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MIGRATION OF CESIUM-137 THROUGH A SOLID
CORE OF MAGENTA DOLOMITE TAKEN FROM THE
RUSTLER FORMATION IN SOUTHEASTERN NEW MEXICO

A. W. Lynch, R. G. Dosch and C. R. Hills

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Migration of Cesium-137 Through a Solid Core of Magenta
Dolomite Taken from the Rustler Formation in Southeastern
New Mexico

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ABSTRACT

A column-flow cesium migration experiment has been conducted in support of the safety assessment program for the Waste Isolation Pilot Plant (WIPP) in Southeastern New Mexico. The column was a monolithic cylinder of Magenta dolomite obtained from a core sample drilled near the proposed WIPP site. The fluid was a Magenta equilibrated de-ionized water solution that had been doped with 0.001 ppm of ^{137}Cs . The experiment ran continuously for an 18 month period at flow rates comparable to those measured for natural groundwater at the site. After flowing for 18 months, the apparatus was disassembled and the dolomite column examined for cesium distribution. Cross sectional radiographs of the sample showed that most of the cesium had penetrated only 0.4 cm into the solid Magenta column with a well defined wave front. On the other hand, trace quantities (2×10^{-7} ppm) of ^{137}Cs were detected in effluent samples collected during the experiment.

The distribution coefficient (120 ml/gm) calculated (assuming porous flow) from the average depth of cesium penetration (0.3 cm) is considerably less than the value obtained from a batch equilibration measurement made on the same system (650 ml/gm), indicating that cesium will migrate through Magenta rock at a faster rate than batch equilibration value predicts.

Transmission electron microscope analysis of a piece of Magenta dolomite from the same core that had been soaked in 1 molar CsCl solution indicated that the cesium was preferentially sorbed on a montmorillonite clay phase that was inhomogeneously dispersed throughout the sample. These results indicate that if groundwater travels through Magenta rock the dissolved cesium may be retarded by the clay content of the rock.

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Introduction

Sorption of radionuclides by geomedia could provide major time delays in the migration of radionuclides from a radioactive waste repository to the biosphere. The long term safety assessment for the proposed Waste Isolation Pilot Plant (WIPP) required columnflow and batch equilibration sorption coefficient measurements to determine the sorptive properties of geologic media surrounding the repository [1]. Several scenarios for potential radionuclide migration assumed that groundwater reached the repository and leached the waste form. The contaminated water was assumed to flow into the Culebra and Magenta dolomite aquifers which lie above the proposed salt repository [2] and are potential conduits to the biosphere. This communication presents the results of two experiments using samples of Magenta dolomite.

The first was a column-flow experiment using the Magenta, as a column material, and a fluid doped with ^{137}Cs . This cesium isotope was used because it is one of the more mobile fission products found in radwaste and also because cesium does not form precipitates or hydrolysis products in this system.

Sorption coefficients measured by the batch equilibration method are used to denote the overall summation of the interactions taking place between a dissolved radionuclide and a crushed geologic sample. If the coefficient is high, it is assumed that the migration rate of the radionuclide will be retarded during transport through the same geologic

material by porous flow. The batch Kd measured using another sample of the same material described in this report gave a value of 650 ml/gm, which predicts a significant interaction and, therefore, retardation of migration of cesium through Magenta dolomite [3]. The column flow measurement was performed to verify the results of the batch Kd measurement. A Kd of 120 ml/gm was calculated from the average depth of ^{137}Cs . The TEM was used to identify the phases present and to determine in which phase(s) the cesium was selectively sorbed.

Analysis of the results obtained from autoradiographs and scanning a cross-section of the column indicate that most of the ^{137}Cs is deposited near the sample surface originally contacted by the solution. The TEM results indicate that the ^{137}Cs is selectively sorbed on clay particles distributed throughout the samples and this suggests that at lower concentrations the ^{137}Cs may also be sorbed on the clay particles.

