CONF-901116--1

NATURAL-ANALOG STUDIES FOR PARTIAL VALIDATION OF CONCEPTUAL MODELS OF RADIONUCLIDE RETARDATION AT THE WIPP

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SAND--90-1275C
DE91 001339

ABSTRACT

Transport by groundwater within the Culebra Dolomite, an aquifer above the Waste Isolation Pilot Plant (WIPP), is the most probable mechanism for long-term release of radionuclides to the accessible environment. Radionuclides could be retarded by sorption if the groundwater is exposed to sufficient amounts of fracture-lining clays. In this natural-analog study, distributions of U and trace metals have been examined to constrain the strength of clay/solute interactions within the Culebra.

Uranium solid/liquid distribution ratios, calculated from U concentrations of groundwaters and consanguineous fracture-filling clays, range from ~80 to 800 mℓ/g and imply retardation factors of 60 to 500 using a fracture-flow model. Retardation factors inferred from uranium-series disequilibria and ^{14}C ages in Culebra groundwaters alone are much lower (~10), implying that clays may contain a significant unreactive component of U. Such a possibility is corroborated by Rb/Sr ages; these imply long-term stability of the clays, with resetting occurring more than 250 Ma ago. Factor analysis and mass-balance calculations suggest, however, that Mg-rich clays are dissolving in Pleistocene-age groundwaters and/or are converting to Na-rich smectites, and that B and Li are taken up from the water by the clays. Apparently, the solution chemistry reflects gradual equilibration of clays with groundwater, but thus far the bulk of the clays remain structurally intact. Measurements of the distribution of U in the Culebra will be more meaningful if the inert and exchangeable components of the U content of the clays can be quantified.

INTRODUCTION

Transport by groundwater within the Culebra Dolomite, the most significant of the two aquifers overlying the Waste Isolation Pilot Plant (WIPP), is the most probable mechanism for the long-term release of radionuclides to the accessible environment. Thorough discussions of WIPP-site stratigraphy are available elsewhere;[1,2] briefly, the WIPP is located in southeastern New Mexico, U.S.A., in strata of the Permian Delaware Basin. It lies within the Salado Formation, a thick bedded evaporite sequence of halite and minor K-rich salts. Overlying the Salado is the Rustler Formation, comprised predominantly of anhydrite and, in the eastern WIPP site, halite. The Rustler Formation contains two hydrologically important horizons, the Culebra Dolomite and the stratigraphically higher Magenta Dolomite. The transmissivity of the Magenta is two to three orders of magnitude lower than that of the Culebra throughout the WIPP region.

Within the Culebra, sorption of radionuclides by fracture-lining clays is a potentially important process by which their transport could be significantly retarded, but groundwater must be exposed to sufficient amounts of such clays. Additionally, the waste elements must bind very strongly to the clays in order to compete effectively for sorption sites with major ions which are present at high concentrations (> 0.1 *M*). The distributions of U and trace metals between groundwaters and minerals along modern flow paths are being evaluated as natural analogs for interactions between radionuclides potentially released from the WIPP and clays that line the probable flow paths.

CLAY MINERALOGY OF THE CULEBRA DOLOMITE

Within the Culebra Dolomite, clay minerals comprise 1 to 10 wt. % of the bulk rock and 1 to 43

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wt. % of the fracture lining material.[3] The most commonly occurring clay minerals are: corrensite (an ordered saponite-chlorite mixed-layer clay) > illite > chlorite > serpentine. Within the matrix, the clays occur as disseminated particles and as thin layers which appear to represent primary horizontal bedding planes. Horizontal fractures, formed in response to stresses induced by dissolution of underlying salt beds, occur along zones where clay is concentrated.

