Ground Water Monitoring&Remediation

Collection of Unsaturated-Zone Water Samples for Tracer Analysis: Evaluation of an Inverting Membrane Technique

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Abstract

As part of the site characterization efforts at Yucca Mountain, Nevada, a series of unsaturated zone tracer tests has been performed at nearby Busted Butte. The phase 2 tracer test was conducted within an instrumented 10 m by 10 m by 7 m in situ block of vitric tuff. A complex tracer solution containing both reactive and nonreactive tracers was pumped into the block during a period of 27 months. Throughout the test, thousands of unsaturated zone pore water samples were collected on sorptive pads attached to inverting membranes and then analyzed for tracer concentration. Partway through the experiment, three new boreholes were drilled into the block, and two intercepted the tracer plume. The rock core was removed for pore water extraction and analysis, and the boreholes were then instrumented with inverting membranes and sorptive pads. The initial set of pore water-soaked pads was removed from the boreholes a week after they were emplaced, and the pore water was extracted and analyzed. This paper compares the tracer concentration data from the rock cores and the pads to evaluate the effectiveness of the inverting membrane collection technique for a variety of tracers. While the sorptive pads sample only dissolved tracers, rock cores contain both dissolved and sorbed tracer. For nonreactive tracers, such as halides (bromide and iodide) and fluorinated benzoic acids (FBAs), this distinction is immaterial, and the rock and pad data agree quantitatively for the halide tracers, and qualitatively for the FBAs. For reactive tracers, such as lithium, the dissolved tracer concentrations can be estimated from rock analyses by dividing by the tracer's retardation factor; when this correction is applied, the rock and pad lithium data are also in reasonable agreement.

Introduction

The Busted Butte unsaturated zone transport test (UZTT) was a field-scale tracer test undertaken to validate numerical and conceptual models of flow and transport at Yucca Mountain, Nevada, the proposed site for a U.S. high-level nuclear waste repository. Busted Butte is located ~6 km southeast of the repository site. The vitric tuffs of the Topopah Springs and Calico Hills units that underlie the proposed repository crop out at Busted Butte, thus providing a good site for examining the field-scale behavior of these units and testing the ability of project numerical models to accurately predict field-scale transport behavior (Bussod et al. 1998).

The Busted Butte UZTT consists of an underground facility in which a test block, 7 m by 10 m by 10 m, was instrumented with a set of eight horizontal and parallel 10 cm diameter injection boreholes and, perpendicular to those, a set of 12 parallel 10 cm diameter collection boreholes (Figure 1). From July 1998 until October 2000, a mixture of reactive and nonreactive tracers (Table 1) was continuously pumped into the injection boreholes at rates low enough to ensure that the block remained unsaturated. The tracers traveled through the rock and were collected, up to 3 m below, at the collection boreholes. The resulting tracer breakthrough curves were compared to modeling predictions based on laboratory-derived hydraulic and transport parameters (Tseng et al. 2003). This comparison highlights both the successes and the shortcomings of our current conceptual and numerical models for unsaturated zone transport (Tseng et al. 2003).

Key to the success of the field test was the ability to collect repeated samples of pore water under unsaturated conditions in order to obtain a time-series of tracer breakthrough. We needed to be able to obtain samples that were representative of the whole system including the entire range of pore sizes as well as preferential pathways such as fractures and faults, while creating as little disturbance to the system as possible. Traditional methods of obtaining unsaturated zone pore fluid samples were not appropriate for various reasons. Coring of the rock and extraction of pore fluids gives good samples as this approach catches fluids from all the pores and fractures and can be carried out under unsaturated conditions. Unfortunately, coring the block is a form of destructive testing that cannot be repeated indefinitely to produce a time-series breakthrough curve. Drip collectors can be sampled

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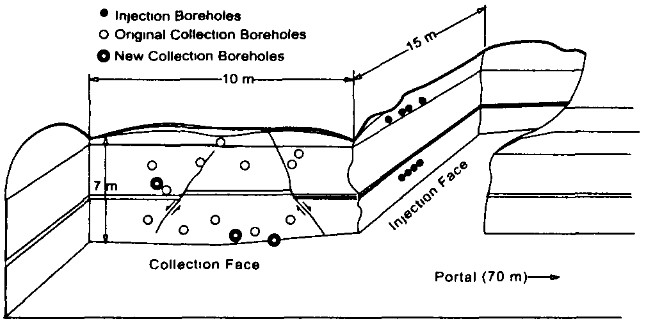

Figure 1. Schematic of the Busted Butte test block, showing injection and collection faces and borehole locations.

over time; however, they require that ponding of fluid has occurred and, therefore, do not represent transport under unsaturated conditions. Sampling techniques requiring the use of suction into a porous cup allow collection of repeatable samples under unsaturated conditions, but tend to be biased toward collection from the larger pores and fractures (Boll et al. 1992).

