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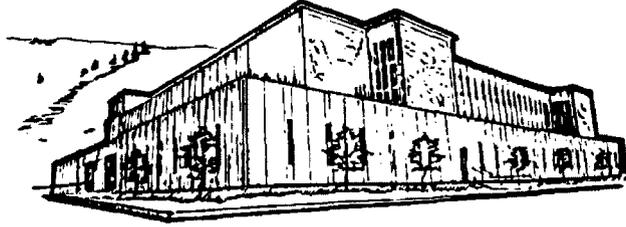
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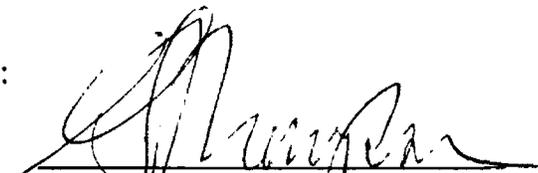
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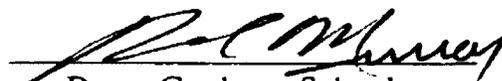
A Survey of the Clay Mineralogy
and Diagenesis in the Williston Basin.

by
Jeffrey A. Moe
B. A. University of Montana, 1988

Presented in partial fulfillment of the requirements
for the degree
Master of Science
University of Montana
1990

Approved by:


Chairman, Board of examiners


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Moe, Jeffrey A., M.S., June 1990

Geology

A survey of the clay mineralogy and diagenetic petrology of the Williston Basin (26 pp.)

Director: Graham R. Thompson

GRT

The Williston basin occupies large areas of eastern Montana, western North Dakota, and southern Saskatchewan. Cuttings from a well near the depocenter of the 12,000 foot basin provides samples from the entire Phanerozoic section occurring in the basin. The clay mineralogy reflects diagenetic trends in response to increasing burial. A mineralogic discontinuity coincides with a major stratigraphic unconformity.

Illite is present throughout the well. In the top 5,700 feet, $R = 0$ mixed layer illite/smectite (I/S) coexists with discrete illite and minor amounts of kaolinite and chlorite. The expandability of the I/S decreased with depth. Near the 5,700 foot level I/S begins to exhibit $R = 1$ ordering. At 5,700 feet the mineralogy changes at or near a major stratigraphic unconformity.

Below this unconformity illite coexists with mixed-layer chlorite/smectite (C/S) and discrete chlorite. The expandability of the C/S decreases with increasing depth and consistently exhibits $R = 1$ or greater ordering. This trend continues until the deeper parts of the well contain only illite and chlorite in the clay fraction.

TABLE OF CONTENTS

ABSTRACT	ii
TABLE OF CONTENTS	iii
LIST OF FIGURES	iv
INTRODUCTION	1
GEOLOGIC SETTING	3
The Williston Basin	3
Stratigraphy of the Williston Basin	4
SAMPLING AND ANALYSIS	5
Sample Preparation	5
X-ray Diffraction.	6
Modeling	6
RESULTS	9
Overview of the Mineralogic Trends	9
The Zuni And Tejas Sequences	10
The Absaroka and Kaskaskia Sequences	14
The Tippecanoe and Sauk Sequences	17
DISCUSSION	18
ACKNOWLEDGMENTS	21
REFERENCES CITED	23

TABLE OF CONTENTS

ABSTRACT	ii
TABLE OF CONTENTS	iii
LIST OF FIGURES	iv
INTRODUCTION	1
GEOLOGIC SETTING	3
The Williston Basin	3
Stratigraphy of the Williston Basin	4
SAMPLING AND ANALYSIS	5
Sample Preparation	5
X-ray Diffraction.	6
Modeling	6
RESULTS	9
Overview of the Mineralogic Trends	9
The Zuni And Tejas Sequences	10
The Absaroka and Kaskaskia Sequences	14
The Tippecanoe and Sauk Sequences	17
DISCUSSION	18
ACKNOWLEDGMENTS	21
REFERENCES CITED	23

