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LABORATORY EVALUATION OF COLLOIDAL ACTINIDE TRANSPORT AT THE WASTE ISOLATION PILOT PLANT (WIPP): 1. CRUSHED-DOLOMITE COLUMN FLOW EXPERIMENTS

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Colloid-facilitated transport of Pu, Am, U, Th, and Np has been recognized as a potentially important phenomenon affecting the performance of the Waste Isolation Pilot Plant (WIPP) facility being developed for safe disposal of transuranic radioactive waste. In a human intrusion scenario, actinide-bearing colloidal particles may be released from the repository and be transported by brines (~0.8 to 3 molal ionic strength) through the Culebra, a thin fractured microcrystalline (mean grain size 2 μm) dolomite aquifer overlying the repository. Transport experiments were conducted using sieved, uniformly packed crushed Culebra rock or nonporous dolomite cleavage rhombohedra. Experiments with mineral fragments and fixed and live WIPP-relevant bacteria cultures showed significant levels of retardation due to physical filtration effects. Humic substances were not attenuated by the Culebra dolomite. Comparison of elution curves of latex microspheres in columns prepared with microcrystalline rock and nonporous rock showed minimal effect of Culebra micropores on colloid transport. These data form part of the basis (see also Lucero et al., this volume) to parameterize numerical codes being used to evaluate the performance of the WIPP.

MASTER

INTRODUCTION

Under the authorization of Public Law 96-164 (1979), the U.S. Department of Energy (DOE) has been developing the Waste Isolation Pilot Plant (WIPP) facility, located approximately 42 km east of Carlsbad, New Mexico for the safe disposal of nuclear wastes produced by the defense nuclear-weapons program. The U.S. Environmental Protection Agency has established the regulatory standards (U.S. EPA, 1993) for cumulative radioactive release to the accessible environment over 10,000 years to demonstrate the WIPP facility compliance.

The only credible potential mechanism for escape of radionuclides to the accessible environment involves human intrusion scenarios, i.e., repository breach during drilling for natural resources. In human intrusion scenarios, the brines present in the repository together with introduced drilling muds are transported up the intrusion boreholes under the driving force of pressurized fluids in the disposal area. Some of the radioisotopes could reach the surface, while others could move laterally toward the WIPP Site boundary, principally through the Culebra Dolomite Member of the Rustler Formation (Culebra). Performance assessment calculations (WIPP PA Division, 1991a,b) showed that releases of radioactive isotopes of the actinide elements Th, U, Np, Pu, and Am would contribute the most to the total release of radioactivity, with Pu and U being the largest contributors. These calculations did not include colloid-facilitated transport.

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Transport of actinides by colloidal particles has been recognized as a phenomenon of critical importance to the performance of nuclear waste repositories (Jacquier, 1991; Avogadro and de Marsily, 1984). Colloidal particles, which are generally defined as particles with sizes between 1 nm and 1 μm in a liquid dispersant, will be generated in the repository environment as a result of microbial degradation of cellulosics, corrosion of steel waste containers and waste constituents, by the hydrodynamic entrainment of colloidal-sized mineral fragments, and several other mechanisms. Those colloidal particles may sorb dissolved actinides or the dissolved actinides themselves may form colloidal-sized particles. Additional colloidal particles may be present in natural Culebra groundwater and could form additional actinide-bearing colloidal particles.

The objective of the WIPP colloid research program is to provide the WIPP PA Department with sufficient information to quantify the concentration of colloidal actinides that reaches the accessible environment (Papenguth and Behl, 1996). One of the tasks required to meet this objective includes the quantification of colloid-facilitated transport of actinides in the overlying Culebra, in the event of a repository breach. The laboratory experimental program identified two types of transport experiments. Crushed-rock column flow experiments, the subject of this paper, were used to provide quantification of retardation factors and filtration coefficients for WIPP colloids, in the absence of actinides. Intact core column flow experiments were used to confirm results of crushed-rock column flow experiments for humic colloids and live microbes, in the presence of some of the important actinides (see Lucero et al. (this volume)).

CRUSHED-ROCK COLUMN FLOW EXPERIMENTS DESIGN

Columns used for crushed-rock column experiments were 1 cm in diameter and generally 5 to 44 cm in length. Columns were hand packed with crushed and treated Culebra rock collected at Culebra Bluffs near Carlsbad, New Mexico, and from the horizontal cores collected from the Air Intake Shaft for use in the intact core column flow experiments (Lucero et al., 1995). In addition, some non-porous mineralogically pure crystalline dolomite cleavage rhombohedra (referred to as Butte) were obtained commercially (Ward's Natural Science Establishment, Inc., Rochester, NY, part number 47E2715). Crushed-rock column packing material was prepared in three size fractions using pairs of sieves: 125-250 μm, 250-500 μm, and 500-1000 μm. The material was cleaned, crushed, rinsed thoroughly, acid washed with dilute HCl, rinsed thoroughly, and dried at 40°C.