Corrensite commonly constitutes ~25 wt. % of the material scraped from the surfaces of horizontal fractures in hydrologically transmissive intervals of core and from similar fractures exposed in WIPP shafts. Based on studies of morphology and composition of clay crystals from the Culebra,[3] it appears that corrensite formed from alteration of detrital dioctahedral smectite in Mg-rich pore fluids during deposition of the Rustler Formation. The transformation involved replacement of Si by Al in tetrahedral sites, replacement of Al by Mg in octahedral sites, and deposition of a brucite ($Mg(OH)_2$) layer in every other interlayer space of the clay. The Si released by Si-Al exchange is most likely locally redeposited as amorphous silica. Such transformations create a reactive Mg-Si-rich layer, similar to that proposed for neoformed Mg-rich clays in Lake Abert sediments.[4]

URANIUM PARTITIONING BETWEEN GROUNDWATER AND CLAY

Laboratory studies indicate that U is strongly sorbed by corrensite from simple electrolytes (0.1 *M* $NaClO_4$) in the pH range 6–8.[5,6] The presence of Mg and Ca at concentrations typical of Culebra waters (10^{-4} to 10^{-2} mol/kg H_2O) has little effect on sorption. Complexing ligands (CO_3^{2-} and SO_4^{2-}) that sequester U and/or block surface complexation sites may lower the amount of sorption in Culebra waters.[6] In addition, the high concentrations of Na (0.14 to 2.5 mol/kg H_2O) typical of Culebra waters may also inhibit U uptake by clays. The available data, however, are insufficient to predict the degree of sorption in WIPP groundwaters. Direct measurement of the *in situ* partitioning of U between Culebra waters and fracture-lining clays is needed to complement ongoing laboratory studies.

A set of U distribution ratios ($\equiv [U]_{solid}/[U]_{liquid}$) has been assembled from concentration data on WIPP waters (determined by ID-TIMS on 200 mℓ aliquots[7]) and new INAA data on presumably consanguineous core material previously separated according to size for Rb/Sr work[8]. From the Magenta aquifer, a value near 200 mℓ/g was obtained from well WIPP-25 on dolomite; values on clays from WIPP-27 in the Culebra aquifer fall in the range 240 to 830 mℓ/g, whereas those from WIPP-29 are near 100 mℓ/g. The Magenta value was determined using massive dolomite which, because of the absence of fractures, seems unlikely to be a major factor affecting U mobility in Magenta groundwater. At WIPP-27 and WIPP-29, groundwater may contain a significant portion of admixed water from potash mining activities,[9,10] and so may differ somewhat from fluids typical of the WIPP site proper.

The distribution ratios from Culebra clays were determined using separated clay fractions of various sizes, all ≤2.5 μm, from clay-rich horizons within the Culebra aquifer. There is no correlation between distribution ratio and particle size, implying little re-distribution of U during the diagenesis of detrital clays or the more recent alteration of corrensite. The clays may play a more vital role in sorption/desorption than massive dolomite, but Rb/Sr studies[8] indicate that these clays have maintained structural integrity since at least the early Mesozoic; if such clays contain significant structural relative to exchangeable U, the measured coefficients will be erroneously large. In opposition, however, the cleaning process for Rb/Sr analyses remove evaporitic salts from the clays used may have stripped away a significant portion of adsorbed U, causing underestimation of the distribution ratio.

The retardation factor (R_F) is the rate of transport of a solute divided by the rate of groundwater flow, and may be estimated by appropriately modeling groundwater movement, and by equating the distribution ratio with the distribution coefficient (K_D), implicitly requiring the observed partitioning of U to be the consequence of sorption/desorption equilibrium rather than dissolution/precipitation effects. Because groundwater movement in the Culebra is predominately through clay-lined horizontal fractures, a fracture-flow model should be used for the clays:[11]

$$R_F = 1 + \rho_c X_c K_D / (\delta_c / \delta), \qquad \{1\}$$

where ρ_c is the density of clay (~2.5 g/cm^3), X_c is the proportion of clay in the fracture lining (~25%; the remaining constituents, primarily gypsum and anhydrite, are assumed inert here), and δ_c/δ is the ratio of the thickness of the fracture lining to the fracture aperture (here assumed to be ~1). With these values, fracture-flow R_F's for clays are 37% smaller than the associated K_D's. The more reliable data of this work are consistent with R_F values between 60 and 500. These values are minima with respect to U transport in fractures, however, because 75% of the fracture-lining material is assumed to be non-sorbing, with $K_D \approx 0$.