Repeatable and reproducible collection of unsaturated zone pore water samples was accomplished at Busted Butte by instrumenting the collection boreholes with pneumatically emplaced inverting membranes with absorbent paper pads attached along their length. Every one to two weeks, the pads were changed and the pore water-soaked pads were shipped to the Los Alamos National Laboratory (LANL) for analysis.

In March 2000, after 19 months of tracer injection, three new boreholes were drilled into the collection face of the test block (Figure 2). Borehole 49 was cored between March 1 and 8, and borehole 50 was cored between March 9 and 14; both intercepted the tracer plume in the nonwelded Calico Hills Tuff in the lower half of the test block. Borehole 51 was cored between March 15 and 22 into the lowermost unit of the Topopah Springs Tuff, but did not intercept a tracer plume and will not be discussed further. The Calico Hills Tuff consists of alternating beds of poorly cemented, salmon pink massive tuff and variably cemented white ash beds. Core collected during drilling was returned to LANL for tracer analysis, and the boreholes were then instrumented with inverting membrane collection systems. Pore water samples were collected from boreholes 49 and 50 on March 22 for halide and fluorinated benzoic acid (FBA) analysis, and on March 29 for lithium analysis. This sequence of events provided two independent measurements of in situ tracer profiles along the boreholes, one using rock core samples, the other using pore water extracted from pads collected one to four weeks later. The purpose of this paper is to compare these two datasets, in order to evaluate the effectiveness of the inverting membrane collection technique. Significant differences between the rock and the pad profiles will be attributed to tracer migration during the one to four week lag time, to conceptual differences between rock and pore water concentrations for sorbing tracers, or to sampling and analytical uncertainty and errors.

Methods

Tracer Mixture

The tracer solution injected at Busted Butte was a complex mixture of reactive and nonreactive components (Table 1) (Turin et al. 2002). Bromide and iodide are nonsorbing halide tracers and were used to track breakthrough; bromide was injected throughout the tracer test (July 1998–October 2000), while iodide injection began in August 1999 after a hydraulic steady state was approached. Sodium fluorescein (henceforth, fluorescein), rhodamine water tracer (WT), and carbomoyl-2(1H)-pyridone (pyridone) are all fluorescent tracers. Fluorescein was quite visible on the pads under UV light and enabled real-time field determination of tracer breakthrough. Rhodamine WT-stained rock was useful in identifying the injectors during posttest excavations. Pyridone is an experimental tracer that has been used elsewhere at Yucca Mountain. Lithium is a mildly reactive tracer that provided insight into the rates and pathways of metal transport. The other injected metals and rare-earth elements (cerium, samarium, nickel, cobalt, manganese) are all highly reactive and sorbing. Each injection borehole was labeled with one of five different FBAs, which made it possible to identify tracer pathways and mixing. Turin et al. (2002) provide more details on the properties of the tracer mixture. To minimize the reactivity of the tracer solution with the country rock, the tracers were injected in a matrix of synthetic pore water (Table 2).

Field Techniques—Tracer Injection

Two pneumatically emplaced borehole sealing and measurement systems for tracer injection and collection were developed by Science and Engineering Associates (Santa Fe, New Mexico). The injection system consisted of transparent packers made of 0.15 mm polyethylene surrounding a polycarbonate pipe that provided structural support, installed in 10 cm diameter injection boreholes, ~10 m in length. Injection ports, consisting of an injection pad and moisture sensor, were installed at 75 cm intervals along the outside of the packers. The packers were inflated to a pressure of 1.7 to 3.5

Table 1
Composition of Tracer Solution Injected During the Phase 2 Experiment at Busted Butte

Tracer Component	Concentration (mg/kg)
Bromide	920.08
Iodide (starting August 18, 1999)	382.2
Fluorescein	10
Rhodamine WT	10
Pyridone	10
FBA	100
Lithium	79.92
Nickel	2.47
Manganese	2.78
Cobolt	2.48
Samarium	2.06
Cerium	1.88