Culebra brine simulants were prepared to closely match brines from the Culebra at the H-17 well (H-17 brines) or from seeps in the Air Intake Shaft (AIS brines). These two brines are primarily NaCl in composition with ionic strengths of approximately 3 and 0.8 molal, respectively. The pCO_2 was generally kept at atmospheric ($10^{-3.5}$ atm), but was scrubbed in some experiments. For experiments designed to isolate certain phenomena, relatively dilute NaCl solutions were used, with concentrations of 1 or 10mM. Flow rates were 0.1 to 0.5 mL/min. Colloidal particles

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(described below) consisting of a variety of humic substances, preserved and live microbes, mineral fragments, and latex microspheres (non-functional) were injected as spikes or as step injections. The concentration of colloids and tracers in the effluent were analyzed using inductively coupled plasma-atomic emission spectroscopy (ICP-AES), scanning fluorometry, fixed wavelength fluorometry, epifluorescence microscopy, and laser particle spectrometry. For some experiments, effluent samples were collected in generally 2-mL aliquots with the aid of an automated fraction collector. The particle spectrometry (Particle Measurement Systems model HSLIS S100) was used on-line to count number populations in real time.

Porosity of the packed columns was calulated from gravimetric and volumetric measurements using a grain density of dolomite of 2.82 g/cm^3 (Kelley and Saulnier, 1990). These results were confirmed with transport experiments using a nonsorbing tracer, fluorescein. These tests indicated that effective and actual porosities (typically about 45%) were equivalent, and so it was not necessary to perform a conservative tracer test with each crushed-rock column.

A series of tests were conducted to test the experiment configuration and to evaluate the effect of potential artifacts. In those tests column lengths, flow rates, injection mode, packing diameter, and colloid diameter were varied. The tests demonstrated that the test design was robust, in that varying test configuration did not have an appreciable effect on results. In some of the filtration experiments, the direction of flow was reversed to confirm that filtration was irreversible.

INTERPRETATION

In the column transport experiments, the main phenomena affecting peak elution time and peak breadth are the following: advection; dispersion and diffusion; reversible sorption; and filtration including interception and sieving, sedimentation, and irreversible sorption. To describe transport of colloidal bound actinides through the crushed-rock columns, the advection-dispersion-filtration equation with adsorption is (Harvey and Garabedian, 1991):

$$\frac{dC}{dt} = D\frac{d^2C}{dx^2} - \frac{v}{R}\left(\frac{dC}{dx} + \gamma C\right) \qquad (1)$$

It reduces to the advection-dispersion equation with adsorption for $\gamma = 0$:

$$\frac{dC}{dt} = D\frac{d^2C}{dx^2} - \frac{v}{R}\frac{dC}{dx} \qquad (2)$$

where C is the contaminant concentration; D is the dispersion coefficient; v is the average linear flow velocity; R is the retardation coefficient for reversible sorption; and γ is the filtration coefficient.

The solution of Eq. (1) can be fitted to the experimental breakthrough curves (BTC) to estimate parameters of interest. However, the experimental BTCs were often noisy and were not suitable for automated curve fitting procedures. Therefore, to interpret the crushed-rock column flow experiments conducted with colloidal particles, the following steps were taken. First the retardation factor was estimated from the peak position of the elution curve. A conservative value for the dispersion was estimated on the basis of suggestions provided in Parker and van Genuchten (1984), tests with conservative tracers, and sensitivity analyses. For the tests described herein, values for the dispersion coefficient were 200, 300, and 500 cm^2/d for the columns which ranged in length from 5 to 44 cm. The larger dispersion values were used for the longer columns. The theoretical peak height without filtration was then calculated using CXTFIT (Parker and van Genuchten, 1984) with the retardation and dispersion values determined above. The difference in the calculated peak height without filtration and the observed peak height were attributed to filtration effects. The value of the filtration term was then determined as follows (Harvey and Garabedian, 1991):

$$\text{observed peak concentration} = \text{theoretical peak concentration} * \exp(-\gamma L) \quad (4)$$

where γ is the filtration coefficient and L is the column length.

RESULTS AND ANALYSIS

Crushed-Rock Column Flow Experiments with Latex Microspheres

To design experiments for WIPP-relevant colloids, a set of twelve experiments were performed using monodisperse non-functionalized latex microspheres with diameters of 0.48 and 1.05 μm (Table 1). Non-functionalized microspheres were used to minimize chemical interaction with the column packing material and isolate filtration effects. Two different sizes of latex microspheres were used to study the effects of colloid size on physical retardation and filtration. To test the possibility that the intercrystalline porosity of the Culebra enhances physical retardation, parallel tests were conducted with Culebra rock and non-porous dolomite (containing no intercrystalline porosity). Culebra rock was crushed to sizes which preserved a significant fraction of intercrystalline pore throats (crushed particle sizes ranged from 125 to 1000 μm, compared to microcrystalline grain sizes of 2 μm, and mean pore throat diameters of 0.63 μm). An ionic strength of 0.01 M was selected for the eluant to minimize coagulation and settling.

In general, elution curves for the latex microsphere experiments appeared qualitatively like typical elution curves for a dissolved solute spikes injected onto a column with a retardation factor of about 2.2, based on the peak position being at 2.2 pore volumes of the effluent (Figure 1). Note, however, that the peak concentration in the effluent at 2.2 pore volumes is only about 2% of the injected concentration. Typically, in dissolved solute transport, a much higher

retardation factor is observed with such a large reduction in the peak concentration, suggesting that most of colloid particles are removed by physical filtration or irreversible adsorption.

