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SORPTION COEFFICIENTS FOR RADIONUCLIDES ON
SAMPLES FROM THE WATER-BEARING MAGENTA AND
CULEBRA MEMBERS OF THE RUSTLER FORMATION

A. W. Lynch and R. G. Dosch

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Sorption Coefficients for Radionuclides on
Samples from the Water-Bearing
Magenta and Culebra Members of
the Rustler Formation

A. W. Lynch and R. G. Dosch
Sandia National Laboratories
Albuquerque, New Mexico 87185

ABSTRACT

The sorbing characteristics of samples from the Magenta and Culebra dolomites of the Rustler Formation (which overlies the proposed site for the Waste Isolation Pilot Plant (WIPP) in southeastern New Mexico) for various radionuclides have been studied. Core samples extracted from various depths and boreholes within a given formation were selected to provide a cross section of materials which are present in the formations. They varied in composition from pure dolomite to nearly pure anhydrite. The radionuclides used in the sorption coefficient measurements (isotopes of Cs, Sr, Tc, Eu, Am and Pu) were dissolved in a simulated Culebra groundwater at concentrations of 0.1 $\mu\text{Ci/ml}$ or less.

The results indicate that the sorption coefficients (K_d 's) of these water-bearing units may vary widely depending on the borehole area and over several feet in depth in a common borehole. Cesium K_d values varied from 0.6 to 218 ml/g and 1.2 to 68 on Magenta and Culebra samples, respectively. Strontium K_d values ranged from 1.1 to 30 on Magenta Samples and from 0.6 to 32 on Culebra samples. Variations of Eu, Pu and Am K_d 's were generally

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less than an order of magnitude on both Culebra and Magenta samples. Technetium was not sorbed by any of the samples.

With few exceptions, samples with high dolomite contents had higher sorption coefficients than those with high anhydrite concentrations. Clay content may also be a factor for some radionuclides, particularly Cs, which has been shown to sorb preferentially on clay particles in Magenta.

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INTRODUCTION

Cavities mined from deep geologic formations are being considered for storage and isolation of radioactive waste. It is postulated that water, either from natural groundwater flow or a catastrophic intrusion may contact and leach the wasteform and transport radionuclides away from the burial site to the surrounding geologic formations. In the case of the WIPP site in southeastern New Mexico, this transport fluid would contact various minerals including salt, clays, anhydrite, polyhalite and dolomite. A quantitative description of reactions between the radionuclides and the rocks they contact is required for the safety assessment of any proposed repository site. The sorption coefficient measurements initially conducted at this laboratory used rocks obtained from the site of the proposed Waste Isolation Pilot Plant (WIPP) in southeastern New Mexico, in contact with radionuclides which had compounds that more or less, dissolved in several simulated groundwater/brine compositions. General sample selection was based on the known hydrology of the WIPP site, a scenario describing potential radionuclide transport from a salt repository, and availability of core samples. The samples included halite containing clay, anhydrite, polyhalite, sandstone, and dolomite. The results of the survey study have been previously reported [1].

The survey study included samples from a number of formations, but in most cases a single core sample was used to represent a given formation. Because the Magenta and Culebra dolomite beds that exist more than 1300 ft. above the repository horizon are identified as water bearing units [2], a more detailed study of the interaction

between radionuclides and samples from those units was of interest. Since the survey study was completed, additional cores from the site have become available, including more samples from the Magenta and Culebra units of the Rustler Formation.

This work was performed to determine differences in sorption properties of Magenta and Culebra materials with respect to sample location. The question addressed was whether a single value such as sorption coefficient (K_d) could adequately describe the sorption characteristics of a geologic formation using an experimental procedure in which the only variable was location from which the geologic samples were obtained within a given formation. Results showing large variations in K_d are probably attributable to differences in material composition which typically varies with both horizon and borehole location within the same formation. For example, the amount of gypsum/anhydrite present at different horizons in a vertical section through Magenta (25 feet thick) formation may vary from ~5 to ~95 percent.

In this study, samples from the Magenta and Culebra units were contacted with Cs-137, Sr-85, Tc-99, Eu-152, Pu-238 and Am-243 dissolved in a Culebra water simulant. Large differences in sorption coefficients (a factor of 10 or greater) for the same isotope in contact with different samples from the same formation were not uncommon.