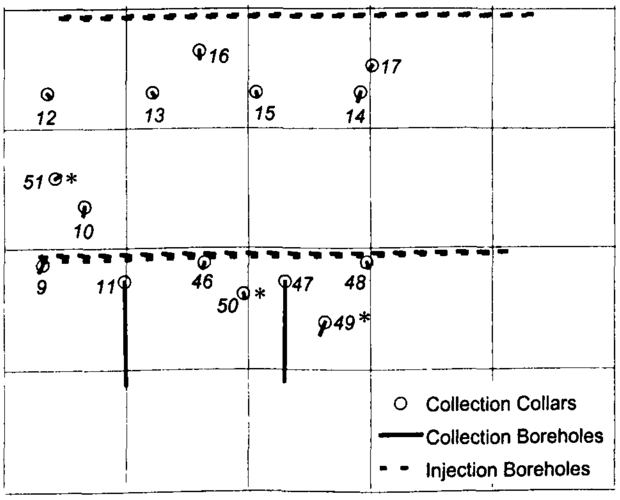

Figure 2. Diagram of the collection face showing the collection boreholes. Boreholes marked with an asterisk are the three new boreholes (49, 50, and 51). The horizontal lines represent the injection boreholes coming in from the injection face. The grid shown is 2 m by 2 m squares.

kPa to hold the injection pads and moisture sensors against the walls of the boreholes and to prevent air from circulating within the hole and causing drying.

Individual syringe pumps pumped tracer solution from large (up to 500 L) tanks to each injection point. Running alongside the tracer tubes were wires that connected a simple electrical-resistivity moisture sensor at each injection point to a moisture-sensor terminal block at the rock face. The tubes and wires ran down the borehole between the inside of the borehole and the outside of the inflated membrane. At each injection port, the tracer solution was delivered to a polypropylene absorptive pad that confined flow to a well-defined injection point and that held a moisture sensor in place. These simple sensors, consisting of two parallel bare wires ~1 cm apart fixed between two 2 cm squares of fusible fabric, were automatically monitored by the field datalogger system to provide an uncalibrated semiquantitative measurement of moisture changes along the packer.

Field Techniques—Pore Water Collection Pads

The inverting membranes for tracer collection were welded tubes of double-coated polyester fabric ~10 m long and 10 cm in diameter. A mesh pocket ran the length of each membrane and held the sampling pads and moisture sensors (Figure 3). This approach allowed the collection pads to be placed at any desired spacing along the boreholes. The sampling pads used in the phase 2 experiment consisted of bundles of three strips of Whatman #42 filter paper (Whatman Inc., Clifton, New Jersey) ~3 cm wide and 20 cm long. Like the injection membranes, the collection membranes were inflated to a pressure of 1.7 to 3.5 kPa to ensure good contact between the collection pads and the borehole walls.

The membranes were removed from the collection boreholes every one to two weeks, and the pore water-soaked pads were replaced with new dry pads. The pore water-soaked pads were packaged individually in 60 mL amber polyethylene bottles, and returned to LANL for analysis. The hydraulic behavior of the initially dry pads, and their subsequent sorption of pore water, was the subject of a detailed modeling investigation by Tseng and Bussod (2001).

Laboratory Analysis of Collection Pads

In the laboratory, tracers were extracted from the pads in their original field collection bottle by adding 40 mL of an extractant solution and mixing on a rotary mixer for four hours. For nonreactive tracers, the extractant was a bicarbonate/carbonate (BCC) buffer solution (192 mg/L $NaHCO_3$ and 3.20 mg/L Na_2CO_3; pH = 8.2 to 8.5); for metals and rare-earth elements, a 2% nitric acid solution was used. Controls consisting of clean pads spiked with tracer solution for halide and FBA extractions, and a metals solution for metals analysis, were extracted alongside the experimental samples. After mixing, the extract was decanted, filtered through a 0.2 µm filter, and then analyzed for tracer concentration by inductively coupled plasma mass spectroscopy, ion chromatography, and high-performance liquid chromatography for metals, halides, and FBAs, respectively.

Pad loadings (µg tracer/pad) were calculated using the measured extract concentration and a dilution factor accounting for the amount of extractant added. Analysis of the control pads showed that recovery of halides and FBAs from pads was close to 100% with an overall reproducibility of ± 12% to 15%. Extraction of metals yielded recoveries close to 100%, except for nickel, which showed anomalously high recoveries. Reproducibility for the metals analyses was ± 10% for manganese, nickel, cobalt, cerium, and samarium, and ± 20% for lithium.