LIST OF FIGURES

Figure 1. Location of the Williston Basin	2
Figure 2. Stratigraphy of the Williston basin.	4
Figure 3. Chlorite/smectite 002 reflection versus expandibility.	7
Figure 4. Chlorite/smectite 004 reflection versus expandibility.	8
Figure 5. Summary of the clay mineralogy.	9
Figure 6. Representative XRD patterns of the clay fraction.	11
Figure 7. XRD and calculated patterns at 3000 feet below surface.	13
Figure 8. XRD and calculated patterns from 5700 feet below surface.	14
Figure 9. XRD and calculated patterns from 6300 feet below surface.	15
Figure 10. XRD and calculated patterns from 7400 feet below surface.	17

INTRODUCTION

A suite of clay minerals from the Williston Basin shows progressive diagenetic changes with increasing depth. A major mineralogic discontinuity coincides with a major stratigraphic boundary.

Low-grade metamorphic clay mineral reactions in response to increasing burial depth in sedimentary basins have been described in a number of different sedimentary and tectonic environments. Diagenetic reactions in marine continental margin basins have been described by Hower, et al (1976), Boles and Franks (1979), and Howard (1987) for the North American Gulf Coast; by Jennings and Thompson (1986) for the Colorado River Delta; by Velde et al (1986) for the Niger Delta, by Anjos (1986) for the Compos Basin of Brazil; and by Chang et al (1986) for the Cassipore, Ceara, Ilha De Santana, and Potigar Basins of Brazil. Clay mineral reactions in non-marine continental basins have been described by McLeod (1987) and by McCarty and Thompson (1989). Clay diagenesis in intra-cratonic marine basins have been described by Dunoyer De Segonzac (1970).

The most common clay mineral reaction described for burial diagenesis in clay-rich sedimentary basins is progressive illitization of dioctahedral smectite with increasing burial depth. Temperature, system composition, and time are thought to control the illitization reaction (Hower, et al, 1976; Jennings and