ROCK DESCRIPTIONS

The core samples (described below) used in this study were from various boreholes drilled at the proposed WIPP site. Hole locations and a detailed description of most of the borehole

samples may be found in the WIPP Project Basic Data Reports [3].

Magenta

<u>Sample Borehole/depth</u>	<u>Major mineral Phase</u>	<u>Minor mineral Phase</u>
AEC8 ⁽¹⁾ - 727.5 ft.	dolomite	gypsum/anhydrite
AEC8 - 735.5 ft.	dolomite	gypsum/anhydrite
AEC8 - 743.4 ft.	dolomite	gypsum/anhydrite
AEC8 - 749.5 ft.	dolomite	gypsum/anhydrite
H4A ⁽¹⁾ - 388 ft.	dolomite	gypsum/anhydrite
H4A - 391 ft.	dolomite	gypsum/anhydrite
H4A - 395.2 ft.	dolomite	gypsum/anhydrite
H4A - 399.6 ft.	dolomite	gypsum/anhydrite
H5A - 800.8 ft.	dolomite	gypsum/anhydrite
H5A - 811 ft.	gypsum/anhydrite	---
H5A - 818 ft.	gypsum/anhydrite	---
<u>Culebra⁽²⁾</u>		
H4B - 517 ft. ⁽³⁾	dolomite	---
H4B - 526.7 ft.	gypsum/anhydrite	---
H5B - 900 ft.	dolomite	---
H5B - 906 ft.	dolomite	---

(1) The amount of gypsum/anhydrite increased and the bedding planes became well defined as the depth increased in boreholes AEC8 and H4A.

(2) The Culebra samples contained only poorly defined bedding planes.

(3) This core sample was interlaced with voids (1/16 - 1/8" dia.).

SAMPLE PREPARATION

The samples used in the batch Kd measurements were prepared by crushing and sieving a portion of a core sample (50-1000 gms). The material was ground with an alumina mortar and pestle until the entire sample passed through a 200 mesh sieve (<74 micron particle size) resulting in a powder having a surface area between one and ten square meters per gram. One gram samples of powdered rock were used in the batch equilibrations.

CULEBRA WATER SIMULANT

Simulant solutions, whose compositions are derived from analysis of waters found at the WIPP site [2], are used in the Kd measurements because of the limited amount of naturally occurring water and because they represent a reproducible matrix for comparison with other experiments. The composition of the Culebra water simulant used in this work represented an average composition of water found in boreholes H2b and H2c in the Culebra member of the Rustler Formation. The composition (Table I) lies between those of brine B and groundwater C used in previous measurements [1].

RADIOISOTOPE CONCENTRATIONS

Radioisotopes were added to the Culebra water simulant in two different ways:

- 1) Addition of an aliquot of a stock solution of a given radioisotope directly to the simulant, or
- 2) Drying an aliquot of radioisotope stock solution (room temp.), contacting the residue with the simulant, and then filtering (0.8 μ filters, Gelman

Acropore, Gelman Instrument Co., Ann Arbor, Mich.)

the entire solution.

Cesium, strontium, and technetium were added by method 1) while europium, plutonium and americium were added using method 2). The lanthanides and actinides were difficult to keep in solution due to sorption on the walls of the container and/or precipitation of hydrolysis products. Addition of the radioisotope by method 2) appeared to make a more stable solution because the simulant solution pH did not change; however, not all the radioisotope redissolved in the simulant.

The initial isotope concentrations used in the measurements were: $^{137}\text{Cs} = 0.1 \mu\text{Ci/ml}$, $^{85}\text{Sr} = 0.1 \mu\text{Ci/ml}$, $^{99}\text{Tc} = 0.001 \mu\text{Ci/ml}$, $^{238}\text{Pu} = 0.03 \mu\text{Ci/ml}$, $^{243}\text{Am} = 0.001 \mu\text{Ci/ml}$, $^{152}\text{Eu} = 0.1 \mu\text{Ci/ml}$ (Culebra), $^{152}\text{Eu} = 0.07 \mu\text{Ci/ml}$ (Magenta). Concentrations by weights (ppm radionuclides dissolved in the simulant) may be found in Table II. The values given for ^{152}Eu , ^{243}Am and ^{238}Pu represent the amounts that redissolved in the simulant and were found in solution after filtering.