Collection and Analysis of Rock Cores

Rock cores were drilled using a 10 cm diameter carbide bit, packaged, refrigerated, and sent to LANL for analysis. Each core was divided into ~15 cm sections along its length and crushed; alternate sections were processed for metals, and for halides and FBAs. An extractant solution (BCC buffer as previously noted for halides and FBAs, 5% nitric acid for metals) was added to the crushed rock (rock extractant ratio = 1:1 by weight for BCC and 1: ~0.7 by weight for acid), and the samples were then shaken for four hours. The extract was then decanted and filtered through a 0.2 µm filter. Spiked control samples were extracted alongside the experimental samples. Extracts were then analyzed for tracer concentrations as previously described.

Table 2
Synthetic Pore Water Composition

Chemical Formula	Concentration (mg/kg)
$CaCl_2 \cdot 2H_2O$	36.8
$Ca(NO_3)_2 \cdot 4H_2O$	44.8
NaF	3.8
Na_2SO_4	10.7
$NaHCO_3$	51.2
$KHCO_3$	9.0
$MgSO_4 \cdot 7H_2O$	36.9
$Ca(OH)_2$	7.8

Note: Synthetic pore water was prepared in deionized ground water that contained ~30 mg/kg dissolved SiO_2.

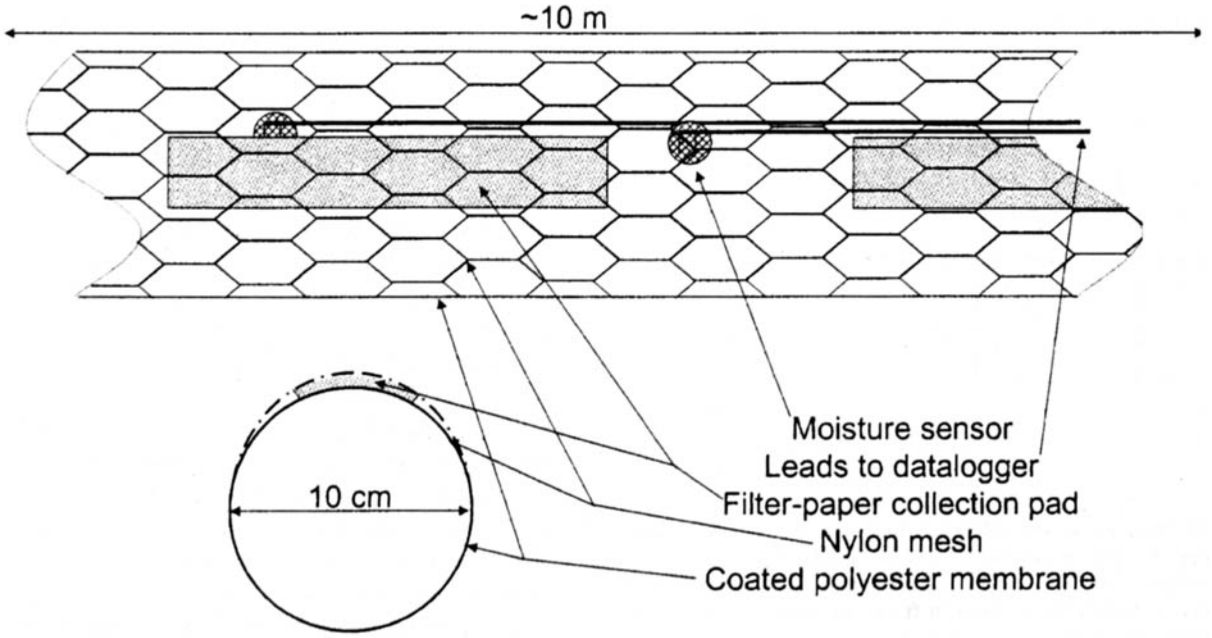

Figure 3. Inverting membrane with mesh pocket holding sorptive pads and moisture sensors. Membrane diameter is 10 cm; collection pads are bundles of filter paper strips ~3 cm wide and 20 cm long.

Analytical recovery for the six tracer metals from rock samples were 102% ± 24% for cerium, 91% ± 17% for cobalt, 98% ± 11% for lithium, 84% ± 6% for manganese, 125% ± 19% for nickel, and 90% ± 20% for samarium. Previous to this study, we carried out a pilot project that consisted of spiking preinjection core ($n = 15$) with tracer and extracting the rocks with the BCC buffer solution previously described. From this study, we obtained recovery rates of 98% ± 4% for bromide, 101% ± 9% for iodide, and 99% ± 3% for FBA.