Groundwater will interact with dolomite only after penetrating the fracture linings. Because Rustler dolomite is massive and homogeneous, a model of uniform flow in a porous medium is appropriate. In such a model, R_F is related to K_D by:[12]

$$R_F = 1 + K_D\,(\rho_s\,(1-\phi)/\phi), \qquad \{2\}$$

where ρ_s is the density of the solid and ϕ is its porosity (~2.9 g/cm^3 and ~0.15 respectively for Magenta dolomite). Our data are consistent with a uniform-flow R_F near 1,000 for bulk dolomite, but this may be a maximum because some clay is disseminated throughout the rock and may contribute substantially to the distribution ratio for dolomite.

In principle, R_F's for U in the Rustler groundwater system may also be calculated isotopically from uranium-series disequilibrium (USD) data in conjunction with an estimate of the age of the water. A radiocarbon-dating model which accounts for water/carbonate interactions[13] provides the best available estimate of the time required for water to travel from its nearest assumed recharge areas (outcrops) to its present position near the WIPP site; the derived travel-times are 12 ka for well H-6 and 16 ka for well H-4.[10] The times required to generate the observed $^{234}U/^{238}U$ activity ratios (AR's) in the same groundwaters traveling along the same flow paths may be calculated if a mechanistic model which produces high AR's in groundwaters under *reducing* conditions[14,15] is used. Best-estimate assumptions include: 1) bulk density of dolomite = 2.8 to 2.9 g/cm^3; 2) average rock [U] = 0.9 μg/g; 3) observed high AR values developed in water in the presence of rock having [U] $\approx$ 1 μg/g and AR $\approx$ 1 to 2; and 4) initial [U] in solution was 0.024 ng/g and the initial AR was ~2 (the lowest observed values associated with the Rustler, as represented by the Rustler/Salado contact water from WIPP-30). Using these values, the time required for AR's to evolve to values observed near the site are 70 ka for H-6 and 160 ka for H-4.[7] The resulting ratios of U-to-groundwater travel-times are R_F's, and are 5.8 for H-6 and 10 for H-4.

Results of this and other investigations of U mobility in waters typical of the WIPP site are summarized in Table I. Procedures used by other investigators to derive R_F's include: 1) measurement of U-series disequilibria (USD) in waters in which the U-series decay chain has (hopefully) achieved secular equilibrium,[12] and 2) direct measurement in the laboratory using tracers in batch experiments equilibrated over comparatively short periods of time (we have calculated R_F's from such batch K_D's using the uniform-flow model, eqn. {2} above). For dolomite, values obtained from laboratory investigations,[16] and our values derived from direct measurements on Magenta groundwaters and dolomite, are all consistent with uniform-flow R_F's $\approx$ 1,000. Our fracture-flow clay R_F's are significantly lower, in the vicinity of 200, due to the nature of the flow model. The fracture-flow model applied to clays most closely approximates groundwater movement in the Culebra, but the resulting R_F's are still significantly larger than those determined *in situ* by sudy of USD in Rustler groundwaters[17] (which were probably not at secular equilibrium) and our values modeled on AR's and ^{14}C travel-times; these latter are consistent with R_F's $\leq$ 50. Values inferred from measurements of USD in ancient brines[18,19] of the Palo Duro Basin are included for comparison, although such brines may be poor analogs for the comparatively young groundwaters of moderate salinity at the WIPP site.