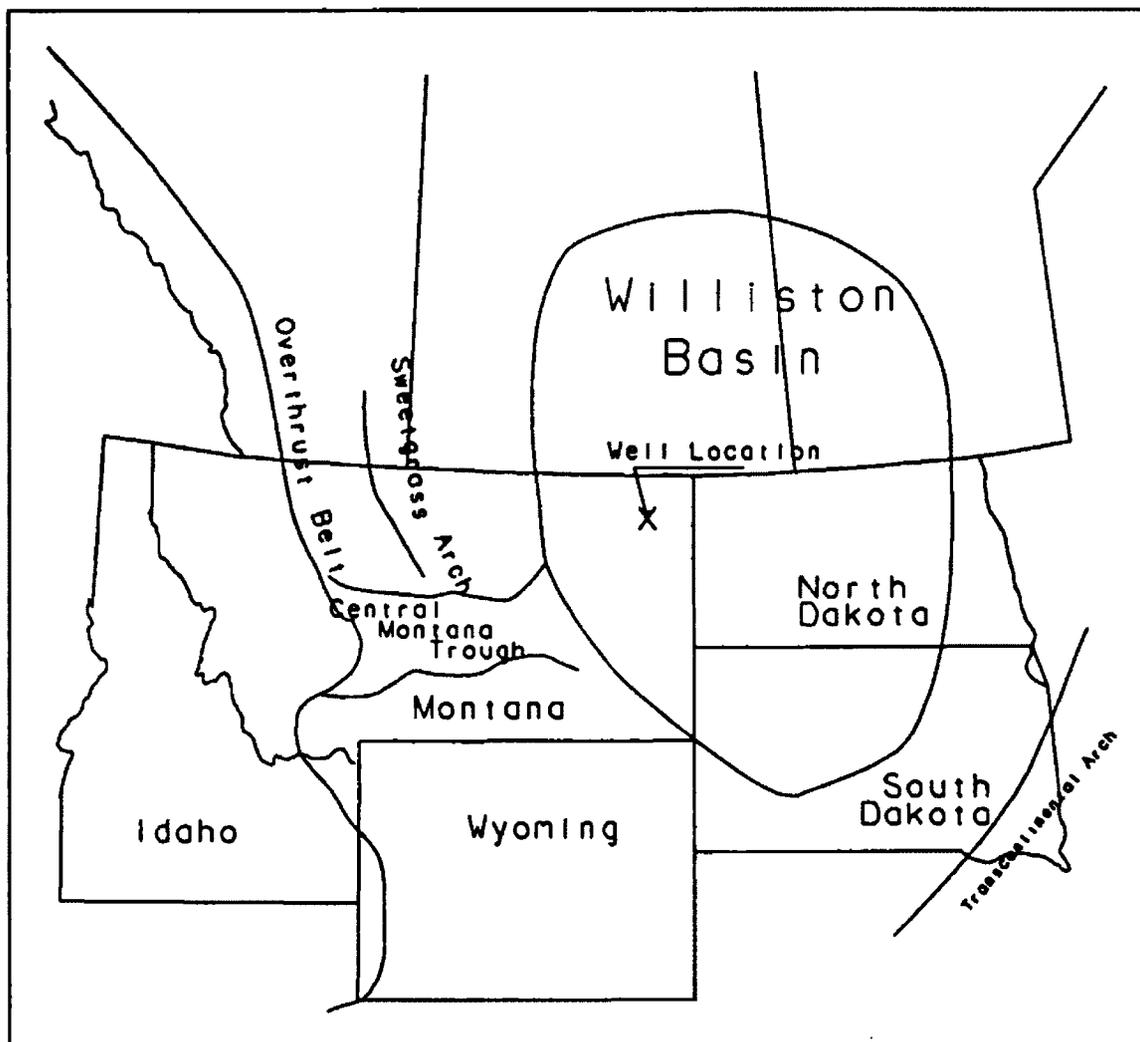


Figure 1. Location of the Williston Basin

Thompson, 1986; Velde et al, 1986; Huang, 1988; Whitney and Northrup, 1988; Boles and Franks, 1979; Howard, 1987).

A few studies show clay diagenetic reactions other than the simple, progressive illitization of dioctahedral smectite. Chang et al (1986), Boles and Franks (1979), Howard (1987), and McCarty and Thompson (1989) have attributed those deviations to variations in system composition. Those compositional variations are thought to result either from stratigraphic variations

in bulk composition, or to stratigraphically controlled variations in permeability of sediments, which in turn cause variations in composition of formation waters.

In this study I describe clay mineralogical trends that reflect progressive low-grade metamorphism with increasing depth in the Williston Basin. However, a major mineralogic discontinuity in that trend coincides with a major stratigraphic boundary. I interpret that discontinuity to result from a change in bulk composition of the sediments produced by a change in sedimentary environment and/or provenance.

GEOLOGIC SETTING

The Williston Basin

The Williston basin is a large intracratonic basin in the North American craton. This oblong basin lies beneath southern Saskatchewan, western North Dakota and eastern Montana (Figure 1). Approximately 12,000 feet of Phanerozoic sediments have accumulated above Precambrian crystalline basement rocks in the deepest part of the basin (Gerhard et al, 1982).