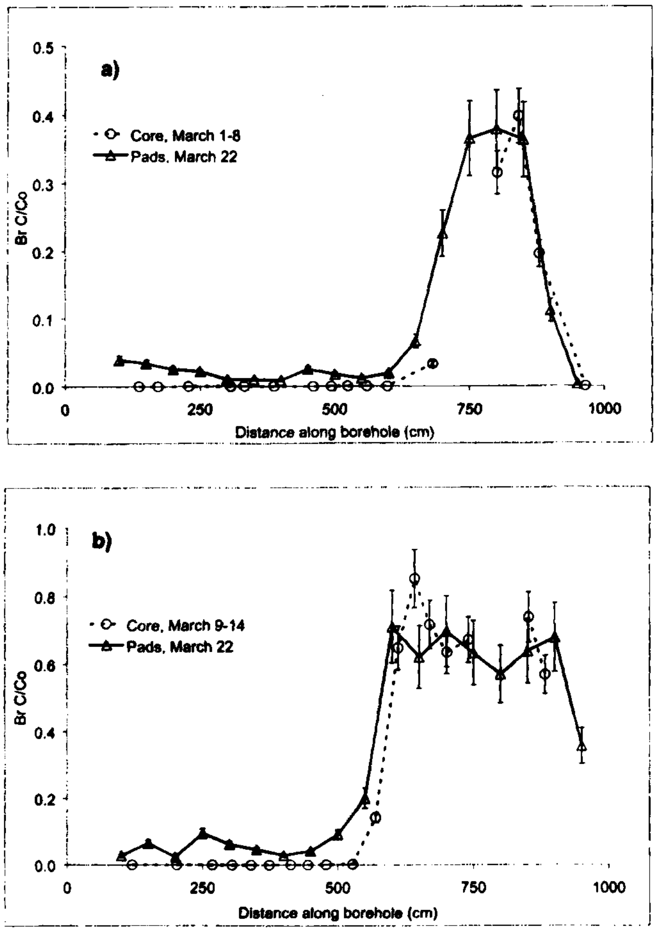

Figure 4. Recovery of bromide from core and pads in (a) borehole 49 and (b) borehole 50. Error bars are based on overall reproducibility of control sample analyses.

Moisture Analysis and Concentration Calculations

Total moisture content of pads and rocks was determined by oven-drying the samples and subtracting the dried weight from the initial weight, measured in the field immediately after collection. Tracer concentrations were calculated by dividing the mass of a given tracer extracted from a sample by the total moisture content of that sample and are, therefore, expressed in terms of $\text{mass}_{\text{tracer}}/\text{mass}_{\text{pore water}}$.

Results

In this section, we present tracer recovery data obtained from the rock cores along with the tracer concentrations obtained from the collection pads for the two boreholes that intercepted the tracer plume (49 and 50). All the charts presented are of the same format; recovery of the analyte of interest (C/C_0 = pad or rock concentration/initial injected concentration) plotted against horizontal distance along the collection borehole.

Bromide

Bromide profiles along boreholes 49 and 50 are shown in Figure 4. Borehole 49 intercepted the front of the plume ($C/Co_{\max} \approx 0.4$), while borehole 50 was within the main body of the plume ($C/Co_{\max} \approx 0.8$). The profiles show very good agreement between concentrations in rock core collected in the first half of March and collection pads retrieved on March 22. There is some evidence that borehole 49 pad concentrations exceed core concentrations, possibly reflecting increasing concentrations during the intervening two weeks.

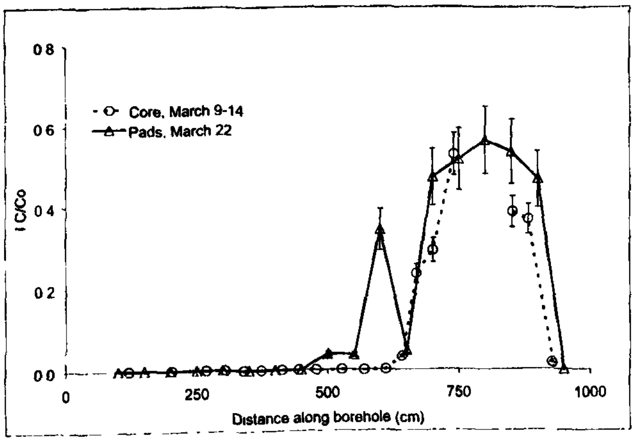

Figure 5. Recovery of iodide from core and pads in borehole 50. Iodide was not detected in either core or pads from borehole 49. Error bars are based on overall reproducibility of control sample analyses.