This dichotomy between R_F's inferred from directly determined distribution ratios and from observations of USD systematics may be rationalized in two ways: 1) Our direct measurements may have included a substantial unreactive U component as noted previously, producing urealistically high distribution ratios and R_F's; and 2) R_F's derived from AR's and independent travel-times require untestable assumptions about the nature of the mechanistic mobility model, e.g., the redox state of the

Table I. Retardation factors (R_F's) for U and Th in brines and rocks similar to those of the WIPP site. Methods: *dir. deter.* = direct determination, i.e., concentrations in aqueous and solid phases were measured directly; *USD* = uranium-series disequilibrium, which yields R_F's in static systems at secular equilibrium[12] or in flowing systems with independent travel-time information[14,15] (e.g., ^{14}C); *laboratory* = determined by measuring partitioning of U tracer between solid and aqueous phases using core samples and synthetic waters. Where available, statistics are reported at the 1σ level. See text for relation between R_F's and distribution coefficients. *Mag* = Magenta horizon, *Cul* = Culebra horizon at the WIPP site.

Well	Method	R_F	Comments & Primary Data
***U Values** (this work)*			
WIPP-25	*dir. deter.*	$1200^{\dagger}$	*Mag*, dolomite: $[U]_{aq}$=10.46 ppb, $[U]_{dolo}$=1.87 ppm, n=1.
WIPP-27	*dir. deter.*	$320 \pm 105^{\ddagger}$	*Cul*, clays: $[U]_{aq}$=8.69 ppb, $[U]_{clay}$=4.4±1.5 ppm, n=9.
WIPP-29	*dir. deter.*	53 to $68^{\ddagger}$	*Cul*, clays: $[U]_{aq}$=41.4 ppb, $[U]_{clay}$=3.8±0.6 ppm, n=3.
H-4	*USD&^{14}C*	5.8	See text. Modeled as U^{IV} (reducing conditions).
H-6	*USD&^{14}C*	10	See text. Modeled as U^{IV} (reducing conditions).
***U Values** (from the literature)*			
AEC-8	*laboratory*	135 to $610^{\dagger}$	*Mag*, dolomite.[16] U^{VI} tracer used. $[U]_{tracer}$=1.4 ppb, n=4.
H-4B, H-5B	*laboratory*	120 to $1870^{\dagger}$	*Cul*, dolomite.[16] U^{VI} tracer used. $[U]_{tracer}$=1.4 ppb, n=4.
H-4, H-6, & DOE-1	*USD*	29, 95, & 36	Young(?) groundwaters.[17] R_F for ^{222}Rn assumed = 1. Possible Rn loss ➡ R_F's for U are minimum estimates.
Palo Duro Basin	*USD*	200 to 30,000	Ancient brines.[18,19] R_F for ^{222}Rn assumed = 1. Mostly reducing conditions (U^{IV}).

†From K_D, uniform porous-medium model (eqn. {2}). ‡From K_D, fracture-flow model (eqn. {1}).

groundwater with respect to U. The available data are insufficient to assess which values will most accurately predict U behavior in the Rustler groundwater system; work in progress includes laboratory experiments on clay-water systems and direct measurements on fracture-lining clays and related waters.

EVIDENCE OF CLAY-WATER INTERACTIONS WITHIN THE CULEBRA DOLOMITE

Rb/Sr ages on corrensite-dominated samples imply long-term stability of the clay-mineral structure, with resetting occurring no more recently than ~250 Ma ago. In a sequence of sizes ranging from ≤0.3 μm to ≤2.5 μm, the coarser clays gave older ages, interpreted as reflecting increasing proportions of detrital (illitic) versus authigenic (corrensitic) clays[8]. All sizes gave ages in the range 260 to 290 Ma (Early to Middle Permian), somewhat older than the Late Permian age of the Rustler Formation. Importantly, the Rb/Sr data cannot reflect ion-exchange processes occurring on clay surfaces presently in contact with Culebra waters, because cleaning during sample preparation necessarily removed any sorbed ions — the clays were leached overnight in an acetic acid-sodium acetate buffer to remove the evaporitic component of Sr contained in dolomite, anhydrite, or gypsum.

