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ABSTRACT

Detailed mineralogical studies of the matrix and fracture-fill materials of a large number of samples from the Rustler Formation have been carried out using x-ray diffraction, high-resolution transmission electron microscopy, electron microprobe analysis, x-ray fluorescence, and atomic absorption spectrophotometry. These analyses indicate the presence of four clay minerals: interstratified chlorite/saponite, illite, chlorite, and serpentine. Corrensite (regularly stratified chlorite/saponite) is the dominant clay mineral in samples from the Culebra dolomite and two shale layers of the lower unnamed member of the Rustler Formation. Within other layers of the Rustler Formation, disordered mixed chlorite/saponite is usually the most abundant clay mineral. Studies of the morphology and composition of clay crystallites suggest that the corrensite was formed by the alteration of detrital dioctahedral smectite in magnesium-rich pore fluids during early diagenesis of the Rustler Formation. This study provides initial estimates of the abundance and nature of the clay minerals in the Culebra dolomite in the vicinity of the Waste Isolation Pilot Plant.
ACKNOWLEDGMENTS

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1.0 INTRODUCTION

Clay minerals created by weathering processes can be carried to new locations by wind or water. Once introduced into a different environment, clays can be transformed (undergo diagenesis) into new clay phases that are more stable in the new environment than the original phases. A striking example of this process is the suite of clay minerals found in marine evaporite deposits. Bodine (1978, 1985a, 1985b) studied the diagenesis of clay minerals in marine evaporite deposits; he found that the original dioctahedral clay mineral assemblage was largely transformed into trioctahedral phases.

This report presents a detailed study of the clay mineralogy of the evaporite deposits in the Rustler Formation of the Delaware Basin. The data include semiquantitative analysis of individual clay mineral components, bulk chemical compositions, chemical compositions and structural formulas of the individual phases, and high-resolution transmission electron microscope images of each phase. Sets of chemical reactions are postulated for the processes by which each clay mineral in the present clay assemblage may have formed.
2.0 GEOLOGICAL FRAMEWORK

The Delaware Basin, occupying an area of approximately 40,000 km² in west Texas and southeastern New Mexico, was formed in early to mid-Paleozoic time as a result of orogenesis. Subsidence allowed the deposition of a series of deep marine clastics in the early to mid-Permian. A ring of limestone reefs surrounding the basin was formed in the late Permian (Ochoan) and partially isolated the basin from adjacent shelf areas. As access to the open sea was restricted, a thick sequence of evaporites was deposited including the Castile and Salado Formations. Near the end of Salado time, the Pedernal land mass, located north of the basin, was uplifted. During the subsequent deposition of the Rustler Formation, clastics from the Pedernal and other source areas washed into the basin, along with both marine and fresh water. The Rustler Formation is approximately 100 m thick in the Carlsbad area; it includes mudstones, many of which are dissolution residues, argillaceous halite beds, anhydrite, gypsum, and dolomite beds. The Dewey Lake Red Beds Formation overlies the Rustler Formation and consists of silt, sand, and mudstones with gypsum cement. It was deposited as the sea retreated completely from the Delaware Basin.

A geologic column showing the major lithologic units of the Rustler Formation is shown in Figure 1. More detailed discussions of the geology of the Delaware Basin can be found in Powers et al. (1978) and Siegel and Lambert (1991).
Figure 1. Lithologic log of Rustler Formation in the WIPP-19 core, modified from Ferrall and Gibbons (1980).
3.0 SAMPLE DESCRIPTIONS

Rock samples used in this study were taken from various drill cores in the vicinity of the Waste Isolation Pilot Plant (WIPP) repository, near Carlsbad, New Mexico, from vertical shafts, and from drill cores bored from within the repository itself. The specific locations, depths, and major mineral components of the samples are shown in Table 1 and Figure 2.
<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Location</th>
<th>Local Depth</th>
<th>Formation or Member</th>
<th>Major Mineral Components</th>
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<td>FH-239</td>
<td>WIPP Repos.</td>
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<td>Dolomite</td>
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<tr>
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<td>Culebra</td>
<td>Dolomite</td>
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<td>Dolomite/Clay/Gypsum</td>
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<td>Local Depth</td>
<td>Formation or Member</td>
<td>Major Mineral Components</td>
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<td>-------------</td>
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<td>Calcite</td>
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<td>Dolomite/Clay</td>
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<td>AIS-17</td>
<td>AIS</td>
<td>711'</td>
<td>LMember</td>
<td>Clay/Gypsum/Quartz</td>
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<td>DLRB-2</td>
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<td>583'</td>
<td>DLRB⁴</td>
<td>Clay/Gypsum/Quartz</td>
</tr>
</tbody>
</table>