The small elution peaks occurring intermittently during the remainder of the test and in the elution curves for other tests show that the colloidal particles trapped in pores created by packing of spherical particles are released back into the flowing water in spikes of varying sizes. However, the total number of particles in the effluent remains small. For example, in one typical experiment (II-a, Table 1), after 46.5 pore volumes were eluted, the total number of particles released is still only 1.4% of the injected particle concentration.

Physical filtration effects are reflected in the number population of latex microspheres observed in the effluent. The mass balances (integrated counts under the first peak and over the entire elution curve) indicate that about 11% or less of the injected colloidal particles were released from the columns (Table 1). In the case of columns packed with the smallest dolomite particle size fraction, essentially all the latex microspheres were physically trapped within the columns. The total number of trapped latex microspheres decreases with increasing size of the crushed-rock particle size fraction. That inverse relationship probably occurs because the average pore throat size is directly proportional to the size of packing material; the smaller pore throats trap particles more effectively than the larger pores.

Entrapment of latex microspheres appeared to be sensitive to the size of the microspheres as well. Comparison of each pair of experiments involving small and large microspheres indicates that a higher percentage of small microspheres are released in the effluent. That could be partly due to the fact the injected concentration of small microspheres in these experiments was about an order of magnitude higher than that of large colloids. However, the trend of enhanced releases of small microspheres is expected even at equivalent number populations of large and small particles, because large particles require larger spaces to move around due to their larger cross-sectional area. Once trapped, it would be harder for large microspheres to break free than the small microspheres.

The small latex microspheres were expected to show enhanced retardation in the Culebra dolomite relative to the non-porous Butte dolomite, but they did not (Table 1). This expectation was based on the fact that the Culebra dolomite has intercrystalline porosity with a mean pore throat diameter of 0.63 μm (Kelley and Saulnier, 1990), but the Butte dolomite is nonporous. Therefore, the microspheres should experience physical retardation (i.e., diffusion in the pores) as well as chemical retardation in the Culebra dolomite, but only chemical retardation in the Butte dolomite.

Crushed-Rock Column Flow Experiments with Microbes

Crushed-rock column flow experiments were conducted with live and preserved microbe cultures developed from samples collected from several sources, including G-Seep, WIPP muck-pile salt, and saline lakes near the WIPP Site (Francis and Gillow, 1994). Naturally occurring halophilic or halotolerant microbes collected at the WIPP and WIPP vicinity are rod-shaped (aspect ratio of about five) and range in length from about 0.5 to 2 μm. Within a particular class of microbes, size will vary depending on the stage of their reproduction and also nutrient conditions. The outer surface of microbes consists of complex organic molecules with exposed surface functional groups. The surface chemistry of microbes is an important characteristic which affects how they interact with the host rock during transport, as well as how they bioaccumulate dissolved actinides. Measurements of electrophoretic mobility of microbes in WIPP brines, however, showed that the surface charges of microbes is near neutral, suggesting limited chemical interaction with rock surfaces.

Most of the experiments were conducted with preserved microbes harvested during the stationary phase of the growth curve, and fixed with formalin. Some experiments were also conducted with live microbes of the WIPP-1A culture in nutrient-free environment. Experiments in which nutrients were added along with the live microbes resulted in nearly complete stoppage of flow through the column and therefore were not used for developing parameters. Tests were conducted with 10^7 to 10^8 cells/mL spike injections; these concentrations are similar to concentrations of microbes observed in natural environments at the WIPP. Step injection tests conducted with 10^6 to 10^7 cells/mL resulted in column plugging and were not used for determination of parameter values.

Generally, H-17 brine simulant (primarily NaCl with an ionic strength of 3 molal) was used as the eluant. Experiments were conducted with 250-500 μm sized packing to acquire data for determination of parameter values.

The concentration of microbes eluting from the column was usually measured in real time, with a particle spectrometer connected to the effluent line. Aliquots were collected periodically for direct counting using epifluorescence microscopy as an independent check that the particle spectrometer was counting accurately. The particle spectrometer records number concentrations in several size intervals. A recurring observation in the microbe tests was that larger microbes (0.6 to 1.0 μm; defined by counting-channel-width of the spectrometer) were more effectively filtered than smaller microbes (0.2 to 0.28 μm; defined by counting-channel width) (Figures 2 and 3). Under nutrient-poor conditions, such as might occur in the Culebra down gradient from the intrusion borehole, microbes become reduced in size. The elution curve representing transport of the smaller microbes was used to develop parameter values. That approach is likely to result in some degree of conservatism.

The results of the tests were interpreted using the approach described earlier. Representative elution curves from tests conducted with-live microbes and preserved microbes are presented in Figures 2 and 3, respectively. A compilation of the values used and results of the numerical interpretation is presented in Table 2. Retardation factors for microbes themselves (actinides were not used in these tests) was about 4; this low value is consistent with our expectations. Filtration coefficients were 0.67 and 0.51 cm^{-1} for preserved and live microbes, respectively (Table 2).

Results of the crushed-rock column flow tests on microbes were evaluated with a flow experiment on intact core under simulated subsurface conditions and were consistent with results of the intact core column flow tests (see Lucero et al., this volume).