Evidence that solute-clay reactions have modified the groundwater chemistry of the Culebra Dolomite comes from studies of solute correlations and mass balances of major ions along modern flow paths. Because the Culebra Dolomite lies within an evaporite sequence, it is likely that the major control on the concentrations of solutes in the Culebra is the dissolution of evaporite salts in the Rustler Formation.[20] Indeed, the concentrations of Mg, Ca, Sr, SO_4, K, Li, Br, and B are strongly correlated with Na and Cl concentrations. R-mode principal component analysis[21] of compositional data from 21 samples from the vicinity of the WIPP site[20] showed that a single principal component (the salinity factor, shown in Fig. 1) accounts for most of the variance of these elements. A significant portion of the variance of elements that may be involved in clay-water interactions (Si, Mg, pH, Li, and B), however, is not related to this principal component. Principal component analysis of the variance and covariance that is not included in the salinity factor yielded two sets of interelement correlations (Fig. 2) that are consistent with several clay-water reactions.

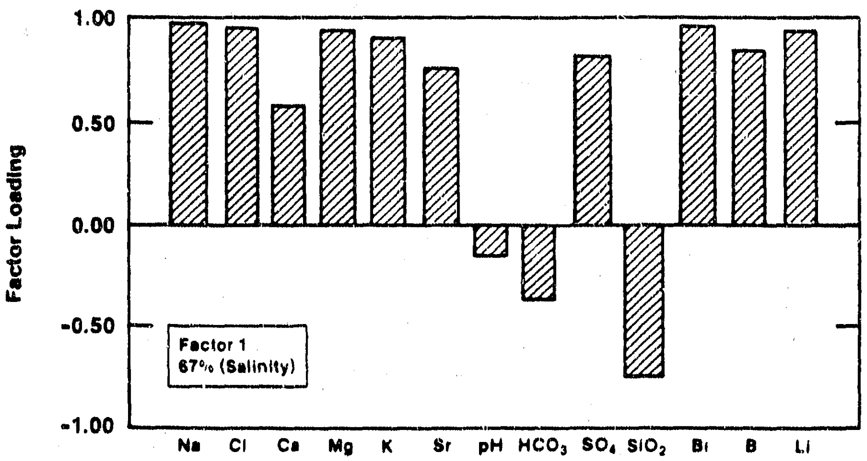

Figure 1. R-mode loadings for first prinicpal component (the *salinity factor*) describing interelement correlations in 21 groundwater samples from the Culebra Dolomite near the WIPP site. This factor accounts for 67% of the total variation in the data.[20]