¹ LMember = lower unnamed member of the Rustler Formation.
² 49er = Forty-niner member of the Rustler Formation.
³ AIS = Air Intake Shaft.
⁴ DLRB = Dewey Lake Red Beds.
Figure 2. Locations of wells and shafts from which clay samples were taken.
4.0 ANALYTICAL TECHNIQUES

4.1 X-Ray Powder Diffraction Analysis

Samples were analyzed with a Scintag PAD-V x-ray diffractometer using CuK radiation. Whole rock samples were analyzed for major mineral components. Samples containing halite were dissolved in deionized water to remove the halite; sulfate and carbonate-containing samples were treated with disodium-ethylenediaminetetraacetic acid (EDTA) to remove these salts (Bodine and Fernalld, 1973). The <2 \( \mu \)m fraction was separated by standard centrifugation techniques, and oriented clay aggregates were prepared by the filter transfer method (Drever, 1973; Moore and Reynolds, 1989). The oriented mounts were analyzed from 2\(^\circ\) to 30\(^\circ\) 2\(\theta\). Four analyses of each sample were made: untreated air-dried mounts; samples saturated with ethylene glycol for 24 or more hours at 60°C; samples heated to 400°C for 1 hour; and samples heated to 550°C for 1 hour. Seward et al. (1991, Figures VII-1 to VII-3) illustrate the effects of these treatments on the x-ray diffractograms in more detail.

Clay minerals were identified by standard techniques (Brindley and Brown, 1980; Moore and Reynolds, 1989). The ratio of chlorite to saponite in disordered interstratified chlorite/saponite was determined by the methods of Reynolds (1980). Semiquantitative analysis of the individual clay minerals was performed using theoretical oriented (glycolated) diffractograms; individual clay minerals present in the samples were calculated using the NEWMOD computer program (Reynolds, 1985). These were compared with the diffraction patterns of the glycolated mounts for the samples in this study. The following peaks were selected for semiquantitative analysis: mixed-layer chlorite/saponite, 3.46 Å; chlorite, 4.73 Å; illite 5.0 Å; serpentine, 3.51 Å; talc, 4.7 Å. The areas under the peaks in the sample diffractogram were divided by the corresponding areas of the peaks in the calculated diffractograms for each mineral; these ratios were normalized to 100% to obtain the mode for each clay mineral.

4.2 Chemical Analysis

Oxide analyses of the <2 \( \mu \)m fraction of the crushed rock were made by x-ray fluorescence spectroscopic analysis (XRF) and atomic absorption spectroscopy (AA).
4.3 Chemical Microanalysis

Electron microprobe analyses (EMP) of bulk clay compositions from polished thin sections were obtained on a JEOL 733 Superprobe using wavelength dispersive spectrometer analysis (WDS). Compositions for the individual clay minerals were obtained on a JEOL 2000-FX transmission electron microscope (TEM) using a Tracor-Northern TN5500 energy dispersive spectrometer (EDS) containing a thin beryllium window. Analyses were carried out using a Gatan low-background cooling stage, cooled with liquid nitrogen to -150°C to minimize elemental volatization.

4.4 High-Resolution Imaging

High-resolution images of the individual clay minerals were obtained on the JEOL 2000-FX TEM operating at 200 kV. Clay mineral separates samples were imbedded in Spurr's epoxy and sectioned to less than 900Å using a diamond knife on a Reichert-Jung ultramicrotome. Selected samples were intercalated with tetradecyl ammonium chloride (Vali and Koester, 1986) prior to being embedded in epoxy, to expand the smectite layers to approximately 18 Å. The tetradecyl ammonium chloride was produced from tetradecylamine was treated with hydrochloric acid. This organic compound was chosen instead of laurylamine hydrochloride because the latter expands these layers only to about 15 Å. Distinguishing between chlorite layers and smectite layers is difficult with the smaller separation, and under these conditions corrensite can easily appear to be chlorite.