Crushed-Rock Column Flow Experiments with Mineral Fragments

Dispersions of mineral fragment colloidal particles were made by mechanically milling quartz (SiO_2) or through chemical precipitation of goethite (FeO·OH). A 1mM NaCl solution was used to minimize the agglomeration of particles due to Brownian motion or induced by hydrodynamic motion. After allowing dispersions to agglomerate and settle, an aliquot of the supernatant was extracted. The aliquot was filtered with a 1.2 μm filter to remove suspended agglomerates and injected as a spike onto a crushed-rock column. The residual concentration of colloidal particles in the supernatant was on the order of 10^6 particles/mL.

The elution curve profiles for mineral colloids (Figure 4) were similar to the latex microsphere experiments and the microbe experiments (Figures 1, 2, and 3, respectively). Elution curves were analyzed using the approach described earlier. As with microbes, the filtration coefficients obtained show effective attenuation of the mineral fragment colloids by the crushed rock (Table 2). A retardation factor (for the colloid; actinides were not used in the test) on the order of 4 to 6 was observed in the tests (Table 2).

Crushed-Rock Column Flow Experiments with Humic Substances

Humic substances represent a variety of high-molecular weight organic compounds. They form by a variety of processes in terrigenous and marine environments, resulting in compounds with different geochemical behaviors. It is believed that humic substances will form after emplacement of the waste at the WIPP, but the nature of the humics cannot be predicted. To span the likely range of behaviors, three types were used in the colloid source term and transport work: aromatic humic acid, aliphatic humic acid, and fulvic acid. Samples of those types were collected by Florida State University, purchased commercially, obtained from colleagues, or purchased from the International Humic Substances Society (Golden, Colorado).

Typical results are illustrated in Figure 5. Elution curve profiles from the final experiments were analyzed using the approach described earlier. Very little filtration of the humic substances was observed (Table 2). This result was anticipated because humic substances, in their ionic form, are dissolved macromolecules and are essentially not susceptible to physical (straining) filtration as are microbes and mineral fragment. The experiments showed that the humic substances eluted at the same number of pore volumes as fluorescein dye, the nonsorbing tracer, indicating that enhanced transport of humic substances did not occur. A hypothesis that was rejected based on these tests was the possibility that the >Mg and >Ca surface functional groups of the dolomite would have reacted with the humics, resulting in sorption.

CONCLUSIONS

Transport experiments using sieved, uniformly packed crushed Culebra rock demonstrated that mineral fragments and fixed and live WIPP-relevant bacteria cultures were significantly retarded due to physical filtration effects, but humic substances were not attenuated by the Culebra dolomite. Comparison of elution curves of latex microspheres in columns prepared with microcrsytalline rock and nonporous rock showed minimal effect of Culebra micropores on colloid transport.

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Table 1. Latex Microsphere Transport Experiments

Column - Test Number	Packing Material	Packing Diameter (um)	Latex Particle Diameter (um)	Retardation Factor	Mass Balance (%)
I-a	Butte	125-250	1.053	No discernible peak	0.072
I-b	Butte	125-250	0.48	1.9	0.007
IV-a	Butte	250-500	1.053	1.5	0.64
IV-b	Butte	250-500	0.48	1.5	1.18
III-a	Butte	500-1000	1.053	3.3	3.44
III-b	Butte	500-1000	0.48	1.7	6.09
V-a	Culebra	125-250	1.053	1.6	0.11
V-b	Culebra	125-250	0.48	4.2	0.11
II-a	Culebra	250-500	1.053	1.6	1.37
II-b	Culebra	250-500	0.48	2.2	10.9
VI-a	Culebra	500-1000	1.053	1.3	8.95
VI-b	Culebra	500-1000	0.48	1.6	8.5

Table 2. Selected Crushed-rock Column Experiments

Colloid	size fraction (um)	Column Length (cm)	Packing porosity	Pump Flow Rate (mL/min)	Spike Volume (mL)	Brine	R	Flow Velocity (cm/min)	Est Dispersion (cm^2/d)	γ (1/cm)
Preserved microbes	0.2 to 0.28	5.1	0.44	0.5	0.5	H-17	2.3	1.46	200	0.67
Live microbes	0.2 to 0.28	5.2	0.46	0.5	0.5	H-17	3.8	1.40	200	0.51
Goethite	0.2 to 0.28	5.2	0.45	0.5	1.0	1 mM*	4.9	1.42	200	0.10
Quartz	0.2 to 0.28	5.5	0.47	0.5	1.0	1 mM*	2.5	1.34	200	0.26
Latex microspheres	0.48	12.0	0.50	0.5	1.0	10 mM*	2.2	1.28	300	0.22
Humic acid	entire range	44.1	0.43	0.111	5.0	H-17	1.0	0.33	500	0.02
Fulvic acid	entire range	44.1	0.43	0.113	5.0	H-17	1.0	0.33	500	0.01