The slight horizontal offset between the two borehole 50 profiles may be due to errors in the precise core or pad locations.

Iodide

Iodide injection lagged bromide injection by more than a year and, at the time of drilling, the iodide plume had not reached the location of borehole 49. Figure 5 shows core and pad profiles for borehole 50, which intercepted the front of the plume ($C/Co_{max} \approx 0.5$). A major iodide plume, at a location of 650 to 900 cm, is clearly evident in both the core and pad data, with some evidence of increased concentrations in the pads collected on March 22. A second, smaller iodide peak at 600 cm appears only in the pad data, indicating breakthrough along this flowpath sometime between early March and March 22.

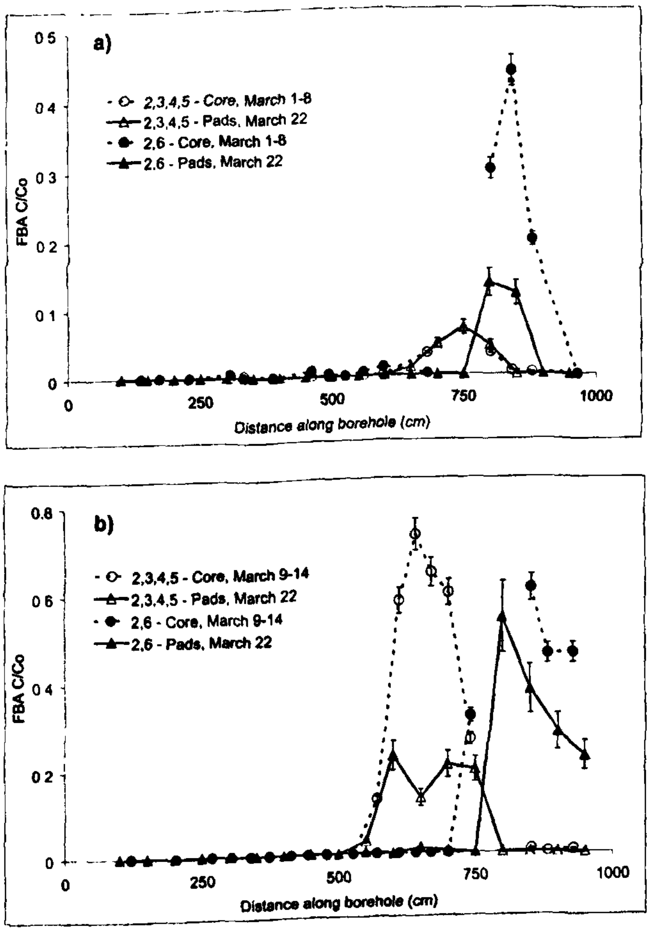

Figure 6. Recovery of FBA from core and pads in (a) borehole 49 and (b) borehole 50. Error bars are based on overall reproducibility of control sample analyses.

Fluorinated Benzoic Acids

Boreholes 49 and 50 both intercepted plumes of two different FBAs: 2,6-difluorobenzoic acid (2,6 in Figure 6) and 2,3,4,5-tetrafluorobenzoic acid (2,3,4,5 in Figure 6). In both cases, the 2,3,4,5 plume is closer to the borehole collar, and partially overlaps the 2,6 plume. Figure 6 shows that both rock and pad sampling methods agree qualitatively, clearly identifying the presence and location of the FBA plumes, but differ in plume concentrations. In all cases of disagreement, core concentrations exceed pad concentrations—the opposite of what would be expected if the discrepancies were due to plume spreading during the interval between coring and pad collection. The consistency of the discrepancy points to some systematic bias, requiring further investigation.

Lithium

There is a natural background level of lithium in the rocks at Busted Butte, which is revealed in both the core and pad concentration profiles shown in Figure 7. The erratic and low concentration profiles in Figure 7a show that borehole 49 did not intercept the tracer lithium plume, while a clear plume is revealed by borehole 50 pad data at the 800 cm location in Figure 7b.