4.5 Cation Exchange Capacity

The cation exchange capacity (CEC) of selected samples from the Culebra Dolomite Member of the Rustler Formation was measured by the sodium acetate method (Chapman, 1965). Samples were saturated with sodium acetate, washed in ethyl alcohol, and resaturated with ammonium acetate. The sodium content of the supernatant was measured by atomic absorption spectrophotometry, and the CEC was calculated as described in Chapman (1965).
5.0 RESULTS

5.1 Whole-Rock and Clay Mineralogy

The major mineral components of the whole-rock samples are listed in Table 1. Rustler Formation samples from the WIPP-19 core were quite varied; all had significant amounts of clay and quartz, while the primary evaporite minerals include gypsum, anhydrite, dolomite, calcite, magnesite, and halite. Eighteen samples were taken from the Culebra Dolomite from other wells and shafts; dolomite was the principal evaporite mineral in all but one of these. The exception, CS-15, contains calcite of apparently secondary origin, and is interpreted to be a product of dedolomitization.

Three samples that are discussed in more detail in later sections of this report were taken from shale layers at or near the contact between the Culebra Dolomite and the unnamed lower member. AIS-15 was taken from a black shale which immediately underlies the Culebra Dolomite proper, and AIS-17 was obtained from a red shale which underlies the black shale. H6B-3 is also from a shale layer in the lower unnamed member of the Rustler. All of these shales are interpreted to be dissolution residues of argillaceous halite.

Clay minerals identified in these samples include serpentine, illite, mixed-layer chlorite/saponite, and chlorite. The mixed-layer chlorite/saponites included a regularly stratified form (corrensite) and a disordered form (disordered chlorite/saponite). Table 2 shows the estimated abundances of these minerals and the percentage of chlorite layers in the mixed-layer chlorite/saponite. Corrensite is the dominant clay mineral in most of the Culebra samples and in the AIS-15, AIS-17, and H6B-3 samples. Most of these samples also contain significant amounts of illite and minor quantities of serpentine and chlorite.

Figure 3 shows x-ray diffractograms for the <2 \( \mu \text{m} \) fraction of glycolated clay separates from samples taken from the Air Intake Shaft. Diagnostic peaks for corrensite, illite, serpentine, and chlorite are also indicated in Figure 3. Corrensite comprises 94% of the clay minerals in the AIS-15 sample. The AIS-3 sample (Figure 3b) was taken from a clay-rich specimen of Culebra dolomite. Corrensite comprises 49% of the clay mineralogy of this sample and is the major clay mineral.

Most of the samples from other units of the Rustler Formation have disordered mixed-layer chlorite/saponite as the major clay mineral. Corrensite is present in only three samples taken from units other than the Culebra and the shales just below the Culebra. In the majority of the clay separates, illite and serpentine are present in roughly equal proportion, and chlorite is present in smaller quantities.
Table 2. Semiquantitative Clay Mineral Abundances (wt.%)

<table>
<thead>
<tr>
<th>Sample</th>
<th>Illite</th>
<th>Serpentine</th>
<th>Chlorite</th>
<th>Mixed-Layer</th>
<th>Chlorite/Saponite</th>
<th>Percent Chlorite in C/S</th>
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Table 2. Semiquantitative Clay Mineral Abundances (wt.%) (continued)

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Figure 3. X-ray diffractograms for glycolated <2 μm fractions of clay separates from Sample AIS-15 (a) and Sample AIS-3 (b).
5.2 Clay Mineral Crystallite Morphology

High-resolution transmission electron microscope (TEM) images of regularly stratified mixed-layer chlorite/saponite (corrensite) in intercalated samples are shown in Figures 4 and 5. Figure 4 is a low-magnification image of clay crystallites from the AIS-15 sample containing illite and corrensite. Figure 5 is a high-resolution TEM image from the AIS-17 sample of an expanded corrensite with the chlorite and saponite layers resolved.