* Pure NaCl

Figure 1

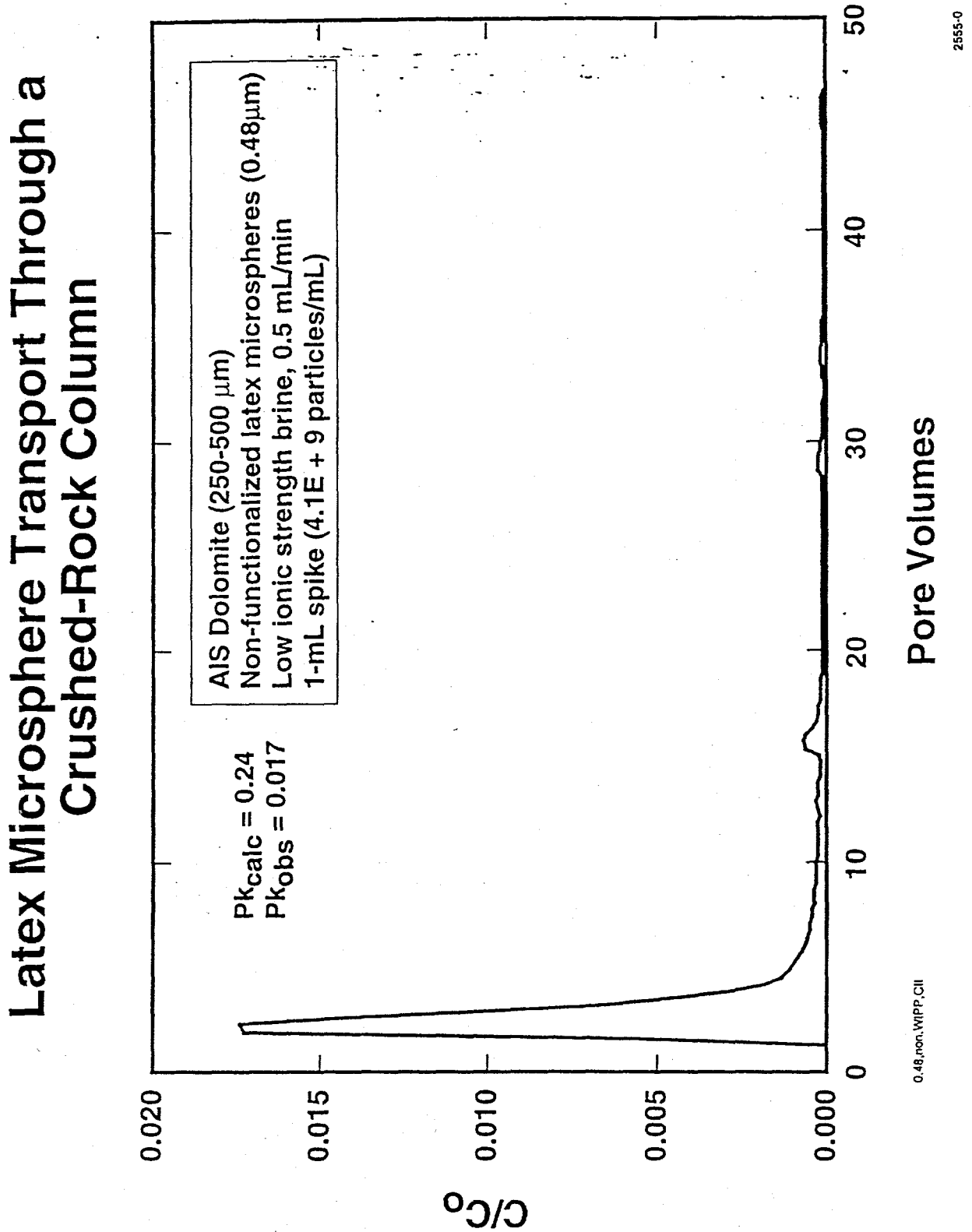

Latex Microsphere Transport Through a Crushed-Rock Column
AIS Dolomite (250-500 μm)
Non-functionalized latex microspheres (0.48μm)
Low ionic strength brine, 0.5 mL/min
1-mL spike (4.1E + 9 particles/mL)
Pkcalc = 0.24
Pkobs = 0.017
C/Co
0.020
0.015
0.010
0.005
0.000
0
10
20
30
40
50
Pore Volumes
0.48,non,WIPP,CII
2555-0