The background concentrations measured in rock cores greatly exceed the background pad data. This is a result of a basic conceptual difference between the rock extraction and collection pad methods for determination of concentrations of a sorbing tracer, such as lithium. While the collection pads sample only dissolved lithium, the nitric acid method extracts both dissolved and sorbed lithium. Thus, analysis of the collection pads yields a pore water concentration (mass of dissolved tracer/mass of pore water), whereas rock extraction yields the total concentration of tracer (total mass of tracer/mass of pore water). The ratio of total mass of tracer to mass of dissolved tracer in a given volume of rock is the retardation factor, a function of the tracer sorption and rock moisture properties. For linear sorption:

$$\frac{M_T}{M_D} = R_F = 1 + \frac{K_D}{w} \qquad (1)$$

where M_T is total mass of tracer in a unit volume, M_D is dissolved mass of tracer in a unit volume, R_F is the retardation factor, K_D is the linear sorption distribution coefficient expressed in mass units (g/g), and w is the gravimetric moisture content (g pore water/g rock).

For nonsorbing tracers, such as the halides and FBAs, the retardation factor is 1, and pad and rock concentrations are the same. For sorbing tracers, such as lithium, the retardation factor is > 1, and rock (total) concentrations exceed pad (dissolved) concentrations, as shown in Figure 7. However, by

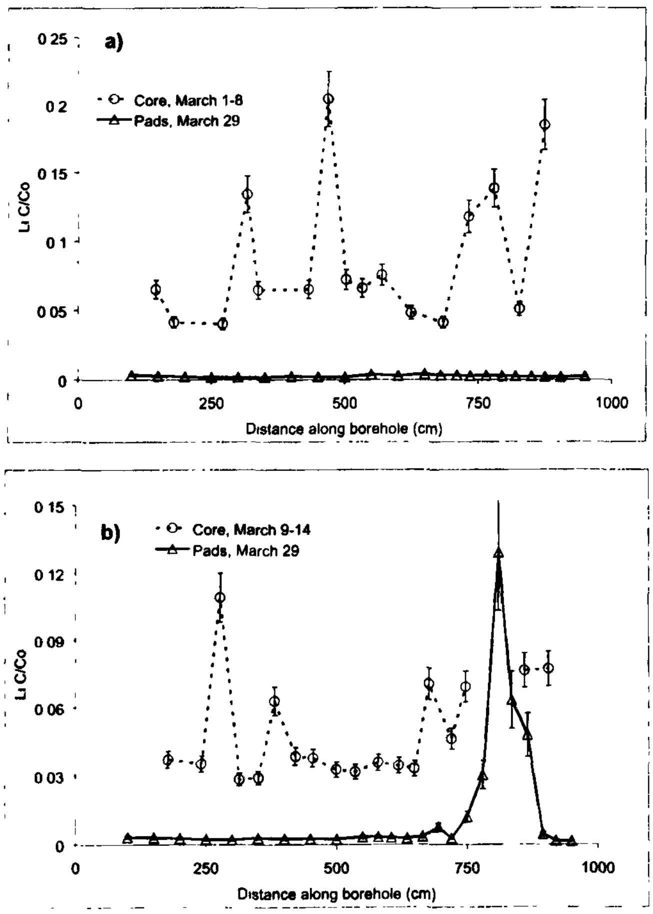

Figure 7. Recovery of lithium from core and pads in (a) borehole 49 and (b) borehole 50. Error bars are based on overall reproducibility of control sample analyses.

using Equation 1 and a measured K_D of 1.3 g/g for lithium sorption to Calico Hills Tuff (Turin et al. 2002), we can determine a retardation factor for each core sample and calculate a corresponding lithium pore water concentration. When these calculated pore water concentrations are plotted alongside pad concentrations, the two datasets generally agree well (Figure 8). Spikes in the core data, especially apparent in the borehole 49 profile (Figure 8a), may reflect dissolution of precipitated or rock lithium, rather than extraction of sorbed lithium.

Unfortunately, the obvious lithium peak that appears in borehole 50 pad data (Figures 7b and 8b) cannot be compared to the core results because the critical core sections were not recovered during drilling operations. Poor core recovery may be due to increased moisture content associated with the tracer plume.