Experimental

A. Column Flow Experiment

The Magenta dolomite was obtained from borehole AEC 8 at a depth of 749 feet [4]. The column measured 10 cm (dia.) by 2.5 cm thick and had the bedding planes (alternating light and dark bands 0.1 to 2.0 mm thick) oriented parallel to flow. A bulk density of 2.37 g/cc for the rock was measured using a water picnometer. The porosity (13.8%) was determined by

measuring the amount of water absorbed by a sample under vacuum. Values ranged from 12-16%, indicating the heterogeneity of the material. The sample was sealed into a metal retainer ring to prevent fluid flow around the sides of the column. A schematic of the apparatus is shown in Figure 1.

The liquid phase was prepared by contacting 50-gram quantities of crushed (-60 mesh) Magenta with 1-liter volumes of deionized water for 24 hours using constant agitation. The water was pre-equilibrated in this manner to reduce possible interaction with the column during the experiment. The solution was filtered through a medium-frit glass filter and doped with 0.1 $\mu\text{Ci/ml}$ of ^{137}Cs (1×10^{-3} ppm). The doped filtrate was added to the reservoir at the top of the column and the apparatus pressurized with 10 psi (or less) of argon to initiate a flow velocity of 0.8 cm/day. (Rustler groundwater velocity ranges from 0.1 to 3.0 cm/day [5].) After one year or approximately 2.3 liters of effluent collected, the flow rate was increased so that the velocity was 1.6 cm/day. Five hundred ml of equilibrated and doped deionized water passed through the column at the higher flow rate. At this point, instead of recharging the reservoir with fresh solution, the effluent was redoped and recycled through the column. A total of 5 liters (123 column volumes) of solution passed through the column depositing 0.5 m Ci of ^{137}Cs . Aliquots of effluent (5 ml) were analyzed for cesium activity on a Searle Model 1185 Automatic Gamma Counting System using a sodium iodide well detector.

Figure 1. Schematic of column flow apparatus.

Autoradiographs, from ^{137}Cs deposited on the column, were made using Polaroid Type 52 Polapan Land film. Exposures were 2 hours long.

Depth of ^{137}Cs penetration was determined by scanning a cross-section of the column mounted on a micrometer drive system. A NaI detector was placed behind a 0.05 cm slit. To determine if any ^{137}Cs had penetrated the length of the column, a monolith (~2g) was cut from the bottom of the sample and counted in the NaI well detector.

B. Transmission Electron Microscopy

Transmission electron microscopy (TEM), electron diffraction and x-ray energy dispersive spectroscopy (EDS) (only elements with $Z > 10$ detected) were used in analyzing structural and compositional inhomogeneities present in Magenta dolomite samples.

Two samples were examined. The first was a 3.0 mm disc cored from a petrographic thin section and thinned on an ion mill. Ion milling used argon gas, 7 KV, 60 μamps and a 15° angle of impingement. This sample, which had not been in contact with a cesium solution, was used as a noncontaminated reference.

The second sample was prepared by soaking a piece of Magenta dolomite in 1 molar CsCl solution for 24 hours and rinsing with deionized water. Particles were then taken from the two areas of interest (light and dark bands) and separately prepared for examination. The individual particles

were placed in a drop of 4% collodion in amyl acetate solution and dispersed by crushing between two glass microscope slides using a continuous shearing motion. When dry, the collodion was stripped from the slide and the sample prepared for microscopy with techniques commonly used in preparing two stage-replicas [6]. Examination was performed on a JEOL JEM 100 C transmission electron microscope equipped with a Kevex 5100 x-ray energy-dispersive spectrometer system.