Figure 2

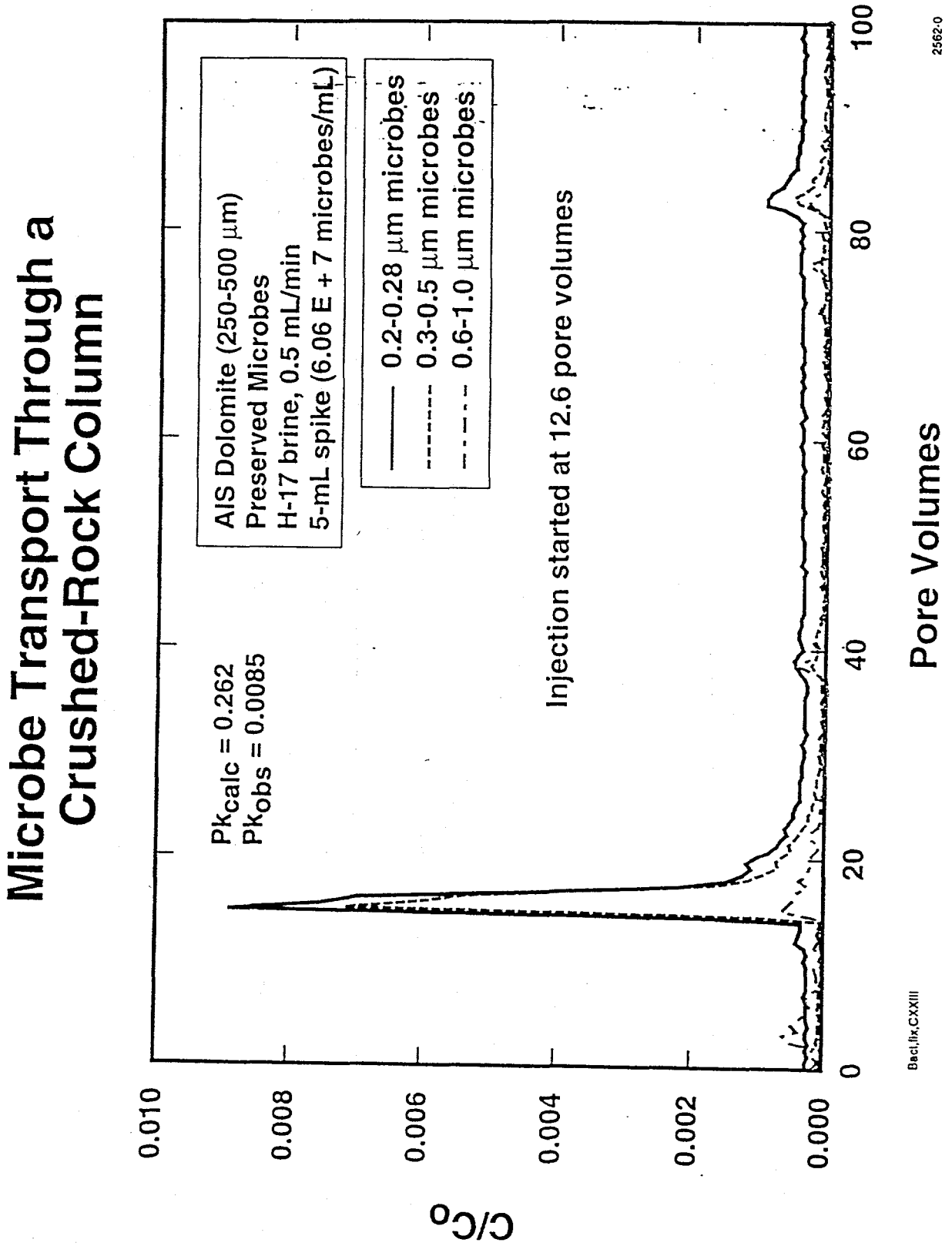

Microbe Transport Through a Crushed-Rock Column
AIS Dolomite (250-500 μm)
Preserved Microbes
H-17 brine, 0.5 mL/min
5-mL spike (6.06 E + 7 microbes/mL)
0.2-0.28 μm microbes
0.3-0.5 μm microbes
0.6-1.0 μm microbes
Pkcalc = 0.262
Pkobs = 0.0085
Injection started at 12.6 pore volumes
C/Co
Pore Volumes
0.010
0.008
0.006
0.004
0.002
0.000
0
20
40
60
80
100
Bacl,llx,CXXIII
2562-0