Crystallites are often restricted to no more than two or three repeats, considered to be "fundamental" clay particles, but can also vary up to 10 or more repeats (about 300 Å). EDS spectra for these crystallites show the presence of Mg, Al, Si, and Fe. The amount of Fe is variable; there appear to be two distinct families of compositions: one with high Fe and one with low Fe.

Corrensite morphology strongly resembles dioctahedral smectite (montmorillonite) morphology (Ahn and Peacor, 1986; Lee et al., 1985). Individual crystallites have the same wavy appearance as smectite crystallites, and the crystallites form a subparallel network as seen in Figure 6.

In general, corrensite from WIPP samples was difficult to image using high-resolution TEM techniques because of rapid beam damage and poor diffraction contrast. Satisfactory images were obtained despite these problems, although interpretation of the images was not straightforward. Expanded 1:1 corrensite should show a regular alternation of 14 Å (chlorite) and 18 Å (expanded smectite) layers. However, the images show that the corrensite usually consists of two layers with a repeat of 14 Å each, alternating with a broader fringe with a basal spacing that varies between 24 Å and 36 Å (e.g., Figure 5). There are considerable ambiguities associated with interpreting high-resolution images of mixed-layer silicates, which have been documented in detail by Guthrie and Veblen (1990) based on calculated lattice images. To avoid some of these problems, periodicities were interpreted only when images were recorded at several focus values, including Scherzer defocus, enabling assessment of the effects of contrast reversal, etc., on the image. This work interprets the corrensite images in Figure 5 as two 14 Å layers corresponding to two chlorite layers rather than the single layer which is usual in a corrensite, with the 24-36 Å repeats representing two expanded smectite layers. Thus, the corrensite in this and other samples appears to be a 2:2 chlorite/smectite, rather than a regular 1:1 structure, and might be more appropriately termed "pseudo-corrensite." In many images, it is clear that the width of the expanded smectite layer varies
Figure 4. Low-magnification image of clay crystallites containing illite (I) and corrensite (chlorite/saponite, C/S) from the AIS-15 sample.
Figure 5. High-resolution TEM image of intercalated corrensite (chlorite/saponite, C/S) showing the chlorite (Chl) and saponite (S) layers resolved (AIS-17 sample).
Figure 6. Low-magnification image of corrensite crystallites in sample H6B-3.
along its length. This variation is attributed to partial expansion of the smectite layers by the tetradecyl ammonium chloride. The overall c-repeat for fully expanded 2:2 corrensite should be approximately 64 Å. In some cases, a basal spacing of this value has been found, but more commonly the repeat distance lies between 54 and 64 Å. Brigatti and Poppi (1985) found that expanded corrensites exchanged with a variety of different cations produced an expansion up to about 58 Å. In some cases, Brigatti and Poppi (1985) attribute this to differences in the compensating layer cations, but it is possible that they were observing expansion of two smectite layers rather than one, as observed in this study.

5.3 Crystal Chemistry

Compositions of clay mineral separates for selected samples, obtained by XRF, AA, and microprobe analysis are listed in Table 3. The high magnesium and aluminum content of all the bulk clay compositions reflect the presence of both dioctahedral and trioctahedral phases in the mineral assemblage.

Corrensite structural formulas (Table 4) were calculated for three samples by subtracting the relative proportion of the elements in illite that were determined by semiquantitative modal analysis (Table 2). The illite compositions was obtained on the TEM (Table 5) from a Salado sample (FH-239). It was assumed that the relatively small amounts of serpentine and chlorite in these samples have compositions similar to those of corrensite and thus did not cause significant error. The formula for sample AIS-15 is probably the most accurate because the sample contained 94% corrensite. Comparison with the structural formula for a sample from Gotra, Italy (Brigatti and Poppi, 1984; Newman, 1987), shows that the interlayer charge in the Italian sample is higher than those in the Delaware Basin corrensites. However, some of the magnesium ascribed to the octahedral layer is probably in the interlayer, so the layer charge in Table 4 may not be accurate. Although all three octahedral layers appear to be trioctahedral, the distribution of aluminum within the individual octahedral sheets is not known. It may be assumed that the brucite-like sheet belonging to the chlorite layer is more magnesian than the other two because it is a product of deposition, rather than transformation from an inherited dioctahedral sheet (as discussed in Section 6.1). The structural formula for a disordered mixed-layer chlorite/saponite was calculated in the same manner and is included in Table 4. Because this phase has roughly equal numbers of chlorite and smectite layers, the formula is similar to that of the corrensites.
Table 3. Compositional Analyses (wt.%) of Clay Size Fractions