Stratigraphic studies of rocks exposed at the margin of the Williston basin and data obtained from exploratory drilling and petroleum development within the basin permit correlation with stratigraphic units recognized outside the basin. The stratigraphic section in the Williston Basin is interrupted by major unconformities recognized throughout the craton. The unconformities have been used to define informal stratigraphic subdivisions known as sequences (Sloss, 1963). These

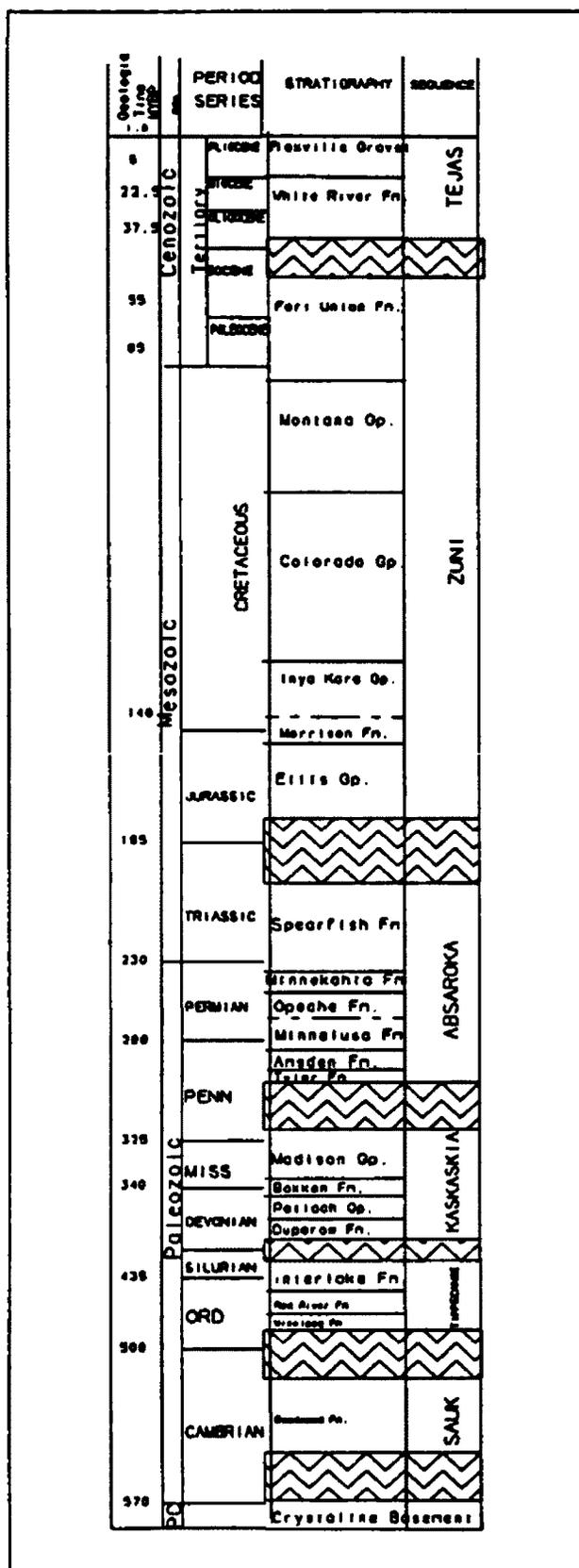


Figure 2. Stratigraphy of the Williston basin.

sequences are used extensively to describe the stratigraphy of the Williston basin (Figure 2). Within each sequence, deposition was continuous. The unconformities at the sequence boundaries reflect intervals of non-deposition or erosion and commonly coincides with major changes in depositional environments and provenance (Gerhard et al, 1982).

Stratigraphy of the Williston Basin

The Tejas Sequence consists of poorly indurated sediments deposited from Oligocene to Recent time (Figure 2) (Bluemle, 1980) (Gerhard et al, 1982). The Zuni Sequence consists of deltaic shale, sandstone, and shallow marine limestone deposited from Jurassic to Eocene time. The detrital component of both the Tejas and Zuni

Sequences was derived from erosion of uplifted volcanic and sedimentary rocks to the west (Gerhard et al, 1982).