Results

A. Column Flow Experiment

Effluent samples were continuously collected and analyzed for ^{137}Cs content. After 18 months of continuous flow, the rock sample was examined to determine the extent of ^{137}Cs migration. Measurable activity was detected in effluent samples after 1.4 liters, or 56 column volumes, had passed through the column. The count rate gradually increased to and leveled off at ~ 5 cpm/ml (count rates of 4.5×10^4 to 5×10^4 cpm/ml are commonly measured in the 0.1 $\mu\text{Ci/ml}$ doped feed solution). The presence of trace quantities of ^{137}Cs measured in the effluent samples suggests that a low concentration ^{137}Cs component may have been migrating faster than the majority of the ^{137}Cs . The following expression

$$\frac{V_n}{V_w} = \frac{1}{1 + \frac{\rho}{\theta} K_d} \quad (\text{Eq. 1})$$

where V_n = velocity of radionuclide, V_w = velocity of water

carrier, θ = porosity, ρ = density and K_d = sorption coefficient was used to predict the velocity of the maximum of the radionuclide concentration wave. (Factors such as media homogeneity, reversible reactions of nuclides and porous flow are assumed. Other factors such as crack flow and dispersion, common in inhomogeneous materials are not taken into account.) This relationship predicts that the average velocity of the ^{137}Cs will be 9×10^{-5} cm/day.

B. Autoradiography

Figure 2 is a photograph of the surface of the rock sample. Figure 3 is an autoradiograph of the same surface. It appears that the ^{137}Cs is sorbed to some extent on the entire surface of the sample. However, more cesium seems to be present in the darker bedding planes. Figure 3 shows the plane through which the column was cross sectioned. An autoradiograph made from the cross section (Figure 4) shows the depth of penetration (~ 0.4 cm) of the ^{137}Cs as well as the concentration variations between bedding planes. The autoradiograph also shows a sharp wave front of ^{137}Cs which had progressed into the column. A sharp wave front is the behavior expected for a system having a favorable adsorption isotherm and a high K_d .

C. Scan of Cross-Section

The results obtained from moving the cross-section across the NaI detector are shown in Figure 5. The curve was obtained by consecutively scanning and then cutting away

Figure 2. Photograph showing surface of column sample.

Figure 3. Autoradiograph from surface of column sample showing the plane through which the sample was cross sectioned.

the most contaminated surface thus reducing background and scatter. A half-maximum (average ^{137}Cs penetration) of 0.3 cm was determined from the curve.

A K_d of 120 ml/gm for the column was calculated from equation (1) using a radionuclide velocity of 5.4×10^{-4} cm/day (0.3 cm penetration of ^{137}Cs in 18 months) and an average water velocity of 1.1 cm/day. This K_d is compared with the batch K_d of 650 ml/gm. Possible reasons for the difference are inhomogeneity in Magenta rock samples and the difference in experimental technique.

The monolith of rock that was cut from the bottom of the column contained ~ 100 cpm/g of ^{137}Cs above background. It is possible that the activity is due to contamination from handling, cutting, etc. However, the activity may be a result of some ^{137}Cs penetrating the length of the column, thus resulting in ^{137}Cs activity in effluent samples beginning with column volume 56.

Figure 4. Autoradiograph made from cross section of column.

Figure 5

D. Transmission Electron Microscopy

Examination of ion-milled samples from the untreated dolomite indicated that two principle phases were present, a coarse grained phase and a fibrous crystalline phase. Electron micrographs of the phases are shown in Figures 6a and 6b. X-ray energy dispersive spectra (Figures 8 and 9) were used to identify the coarse grained phase as dolomite $\text{CaMg}(\text{CO}_3)_2$ and the fibrous phase as anhydrite CaSO_4 . Tables I and II give a comparison of the interplanar d-spacings measured from the patterns with those from the Joint Committee on Powder Diffraction Standards (JCPDS) [7].

Figure 6. Transmission electron micrographs of untreated Magenta dolomite. Coarse grained phase (a) and fibrous crystalline phase (b).

Figure 7. X-ray energy dispersive spectra obtained from coarse grained (a) and fibrous (b) phases in untreated Magenta dolomite.

Figure 8. Selected area electron diffraction patterns from dolomite ($\text{CaMg}(\text{CO}_3)_2$) phase in untreated Magenta dolomite sample.

Figure 9. Selected area electron diffraction pattern from anhydrite (CaSO_4) phase in untreated Magenta dolomite sample.

Figure 10. Transmission electron micrographs of two clay phases occurring in untreated Magenta dolomite sample.