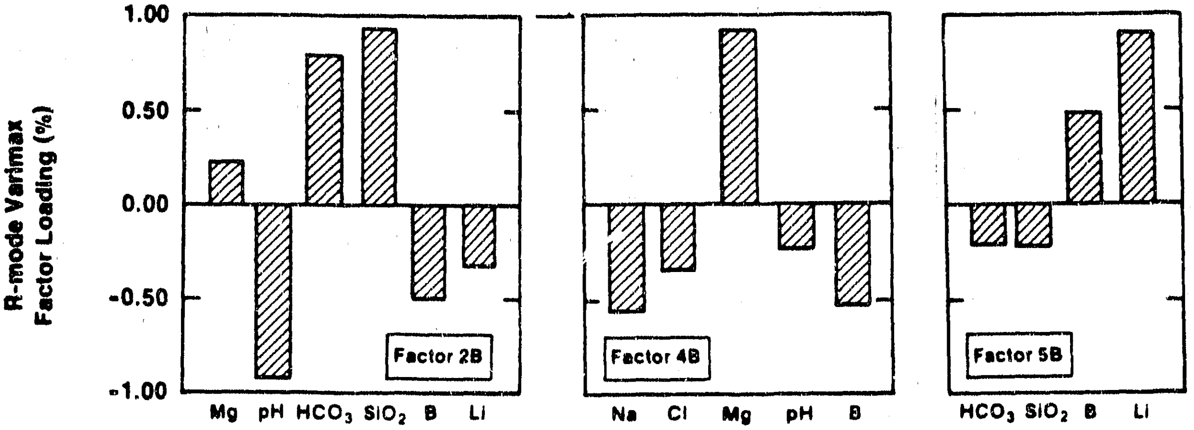

Figure 2. Varimax R mode factor loadings of key elements for factors 2B, 4B, and 5B obtained from partial-correlation matrix with respect to total dissolved solids.[20] Factor loadings are plotted only for elements that have important geochemical significance or strongly influence the orientation of the factors (loading > 0.23).

The principal components shown in Fig. 2 contain a Mg-Si association that is negatively correlated with a Cl-Na-B-Li association. This pattern may be due to a combination of processes that include: 1) dissolution of the neoformed Mg-Si rich layer in corrensite (Mg-Si correlation in Factor 2B) or Mg-Na ion exchange (negative Mg-Na correlation in Factor 4B); and 2) uptake of Li and B by clays (Si-Li, B relations in Factors 2B, 4B, and 5B) by ion exchange or lattice substitution. The correlation of Mg and Si is consistent with the reaction:

$$MgSiX_{(am)} + 2H_2O + 2H^+ \rightarrow Mg^{2+} + H_4SiO_4, \qquad \{3\}$$

where $MgSiX_{(am)}$ is the amorphous reactive Mg-Si-rich layer in corrensite. Li and B could be taken up by the unreactive Al-Si-rich portion of corrensite. In areas of the Culebra where clay is most abundant, elevated Si and Mg concentrations and lower B and Li concentrations would occur and lead to the correlations expressed by the principal components.

Efficacy of the exchange processes in saline environments is substantiated by previous laboratory and field studies,[20] and a similarly reactive amorphous Mg-Si-rich layer in authigenic mixed-layer clays has been described in Salt Lake sediments.[22] The uptake of Li by clays is also indicated by decreasing Li/Cl ratios with increasing Cl concentrations in Culebra waters. Similar trends have been observed in other Li-rich brines in the western United States;[23,24] Li influx to saline lakes is removed by lake sediments and/or fixed by clays in the vadose zone.

Mass-balance calculations for major ions along modern flow paths also suggest that ion-exchange (Na-Mg or Na-Ca exchange) has occurred.[25] The mass transfers required to account for changes in the

Table II. Mass transfer reactions along flow paths within the Culebra Dolomite. A) Well-water compositions. B) Mass transfers: dissolution of a solid phase or gas is indicated by positive values; release of Mg (or Ca) by clay in exchange for Na from solution is indicated by negative values.

A. Well-Water Compositions (mmol/kg-H_2O)

Component	H-3	H-11	H-17
C	0.73	0.76	0.72
S	49.89	68.59	78.59
Ca	36.16	39.02	42.25
Mg	33.66	55.78	78.40
Na	766.10	1815.07	2487.25
K	11.66	21.35	32.50
Cl	816.57	1887.45	2602.18

B. Mass Transfers (mmol/kg-H_2O)

Phase	H-3→H-11	H-11→H-17	
		A	B
Halite	1070.89	714.73	702.04
Gypsum/Anhydrite	−0.68	−12.30	−3.84
Calcite	−0.16	0.15	0.15
Ca-Na Exchange	6.31	−4.23	
Polyhalite	4.85	5.57	3.46
Carnallite			4.23
Mg-Na exchange	−17.27	−17.04	−14.93
CO_2 gas	−0.13	−0.19	−0.19

chemical compositions of groundwaters were inferred from analyses of waters sampled in a series of wells along postulated modern flow paths (Fig. 3). The mass transfers were modeled by writing balanced chemical reactions of the form:

$$\text{Initial Solution} + \text{Reactant Phases} \longrightarrow \text{Final Solution} + \text{Product Phases} \qquad \{4\}$$