The Absaroka, Kaskaskia, Tippecanoe, and Sauk sequences consists of carbonate and clastic sediment derived from erosion of Precambrian basement rocks of the North American craton. Lithologies in these sequences include deep water shale, evaporite, limestone, and sandstone. Deposition of the Tippecanoe sequence marks the beginning of sedimentation in the tectonically sinking Williston Basin (Gerhard et al, 1982) (Figure 2).

SAMPLING AND ANALYSIS

The library of well samples maintained by the Montana Board of Oil and Gas in Billings Montana is the source of all samples for this study. Samples were collected at intervals dictated by sample availability and original site sampling. All samples are well chips and of limited quantity. Well logs, E-logs, and neutron logs were used to determine stratigraphic location of the samples and boundaries used in this study. The well sampled is located in Richland county, Montana approximately 100 km west of Williston, North Dakota (Figure 1).

Sample Preparation

Drilling contaminants were removed from the samples by hand picking under a binocular microscope. The rock chips were further cleaned using an ultrasonic probe. After drying at less than 50°C, the samples were crushed in an agate mortar and disaggregated in deionized water with an ultrasonic probe. The less

than 1.0 micron equivalent spherical diameter size fraction was separated from the disaggregated suspension by centrifugation. Oriented samples of the less than 1.0 micron size fraction were prepared for X-ray diffraction by filter-membrane peel technique on glass slides (Pollastro 1982). All samples were strontium saturated and glycol solvated in a heated solvation chamber for 24 hours to fully expand the expandable component. Selected samples were heated to 580°C for 1 hour to collapse the expandable phases and detect kaolin group minerals. Several were digested in heated HCl for 1 hour to destroy chlorite.

X-ray Diffraction.

X-ray diffraction (XRD) patterns of the prepared samples were run from 2 to 50 degrees two-theta on a Philips X-ray diffractometer with a digitally controlled scanning goniometer using copper K alpha radiation and a graphite crystal monochromator. Identification of clays and detailed structural interpretations were made using methods described by Carroll (1970), Srodon (1980; 1981; 1984), Reynolds (1985), Moore and Reynolds (1989), and Brindley and Brown (1980).

Modeling

Newmod (Reynolds, 1985) was used to model XRD patterns of specific phases i.e.; illite, smectite, kaolinite, mixed-layer illite/smectite (I/S), mixed-layer chlorite/smectite (C/S), and corrensite. Mixmod (Reynolds, 1985) was used to combine the calculated patterns to simulate selected XRD patterns and to aid in the precise identification of all phases in physical mixtures of clay minerals.

Analysis with Newmod and Mixmod provide quantitative interpretations of XRD data.

Relative XRD peak intensities are most affected by both amounts and structural position of iron. The iron content of clay samples was estimated by comparison of XRD patterns with calculated patterns with varied iron content and distribution. Iron contents for the Newmod input parameters are given in percent. Thus, a 50% iron content indicates one half of the octahedral sites are occupied by iron. The remaining sites are occupied by magnesium and aluminum both of which produce similar XRD effects that differ from those of iron.

Scherrer analysis was applied to the mixed-layer I/S peaks in the XRD patterns to estimate the expandability for modelling. Peaks used in the Scherrer analysis were corrected for machine broadening using a mica standard (Eberl et al, 1987).

To estimate expandability for the mixed-layer C/S minerals, a series of C/S patterns was calculated with Newmod for a range of expandabilities from 65% to 0% at 10% intervals. In those calculated patterns both the d002 and d004 peaks migrate toward smaller d-spacings in response to decreasing