In addition to the two major phases described, several other phases were occasionally observed. Electron micrographs and corresponding x-ray energy dispersive spectra for two of these phases are shown in Figures 10 and 11. The d-spacings measured from the electron diffraction pattern shown in Figure 12 matched closely those listed for several hexagonal-structured montmorillonite clays [8]. (Unique identification was not possible because of the close similarity between interplanar d-spacings for the various montmorillonites and because the preferred orientation of the particles prohibited measuring any differentiating basal plane spacings.)

To determine which major mineral phases were concentrated in the light and dark bands and the phase on which the cesium is sorbed, particle fragments were taken from a piece of the same core that had been soaked in 1 molar CsCl and thoroughly rinsed. Examination of the extracted particles gave the following results:

1. The dark bands are composed primarily of anhydrite.
2. The light bands are composed of dolomite.
3. The mixed-morphology montmorillonite clays are located in both the light and dark bands. (Scanning electron microscope analysis indicates that clay particles are more concentrated in the interface between the dolomite and anhydrite beds.) [9].
4. Cesium is associated only with the clay phase.

X-ray energy dispersive spectra of the three major phases

Figure 11. X-ray energy dispersive spectra obtained from rod-like and platelet-like crystals shown in Figure 9.

Figure 12. Selected area electron diffraction pattern obtained from clay crystals shown in Figure 9.

taken from the CsCl soaked sample are shown in Figures 13 and 14. Note that cesium peaks are detected only in the clay spectrum.

Conclusions

The results of the column experiment qualitatively substantiate the results of the batch Kd experiment. The autoradiographs and scans made from the Magenta column indicate a slow and even migration of ^{137}Cs . There appears to be a significant quantitative difference between average ^{137}Cs penetration calculated using equation 1 (0.05 cm) with a batch sorption coefficient of 650 ml/g and the apparent average ^{137}Cs penetration (0.3 cm) measured from the data shown in Fig. 5.

Large variations in batch Kd values, measured with rocks obtained from the same geologic formation, are not uncommon [10].

There is indication of some ^{137}Cs penetrating the entire length of the column as evidenced by the ^{137}Cs activity found both in effluent samples and in the monolith cut from the bottom of the column. This suggests that a second migration mechanism may be a factor. For example, anomalous equilibria at low Cs concentrations, formation of reaction species that have lower Kd values or transport via particulates could result in the presence of ^{137}Cs at the end of the column. Further experiments should be conducted to investigate this phenomenon.

Figure 13. X-ray energy dispersive spectra from dolomite (a) and anhydrite (b) phases in Magenta dolomite sample treated with cesium chloride solution.

Figure 14. X-ray energy dispersive spectra from montmorillonite clay phase in Magenta dolomite sample exposed to cesium chloride solution.

Transmission electron microscopy indicates that the ^{133}Cs is interacting with the clay materials contained in the Magenta sample and not with any of the other minerals present. This could mean that migration of cesium through the dolomite media is a function of clay content.

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Table 1

Interplanar d-Spacings Measured for the Coarse
Crystalline Phase and the JCPDS⁽²⁾ Dolomite Spacings

<u>Measured d-Spacings Å</u>	<u>d-Spacings, Dolomite (Å)</u>	<u>hkl Indices</u>
4.03	4.03	101
3.68	3.69	012
2.87	2.886	104
2.67	2.670	006
2.55	2.540	015
2.39	2.405	110
2.18	2.192	113
2.07	2.066	021

Table 2

Interplanar d-Spacings Measured for the Fibrous
Crystalline Phase and the JCPDS⁽²⁾ Anhydrite Pattern Spacings

<u>Measured d-Spacings (Å)</u>	<u>d-Spacings Anhydrite (Å)</u>	<u>hkl Indices</u>
3.86	3.87	111
3.50	3.498	002,020
3.11	3.118	200
2.82	2.849	210
2.80	2.797	121
2.47	2.473	022
2.30	2.328	202,220
2.20	2.208	212
2.18	2.183	103
2.04	2.086	113
1.97	1.973	301

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