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<th>TiO₂</th>
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XRF = X-ray fluorescence.
EMP = Electron microprobe.
AA = Atomic adsorption.
Table 4. Structural Formulas Derived from Bulk Analyses

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* Interlayer charge
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22
Individual crystallite compositions for corrensites were obtained on the TEM by EDS analysis; the structural formulas calculated from these analyses are listed in Table 5. The formula for corrensite are generally similar to those obtained from the bulk analyses. Differences between formulas obtained from the two methods may be attributed to loss of sodium and potassium during electron beam analysis and the likelihood that some of the magnesium ascribed to the octahedral layer in the EDS analysis is actually in the interlayer region.

One structural formula was calculated for chlorite (sample AIS-17). The dominant cation in the octahedral layer is magnesium; the mineral is thus a clinochlore. The aluminum content of the tetrahedral layer is fairly low in comparison with published clinochlore structural formulas (Newman, 1987).

No compositions for serpentine were obtained; consequently, no structural formulas were determined. However, the EDS spectra of serpentines showed the presence of only Si, Al, and Mg.

5.4 Cation Exchange Capacity

The cation exchange capacities of seven clay mineral separates are listed in Table 2 and plotted as a function of mixed-layer chlorite/saponite content in Figure 7. With the exception of the H10B-8 sample there appears to be a direct relationship between the two quantities. This is reasonable because the CEC of corrensite, which contains expandable layers, is much higher than that of illite, serpentine, or chlorite, which do not contain expandable layers (Weaver and Pollard, 1973). The mineral responsible for the high CEC of the H10B-8 sample has not been identified.
Figure 7. Relationship between cation exchange capacity and percentage mixed chlorite/saponite of clay separates from the Rustler Formation.
6.0 DISCUSSION

6.1 Clay Mineral Diagenesis

The geology of the Delaware Basin and the morphology and composition of the clay minerals examined in this study suggest that the clay mineral assemblage at the time of evaporite deposition included illite and dioctahedral smectite. Isotopic and morphological evidence indicates that illite was relatively stable, even in the extreme conditions of an evaporitic environment (Brookins et al., 1990). The smectite component underwent a series of compositional changes in response to the high salt concentrations in the brines.

Morphologies of saponite crystallites in Dewey Lake Red Beds samples, mixed-layer chlorite/saponite crystallites in the Rustler samples, and chlorite crystallites in sample AIS-17 show strong similarities. Further, these crystallites bear a strong resemblance to dioctahedral smectite crystallites (Lee et al., 1985; Ahn and Peacor, 1986); they have the same discontinuous, wavy, distorted, often anastomosing layers with variable interplanar spacing. These similarities suggest two things about the genesis of these clay minerals: first, that saponite, mixed-layer chlorite/saponite, and chlorite have a common detrital dioctahedral smectite (montmorillonite) precursor; second, that the transformation process conserved the morphology of the original dioctahedral smectite.