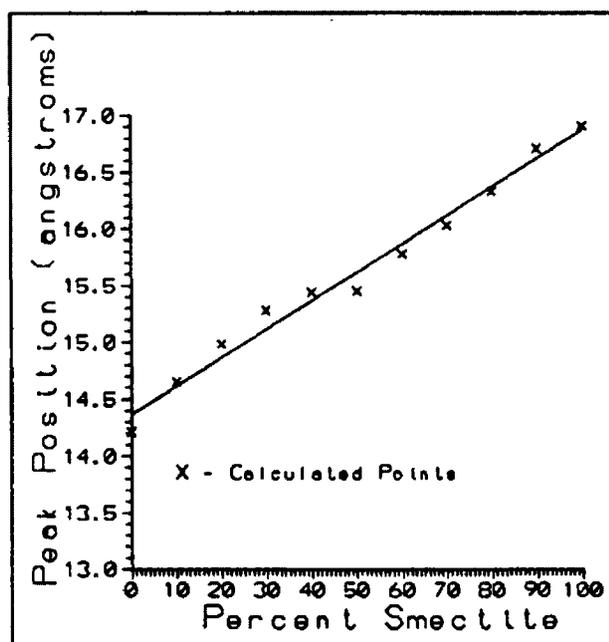


Figure 3. Chlorite/smectite 002 reflection versus expandability.

expandability. Graphs of d002 and d004 peak positions plotted against % expandability show linear relationships (Figures 3 and 4).

Corrensite is a regularly interstratified 1:1 mixed-layer trioctahedral chlorite and trioctahedral smectite. The coefficient of variability (CV) index (Reynolds, 1988) is a measure of

regular interstratification. The AIPEA Nomenclature Committee defines corrensite as having a $CV \leq .75$ (Bailey, 1982). Regular interstratification of chlorite and smectite layers in corrensite produces a superlattice reflection at 32 \AA . The CV index was calculated for XRD patterns with a 32 \AA peak and those samples with a $CV \leq .75$ are referred to as corrensite.

Heat treatment and HCl digestion were used to identify minor amounts of chlorite and kaolin (Carroll, 1970; Moore and Reynolds, 1989). If they were identified in samples, they were included as components in Mixmod calculated patterns used to simulate the XRD pattern for those samples.

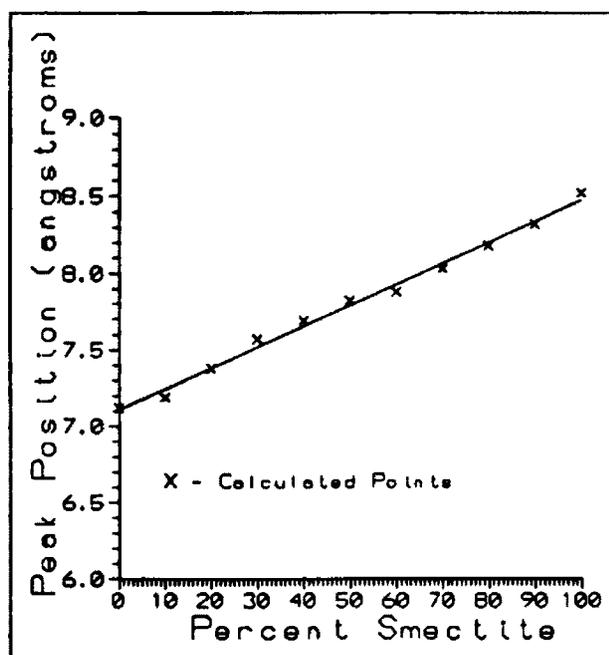


Figure 4. Chlorite/smectite 004 reflection versus expandability.

RESULTS

Overview of the Mineralogic Trends

Approximately 200 samples from this well were analyzed using the methods described above. Figure 5 summarizes the mineralogic data obtained from these analyses.