EXPERIMENTAL PROCEDURE

Rock samples (1 gram) were weighed directly into polyethylene dropping bottles and a 30 ml aliquot of doped simulant was added. A piece of polyethylene film, placed between the bottle and its top, was used as a seal. The bottles were constantly agitated for periods of one or two weeks. Analytical samples were obtained by placing 0.8 micron filters (Gelman Acropore) over the opening of the dropping bottles, replacing the dropper top, and forcing

TABLE I
Culebra Water Simulant Composition

<u>Species</u>	<u>Concentration (mg/liter)</u>
Ca ⁺⁺	685
Mg ⁺⁺	140
Na ⁺⁺	2850
K ⁺	105
Fe ⁺⁺	0.065
Mn ⁺⁺	0.17
HCO ₃ ⁻	59.5
SO ₄ ⁼	3100
Cl ⁻	3750
F ⁻	1.8
NO ₃ ⁻	0.085
BO ₃ ⁼	9.75
SiO ₂	2.6
pH = 8.4	

TABLE II
Analytical Concentrations of Radioisotopes
in Culebra Simulant

<u>Isotope</u>	<u>Concentration (ppm)</u>
¹³⁷ Cs	1.2×10^{-3}
⁸⁵ Sr	$3 \times 10^{-3} - 1 \times 10^{-2}$
¹⁵² Eu	$7 \times 10^{-2} - 1 \times 10^{-1}$
⁹⁹ Tc	5.9×10^{-2}
²³⁸ Pu	1.8×10^{-3}
²⁴³ Am	5.4×10^{-3}

out a few milliliters of liquid by squeezing the bottle. Measured volumes of the filtrate were used to determine isotope activity. Similarly, control solutions were placed in identical dropping bottles and sampled in the same manner as the solid-liquid samples.

The ^{137}Cs , ^{85}Sr and ^{152}Eu activities were counted on a Searle Model 1185 Automatic Gamma Counting System using a sodium iodide well detector. The ^{99}Tc , ^{238}Pu and ^{243}Am were mixed with Packard Insta-Gel Scintillation Cocktail and counted on a Packard Model A3255 Liquid Scintillation Counter.

SORPTION COEFFICIENTS

Sorption Coefficients (K_d) were calculated from analyses of the liquid phase activities. The initial activities used were the activities in the control solutions. The calculation of K_d was made using equation (1).

$$K_d = \frac{\text{volume of soln (ml)}}{\text{weight of sample (gms)}} \times \frac{\text{initial soln activity} - \text{final soln activity}}{\text{final soln activity}} \quad (1)$$

The K_d as calculated represents a measure of the activity lost from solution due to sorption on the rock sample. The use of the activity in the control solution as the initial activity is intended to correct for loss of activity due to sorption on container surfaces or precipitation reactions. The data presented in Table III are K_d values that were obtained using the experimental parameters previously described.

One of the difficulties encountered in these measurements is the loss of activity in the control solutions. The loss is

particularly significant for the lanthanides and actinide elements. This problem is not generally observed for Cs, Tc and Sr solutions [1,4].

For this reason, two sets of data for ^{152}Eu , ^{238}Pu and ^{243}Am are presented in Table III. The first value, K_d , was calculated from equation (1) using the activity of the control solution after the contact period as the initial activity and will be referred to as the apparent K_d . The second value was also calculated using equation (1); however the total activity initially added to the rock sample was used as the initial activity. This value is referred to as the overall K_d and includes all mechanisms responsible for the loss of activity from the sample solution including precipitation and sorption on container. The initial and final activities of ^{137}Cs , ^{85}Sr and ^{99}Tc in the control solutions were the same, so both the apparent and overall K_d 's have the same value.

RESULTS

The general trends found in the data are given below:

Technetium

The technetium K_d values are not given in Table III because within experimental error, Tc was not absorbed by any of the rock samples. The result is typical for all the geologic samples that have been received from the WIPP site.