6.1.1 Dioctahedral Smectite to Saponite to Mixed Layer Chlorite/Saponite Transformation

The transformation from dioctahedral smectite to trioctahedral smectite to mixed-layer chlorite/saponite was originally postulated by Bodine (Bodine and Fernald, 1973; Bodine, 1985b). He concluded that the process occurred by dissolution of the original clay mineral species and recrystallization, rather than by a solid-state transformation (the "layer-by-layer" mechanism of Bethke and Altaner, 1986). This transformation process is driven by two main environmental factors: the burial depth and the very high concentration of magnesium in the pore fluids of the buried sediments. The latter factor is probably the more important, but the two are probably not independent. The morphological evidence suggests that the process of transformation conserved the original 2:1 layer of the dioctahedral smectite. Thus, following Hower et al. (1976), the transformation reaction from dioctahedral smectite to saponite might take the following form:
In this reaction, $\text{Al(OH)}_4^-$ and $\text{Fe(OH)}^+$ are taken to be the predominant aluminum and iron aqueous species, which is approximately valid for pH values above 7.5 (Drever, 1982). The montmorillonite composition is taken from Newman (1987) for a Clay Spur, Wyoming, sample (Earley et al., 1953), while the saponite formula is that of a sample from the Dewey Lake Red Beds (DLRB-2, Table 4). In this reaction, aluminum is considered to be a mobile component. Reaction 1 suggests an additional environmental factor that aids the transformation process: a relatively high pH will help to drive the reaction to the right. The importance of this geochemical variable to the above reaction, however, is ambiguous because both high pH values (Sonnenfeld, 1984) and low pH values (Lambert, 1978) have been reported in evaporite brines wherein these mineralogical transformations could occur.

In Reaction 1, silicon is replaced by aluminum in the tetrahedral layer, while magnesium displaces aluminum in the octahedral layer. Considerable amounts of silica and alumina are released in the process, and, although authigenic quartz and K-feldspar have been identified in these samples (Sewards et al., 1991b), additional work is required to determine if the amounts of these authigenic phases are sufficient to account for the amounts of Al and Si released.

The transformation from saponite to corrensite might take the following form:

$$2\text{Mg}_{0.32}\text{Ca}_{0.68}\text{Na}_{0.07}\text{K}_{0.19}(\text{Al}_{0.57}\text{Fe}_{0.63}\text{Mg}_{4.8})\text{Al}_{1.54}\text{Si}_{6.46}\text{O}_{20}(\text{OH})_4 + 0.01\text{Ca}^{2+}$$

$$+ 0.21\text{Fe(OH)}^+ + 4.6\text{Mg}^{2+} + 9.93\text{H}_2\text{O} \rightarrow$$

$$\text{Mg}_{0.32}\text{Ca}_{0.08}\text{Na}_{0.07}\text{K}_{0.19}(\text{Al}_{0.57}\text{Fe}_{0.63}\text{Mg}_{4.8})\text{Al}_{1.54}\text{Si}_{6.46}\text{O}_{20}(\text{OH})_4$$

$$+ 0.73\text{Na}^+ + 1.26\text{Al(OH)}_4^- + 1.22\text{H}_4\text{SiO}_4 + 10.15\text{H}^+$$

(1)

In Reaction 1, silicon is replaced by aluminum in the tetrahedral layer, while magnesium displaces aluminum in the octahedral layer. Considerable amounts of silica and alumina are released in the process, and, although authigenic quartz and K-feldspar have been identified in these samples (Sewards et al., 1991b), additional work is required to determine if the amounts of these authigenic phases are sufficient to account for the amounts of Al and Si released.
This reaction corresponds to deposition of a hydroxide layer in every other interlayer space of the saponite structure, with rearrangement of the dioctahedral and tetrahedral layers to adjust the charge balance. In Reaction 2, the DLRB-2 saponite structural formula has been used, as well as the corrensite formula for sample AIS-15 (Table 4). As in Reaction 1, the primary factors that drive the reaction to the right are high Mg\(^{2+}\) concentration and high pH. Some of the Al lost from the montmorillonite to saponite transformation appears to be regained; however, because the compositions of corrensite are variable, this may be an artifact of the specific structural formulas chosen to illustrate the reaction.

6.1.2 Chlorite Diagenesis

The formation of chlorite may occur in one of two ways: directly from saponite by deposition of hydroxide layers in all the interlayer spaces, or the formation of a mixed-layer chlorite/saponite may be a necessary step between saponite and the end product. The second mechanism is supported by both Bodine (1985b) and Chang et al. (1986). Chang et al. (1986) have documented the transformation via chlorite/saponite in shales and sandstones as a function of depth.