Figure 3

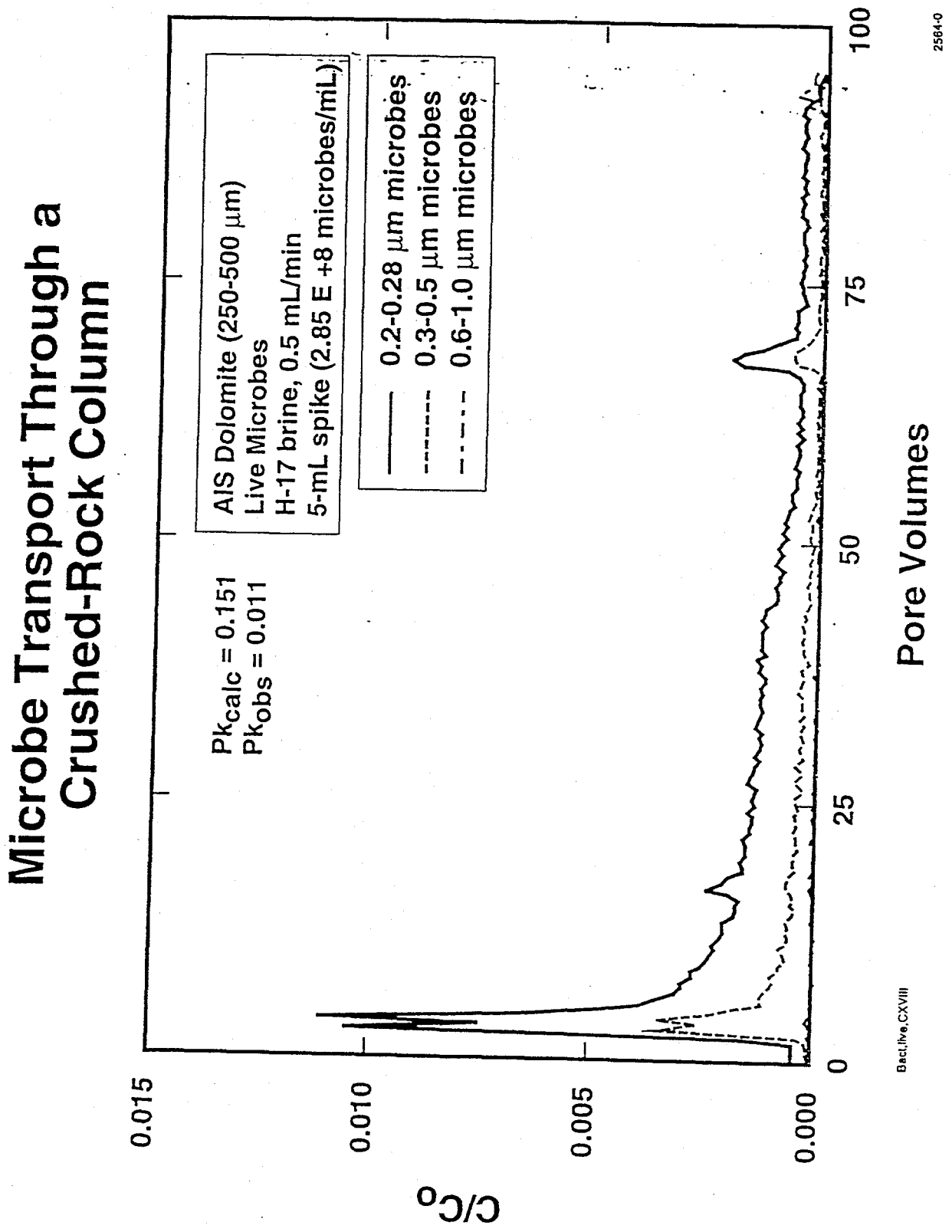

Microbe Transport Through a Crushed-Rock Column
AIS Dolomite (250-500 μm)
Live Microbes
H-17 brine, 0.5 mL/min
5-mL spike (2.85 E +8 microbes/mL)
0.2-0.28 μm microbes
0.3-0.5 μm microbes
0.6-1.0 μm microbes
Pkcalc = 0.151
Pkobs = 0.011
C/Co
Pore Volumes
0.015
0.010
0.005
0.000
0
25
50
75
100
Bact,live,CXVIII
2564-0