Transition and Rare-Earth Metals

The rare-earth tracers were apparently unstable in solution (Kearney et al. 2000) and will not be considered further. The transition-metal tracers all sorb much more strongly than lithium, with linearized K_D values ranging from 98 to 1600 g/g (Turin et al. 2002). Because of their high K_D values, we expect the total rock concentrations to be much higher than pad concentrations. Our results (not shown) show this to be the case, but for all three transition metals, the difference between rock and pad concentrations is not as great as predicted by Equation 1. Manganese results are particularly anomalous, with many concentration values in excess of the injected tracer concentration. Further investigation showed that the transition-metal concentrations encountered in both rock and pad samples are similar to those in areas of the test block completely unaffected by our tracer injection and must therefore represent natural background levels of metals in the tuff. In other words, the transition-metal tracer plume had not yet reached the site of the new boreholes. The high concentrations measured and the lack of agreement with the Equation 1 sorption relationship strongly suggest that our nitric acid extraction procedure is actually dissolving rock material (either in the cores or detritus attached to the pads), rather than simply extracting dissolved and sorbed metals. Because of the high background levels encountered and the fact that the boreholes did not intercept the transition-metal plume, we are unable to test the effectiveness of the pads in collecting samples for reactive metal analysis.

Conclusions

In summary, we can draw several conclusions regarding the effectiveness of the inverting membranes as a technique for collecting pore water samples from the unsaturated zone. Inverting membrane sorption pads accurately and reliably collect pore water samples for analysis of nonsorbing tracers. Bromide and iodide rock and pad data agreed quite closely; FBA pad and rock data agreed qualitatively.

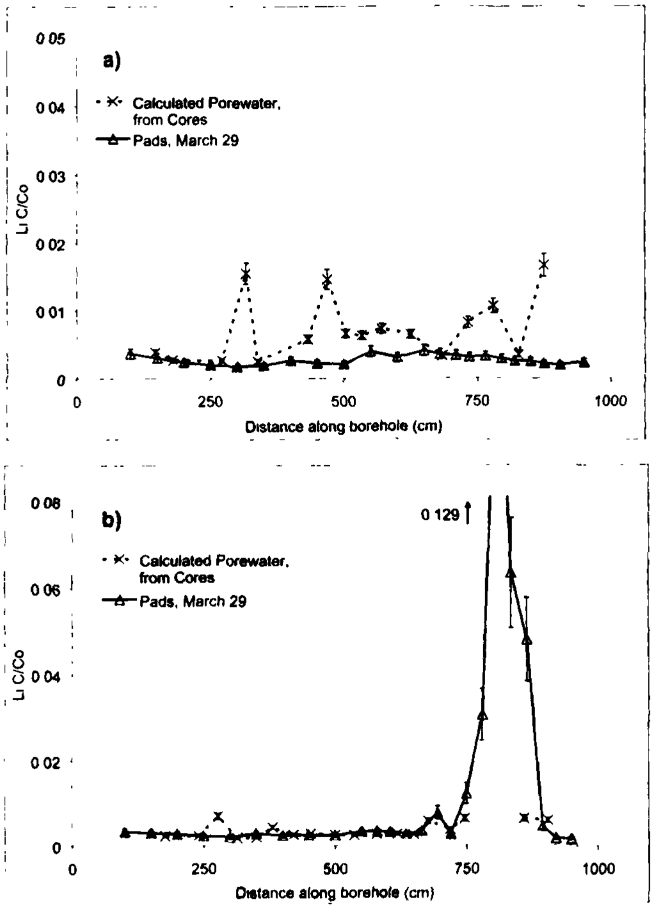

Figure 8. Comparison between lithium concentration in collection pads and calculated pore water concentration based on rock analysis results and Equation 1 for (a) borehole 49 and (b) borehole 50. Error bars are based on overall reproducibility of control sample analyses.

Because the pads collect only dissolved tracers, the concentrations measured for sorbing tracers are less than the total concentration measured in the rock core. This effect can be predicted by calculating appropriate retardation factors. Rock and pad-based measurements of lithium background concentrations agreed when sorption was taken into account. The collection pads clearly detected lithium breakthrough in one borehole; poor core recovery prevents direct comparison of rock and pad data of the plume itself. Our boreholes failed to intercept a transition-metal plume, so the collection pads' effectiveness at sampling transition-metal tracers cannot yet be determined, but it is apparent that background concentrations of metal tracers need to be carefully evaluated.

Based on our experience at Busted Butte, we can unequivocally recommend the use of an inverting membrane/sorbing collection-pad system for future unsaturated zone tracer tests with nonreactive or weakly sorbing tracers. For strongly sorbing tracers, we believe additional development and evaluation of collection and extraction methods is required.

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