The transformation of corrensite to chlorite might take the following form:

\[
\text{Ca}_{0.04}\text{Na}_{0.27}\cdots \text{K}_{0.36}(\text{Al}_{4.88}\text{Fe}_{4.97}\text{Mg}_{6.98})\text{Al}_{3.13}\text{Si}_{12.89}\text{O}_{40}\text{(OH)}_8 + 4.42\text{Mg}^{2+} + 1.79\text{H}_4\text{SiO}_4 + 7.705\text{H}_2\text{O} \rightarrow \\
2(\text{Al}_{2.99}\text{Fe}_{2.14}\text{Mg}_{5.7}\text{Al}_{0.66}\text{Si}_{1.34}\text{O}_{20}\text{(OH)}_{16} + 0.04\text{Ca}^{2+} + 0.27\text{Na}^+ + 0.36\text{K}^+ + 0.65\text{Al(OH)}_2 + 0.69\text{Fe(OH)}_3 + 7.28\text{H}^+ (3)
\]

Here, the corrensite structural formula for sample R-35 and the chlorite formula for sample AIS-17 (Table 5) have been used. The corrensite formula was chosen because of its high iron content, which roughly matches the chlorite Fe content.

6.2 Implications of Clay Mineralogy Studies for Radionuclide Migration at the Waste Isolation Pilot Plant

The WIPP in southeastern New Mexico is being designed for the geologic disposal of defense-generated transuranic (TRU) wastes. Initial performance assessment calculations
(Lappin et al., 1989; Marietta et al., 1989) indicate that significant releases of radionuclides may occur if the repository and a pressurized brine pocket in the underlying Castile Formation are intruded by a borehole after final emplacement of the waste. Culebra mineralogy is important because the Culebra could be a pathway for long-term migration of radionuclides to the accessible environment in the event of such a repository breach.

The rate of radionuclide migration relative to brine flow within the Culebra will strongly depend on solution composition and the nature of the minerals exposed to the solutions. Chemical interactions between radionuclides and clays may be important for radionuclide retardation. The dominant mineral in the Culebra is a fairly pure dolomite which comprises about 85% by weight of the bulk rock (Sewards et al., 1991a). However, in the most transmissive parts of the Culebra, fluid may flow primarily through fractures (Lappin et al., 1989). Gypsum, CaSO$_4$·2H$_2$O, and clays are the most abundant fracture-fill minerals (Sewards et al., 1991a; Sewards, 1991). Clay minerals comprise 1 to 10 wt.% of the bulk rock and 1 to 43 wt.% of the fracture-lining material (Sewards, 1991). Studies by x-ray diffraction, atomic absorption, and analytical electron microscopy indicate that corrensite makes up a significant proportion (31 - 80 wt.%) of the Culebra clay samples examined in this report. Corrensite is also present in mudstone layers within the Tamarisk and unnamed members of the Rustler Formation.

Characterization of the adsorption potential of clay minerals in the Culebra is necessary for estimating effective retardation factors for use in performance assessment calculations. Formulation of a diagenetic model for the clays will aid in understanding their distribution and in estimating clay abundance along potential radionuclide transport paths.

Only small amounts of clay can be sampled from the Culebra fracture coatings; therefore, initial technique and model development for adsorption studies on WIPP clays (Park et al., in review) were carried out with material from a black shale layer in the unnamed member. This material, so-called CorWIPP, is 94% corrensite and is described as Sample AIS-15 in this report. Corrensite has a high cation exchange capacity and affinity for the uranyl ion in dilute solution (Park et al., in review) and could provide significant radionuclide retardation in fractures in the Culebra.

The diagenetic model for clay formation proposed in this report suggests that a suite of mixed-layer clays with moderate-to-high cation exchange capacities will be present in significant amounts along potential radionuclide transport paths. Studies of the morphology and composition of clay crystallites suggest that the corrensite formed from alteration of detrital dioctahedral smectite in Mg-rich pore fluids during deposition of the Rustler
Formation. Such transformations may have occurred over a large areas in the Delaware Basin. Similar reactions leading to creation of a reactive Mg-Si-rich layer have been proposed for the formation of neoform Mg-rich clays in Lake Abert sediments by Jones and Weir (1983).

