NA | NA | 4.6×10^6 UL= 2.1×10^7 LL= 1.0×10^6 | > 11 |
| Saturated Block | Water eluted from saturated block; sample E-0-9 | R $(3.67 \pm .19) \times 10^6$ RN $(1.99 \pm .25) \times 10^6$ | R $>3.00 \times 10^4$ RN $>3.00 \times 10^4$ | $> 1.1 \times 10^4$ | 0.41 UL = 2.9 LL = .056 |
| Saturated Block | Water eluted from saturated block sample E-0-11 | R $(6.03 \pm .02) \times 10^6$ RN $(4.37 \pm .05) \times 10^6$ | R $>3.00 \times 10^4$ RN $>3.00 \times 10^4$ | $> 1.1 \times 10^5$ | < 0.41 |
| Unsaturated Block | Ground tuff from top sampling port | R $(3.27 \pm 1.09) \times 10^2$ RN $(5.09 \pm 1.09) \times 10^2$ | R <1.00 RN <1.00 | < 3.27 | < 4.4 |
| Unsaturated Block | Ground tuff from middle sampling port | R $(4.98 \pm .54) \times 10^3$ RN $(4.33 \pm 2.17) \times 10^3$ | R $(3.98 \pm 3.25) \times 10^1$ RN $(3.58 \pm 10.8) \times 10^0$ | < 3.25 | < 4.4 |
| Unsaturated Block | Ground tuff from bottom sampling port | R $(1.51 \pm .25) \times 10^5$ RN $(1.54 \pm .12) \times 10^5$ | R < 1.0 RN $(7.68 \pm 11.5) \times 10^0$ | < 3.4 | < 4.7 |

CFU = Colony Forming Units
MPN = Most Probably Numbers

UL = Upper Limit
LL = Lower Limit

NRB = nitrate reducing bacteria
SRB = sulfate reducing bacteria

The NRB are often facultative organisms, which use aerobic respiration with O₂ as electron acceptor in the presence of air (even if nitrate is present) but use nitrate as electron acceptor in the absence of O₂. Nitrate-reducing bacteria reduce nitrate, in dissimilatory metabolism using a number of enzymes, via nitrite, nitric oxide and nitrous oxide, to elemental nitrogen. Some can reduce nitrate to NH₄⁺ in dissimilative metabolism. Many NRB will also reduce other electron acceptors anaerobically such as ferric iron, and certain organic electron acceptors, and many can grow by fermentation. Therefore, NRB are metabolically quite diverse in terms of alternative energy-generating mechanisms but nitrate reduction yields less energy than aerobic respiration (6).

CONCLUSION

The results from these large-scale migration experiments have shown that, under unsaturated conditions, transport of Tc is not retarded by the geological formations underlying the proposed repository horizon and that the transport behavior of Np is consistent with that predicted from static batch sorption coefficients. Under saturated conditions, however, a marked decrease in the measured redox conditions in water collected from the block and a concomitant loss of Tc from solution were observed. The transport of ²³⁷Np is also slower than predicted from its sorption coefficient and is thought also be due to the chemically reducing conditions in the block. The causes of these chemically reducing conditions are being investigated and may be facilitated by microbial activity, although the effect of the presence of Na-fluorescein cannot be ruled out at this stage.

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