Figure 4

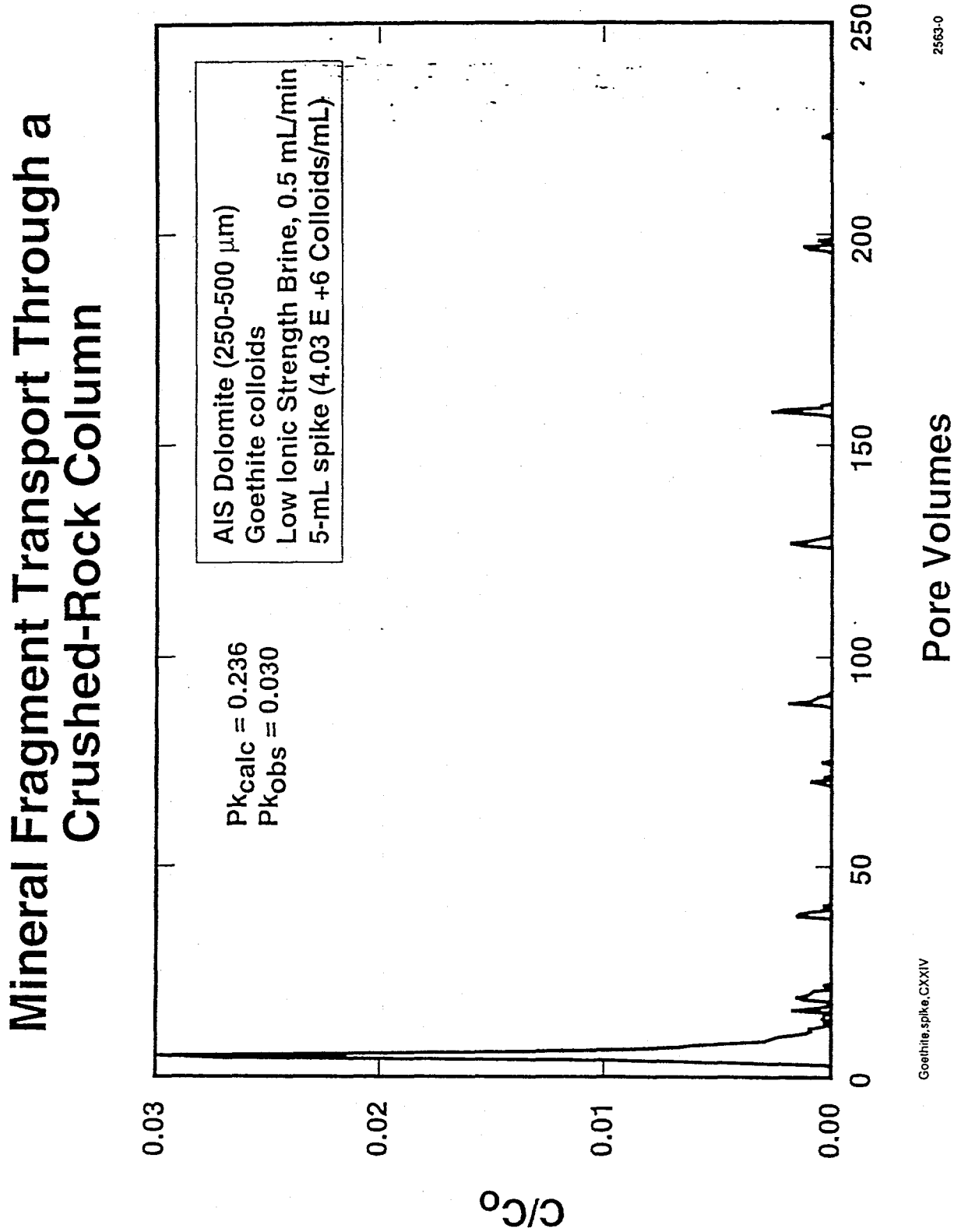
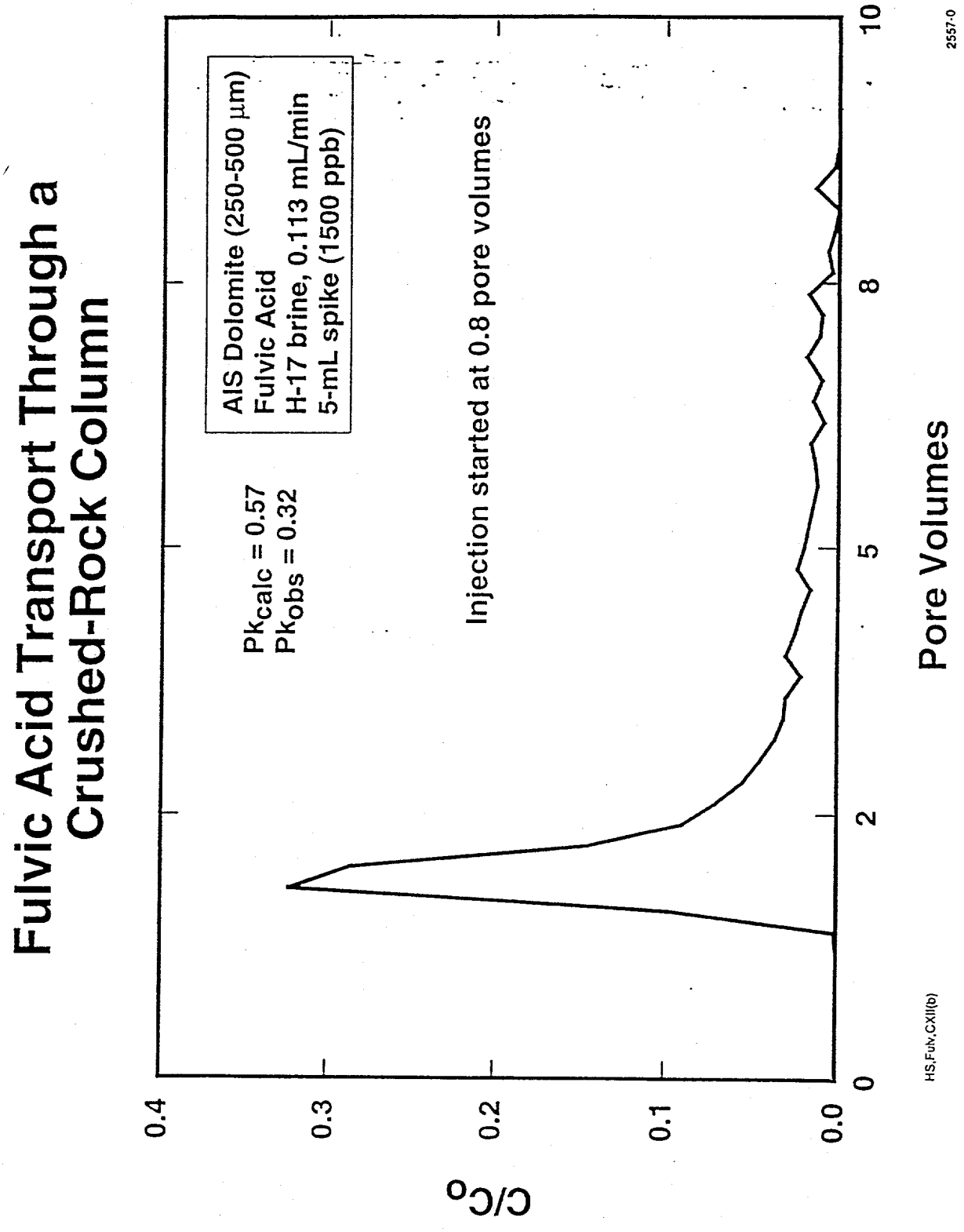

Mineral Fragment Transport Through a Crushed-Rock Column
AIS Dolomite (250-500 μm)
Goethite colloids
Low Ionic Strength Brine, 0.5 mL/min
5-mL spike (4.03 E +6 Colloids/mL)
Pkcalc = 0.236
Pkobs = 0.030
C/Co
0.03
0.02
0.01
0.00
0
50
100
150
200
250
Pore Volumes
Goethite,spike,CXXIV
2563-0

Figure 5


Fulvic Acid Transport Through a Crushed-Rock Column
AIS Dolomite (250-500 μm)
Fulvic Acid
H-17 brine, 0.113 mL/min
5-mL spike (1500 ppb)
Pkcalc = 0.57
Pkobs = 0.32
Injection started at 0.8 pore volumes
C/Co
0.4
0.3
0.2
0.1
0.0
0
2
5
8
10
Pore Volumes
HS.Fuv.CXII(b)
2557-0