Combinations of mineral-gas-water reactions and mixing of different groundwaters which are consistent with mass transfers between pairs of wells along the flow path were calculated using the computer code BALANCE.[26] The candidate phases included halite, gypsum, dolomite, calcite, magnesite, sylvite, polyhalite ($K_2Ca_2Mg(SO_4)_4 \cdot 2H_2O$), carnallite ($KMgCl_3 \cdot 6H_2O$), Ca-Na ion exchange, K-Na ion exchange, Mg-Na ion exchange, and CO_2 gas. A large number of possible reaction sets were consistent with the observed mass transfers, but only those reaction sets which included significant amounts of ion exchange were thermodynamically feasible; others were energetically unfavorable or required dissolution of large quantities of evaporite minerals not observed in the Culebra Dolomite or adjacent strata.[25]

Table II provides examples of mass-balance reactions involving ion exchange. In the mass transfer calculations for flow from H-3 to H-11 and from H-11 to H-17, Mg-Na exchange (release of Mg and uptake of Na) is required in several of the possible models. Such exchange may correspond to the alteration of corrensite to Na smectite by ion exchange and/or dissolution of the Mg-rich interlayer. This alteration would result in the release of Mg and the uptake of Na from solution. Ca-Na ion exchange is required in the mass balance models (H-11/H-17, model A) if dissolution of carnallite is excluded from the calculations.

The antithetical implications of Rb/Sr geochronology vs. factor analysis and mass-balance calculations can be integrated, however. Solution chemistry may reflect gradual equilibration of corrensite with relatively Mg-poor, Na-, B-, and Li-rich groundwater, but because only a tiny surficial portion of the clay has been altered so far, Rb/Sr systematics of the clays remain essentially undisturbed since the time of formation of corrensite.

CONCLUSION — ESTIMATING URANIUM RETARDATION

Uranium distribution ratios calculated from direct measurements of U concentration in groundwater and aquifer materials (clay or dolomite) may be useful indicators of U retardation by sorption, but are suspect unless only exchangeable U in the reactive portion of the solid phase is considered, and unless the observed aqueous U concentrations are the result of steady-state sorption/desorption equilibrium rather than dissolution or precipitation effects. Such considerations may be at the root of differences in U mobility inferred *in situ* from study of U-series disequilibria in comparison attempts to directly K_D's and then infer R_F's. Because the fracture-lining clays appear to consist of both reactive and inert components, quantification of these components of U content is