Cesium

The difference in sorption properties of the various rock samples was most evident for cesium sorption. The K_d values ranged from 1.2 to 68.3 ml/gm with Culebra samples and from

0.6 to 2.8 ml/gm with Magenta samples. The higher values are associated with those samples which contained a high percentage of dolomite. As the amount of gypsum/anhydrite increases in the sample, the Kd decreases. However, the amount of dolomite in the samples is apparently not the only factor governing Cs sorption. The Magenta samples typically do not contain as much dolomite as the Culebra samples, yet the Magenta Kd's are generally higher than the Culebra Kd values. Analysis of some Magenta samples by Transmission Electron Microscopy has suggested that clay particles in the Magenta preferentially sorb Cs [5].

Strontium

The strontium Kd's are typically lower than cesium Kd's (on the same rock samples). The range of values is from 0.6 to 31.7 ml/gm. In most cases, the highest value obtained with ^{85}Sr corresponds to the lowest value obtained with ^{137}Cs and vice versa. Analysis of a Magenta sample by Scanning Electron Microscopy has indicated some small deposits of strontium sulfate [6]. The differences in Kd values may be due to the natural abundance of strontium in the rock samples. The Sr results do not appear to be associated with dolomite content.

Europium, Plutonium, Americium

The most significant factor associated with measuring distribution coefficients (under pH conditions which apply to the WIPP site) for the lanthanide and actinide elements is the loss of activity in the control solutions. The low apparent Kd's listed for europium with Magenta are the result of the loss of most (>98%) of the activity in the control sample solution.

TABLE IIIA

Magenta Sorption Coefficients

Sample Borehole/Depth	Major Phase	Cs	Sr	Sorption Coefficient, ml/g					
				Eu		Pu		Am	
H4A - 388'	dolomite	200	1.3	9.2	5460 ⁽¹⁾	815	4154 ⁽¹⁾	273	5683 ⁽¹⁾
H4A - 391'	dolomite	209	1.4	10.7	5680	591	3042	337	6881
H4A - 395'	dolomite	135	2.2	9.7	5530	471	2451	301	6201
H4A - 399'	dolomite	60.2	1.5	11.2	5750	828	4216	330	6758
H5A - 300'	dolomite	214	2.0	10.2	5600	282	1516	258	5393
H5A - 811'	anhydrite	10.1	9.1	6.9	5140	126	740	246	5165
H5A - 818'	anhydrite	0.6	21.6	6.9	5130	21	240	140	3174
AEC - 727'	dolomite	73.4	5.1	6.9	5140	632	3246	222	4992
AEC8 - 735'	dolomite	162	1.1	13.5	6070	1055	5398	290	5999
AEC8 - 743'	dolomite	218	30	7.1	5170	455	2372	341	6948
AEC8 - 749'	dolomite	90.8	3.4	5.8	4990	414	2166	119	2779

TABLE IIIB

Culebra Sorption Coefficients

H4B - 517'	dolomite	9.0	0.6	1592	1.18×10^4	202	1116	228	4835
H4B - 526'	anhydrite	1.2	31.7	1168	8.7×10^3	82.9	529	185	4021
H5B - 900'	dolomite	26.1	0.3	1377	1.02×10^4	386	2029	278	5764
H5B - 906'	dolomite	68.3	0.6	1236	9.21×10^3	877	4457	383	7741

Note: The sorption coefficients presented are the results of single measurements. Duplicate Kd measurements are typically within a factor of 2.

(1) Overall Kd's which use total activity added to the rock as initial activity in Eq. (1).

The concentration of ^{152}Eu used in the Culebra experiments was 0.1 $\mu\text{Ci/ml}$ while 0.07 $\mu\text{Ci/ml}$ was used in the Magenta measurements. More than 98% of the activity was lost (sorption on walls, precipitation, etc.) from the 0.07 $\mu\text{Ci/ml}$ control while ~85% was lost from the 0.1 $\mu\text{Ci/ml}$ control. This loss could account for the discrepancy between the apparent K_d 's for the Culebra and Magenta samples.

The significant result seen in the plutonium and americium results is that, like cesium, the lowest K_d 's are associated with the samples that are high in anhydrite content. However, the magnitude of the overall K_d 's for plutonium, americium and europium with both the Magenta and Culebra indicate that the lanthanides and actinides will react to some extent with all the minerals in the Culebra and Magenta members. The differences seen between the apparent and overall K_d 's are the result of loss of activity in the control solutions. Therefore the values presented as overall K_d 's appear to be useful in interpreting the results of the measurements with the lanthanide and actinide elements.

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