The wide-spread occurrence of mixed-layer clays along hydrologic flow paths is also supported by studies of the composition of Culebra groundwater (Siegel et al., 1991). Correlations of solute concentrations and mass balances of major ions along modern flow paths suggest that reactions among solutes and clays have modified the composition of Pleistocene-age groundwater in the Culebra. Interelement correlations are consistent with several reactions, including dissolution of amorphous silica and the neoform Mg-rich layer in corrensite, Mg/Na ion exchange, and uptake of Li and B by ion exchange, or lattice substitution. Mass balance calculations for major ions along modern flow paths also suggest that ion-exchange (Na-Mg or Na-Ca exchange) has occurred (Ward et al., 1990).

Ward et al. (1990) calculated the mass transfer required to account for changes in groundwater composition along postulated modern flow paths based on analyses of waters sampled in a series of wells. Many possible reaction sets are consistent with the observed mass transfer. However, those reaction sets without significant amounts of ion exchange were either thermodynamically unfavorable or required dissolution of large amounts of evaporite minerals not observed in the Culebra or in layers adjacent to the Culebra.

Unequivocal identification of corrensite is difficult due to the complex mixture of clays present in the fracture-coatings in the Culebra, uncertainties in the interpretation of the x-ray diffractograms due to the small basal extent of the corrensite crystals, and rapid damage rates and poor diffraction contrast during high-resolution transmission electron microscopy (HRTEM) imaging. However, in spite of these difficulties, estimates of corrensite abundance obtained by HRTEM are in general agreement (about 5-10% lower) with those obtained by x-ray diffraction. Thus, although an exact value for corrensite abundance is uncertain, it is clear that mixed-layer clays similar to corrensite make up a significant fraction of the clay samples studied. Confirmation that such mixed-layer clays are abundant along the flow paths can be obtained by additional mineralogical studies of fracture coatings from available cores.
7.0 CONCLUSIONS

Detailed mineralogical studies have been performed on matrix and fracture-fill materials from a large number of samples from the Rustler Formation. These analyses provide the basis for estimates of the abundance and nature of clay minerals that are present along potential radionuclide transport paths between the WIPP and the accessible environment. In addition, these studies provide the basis for comparison of the properties of the Culebra fracture clays to those of the CorWIPP reference corrensite used in radionuclide adsorption studies.

The commonly occurring clay minerals in the Culebra are (in order of abundance): corrensite (regularly interstratified chlorite/saponite) > illite > chlorite > serpentine. Corrensite is also the dominant mineral in two shale layers of the unnamed member of the Rustler Formation directly below the Culebra. Within other layers of the Rustler Formation, disordered mixed chlorite/saponite is usually the most abundant clay mineral. Estimates of corrensite abundance obtained by HRTEM are in general agreement (about 5-10% lower) with those obtained by x-ray diffraction. Thus, although the exact proportion is uncertain, it is clear that corrensite makes up a significant fraction of the clay in the Culebra.

On the basis of the morphological data obtained on the TEM and the compositional data, four conclusions can be drawn concerning burial diagenesis of clay minerals in the Rustler Formation:

- the original clay mineral assemblage introduced into the Delaware Basin at the time of evaporite deposition probably included illite and dioctahedral smectite;
- illite was relatively stable, even in the extreme conditions of an evaporitic environment, and underwent essentially no compositional or morphological changes;
- dioctahedral smectite was initially transformed into saponite and thereupon into either mixed-layer chlorite/saponite (chlorite/saponite) and finally, in some cases, chlorite; and
- all these diagenetic reactions probably occurred by the "layer-by-layer" mechanism (Bethke and Altaner, 1986), in which the original morphology of the detrital clay minerals was conserved, and compositional changes occurred by solid-state diffusion and deposition of hydroxide layers in smectite interlayer spaces.
8.0 REFERENCES


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Hobbs Public Library
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New Mexico State Library
Attn: N. McCallan
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New Mexico Junior College
Pannell Library
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8523-2 Central Technical Files
9300  J.E. Powell
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