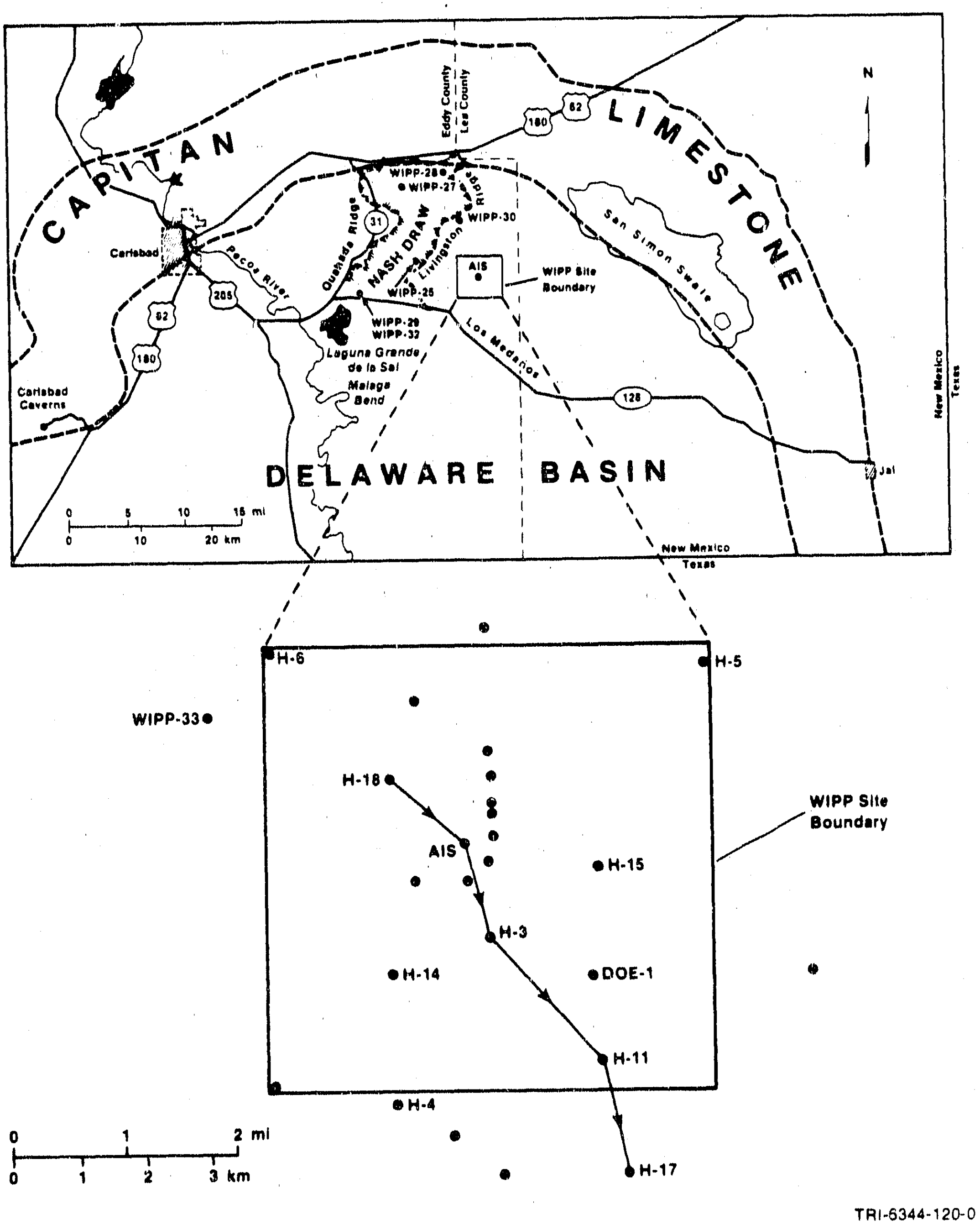

Figure 3. Mass transfer paths assumed in BALANCE calculations. ● = WIPP well locations; AIS = air intake shaft; arrows show mass transfer paths.

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crucial; such data will be obtained from selective-leaching studies combined with detailed mineralogical examination.

Principal components analysis shows that, in spite of the long-term stability of clays indicated by Rb/Sr geochronology, concentrations of major and minor solutes are consistent with the hypothesis that clays lining the water-bearing fractures are accessible to and have altered the chemistry of modern groundwaters in the Culebra Dolomite. Although the *salinity factor* accounts for the majority of the observed variance, a Mg-Si association which negatively correlates with a Cl-Na-B-Li association is most readily explained by interaction with corrensite, the dominant clay. Mass-balance calculations give similar results, with reasonable scenarios requiring Mg-Na and possibly Ca-Na ion exchange as well as dissolution or precipitation of solid phases. Thus, any investigation or model of U transport must take into account the sorptive and reactive properties of the clays as well as those of dolomite, anhydrite, gypsum and other evaporite minerals.

This research has been sponsored by the U.S. Dept. of Energy under Contract No. DE-AC04-76-DP00789.

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