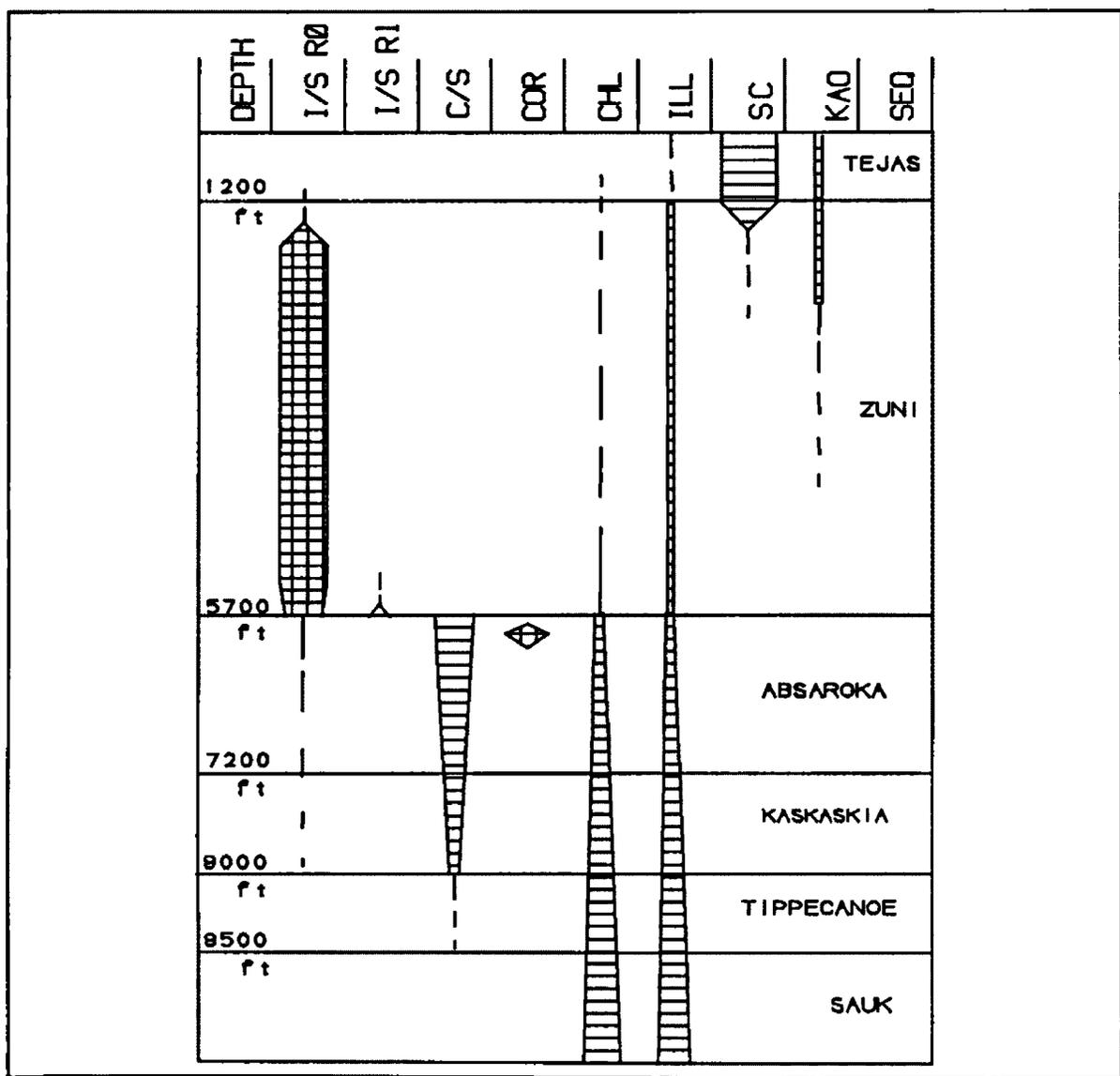


Figure 5. Summary of the clay mineralogy.

Nearly all samples analyzed have 10 Å peaks and integral reflections.

Therefore, illite is present throughout the entire section.

The Tejas and Zuni sequences contain illite, kaolin, chlorite, and I/S. In the upper part of these sequences the I/S is dominated by the expandable component and is R = 0 ordered. Expandability decreases with depth and the I/S phase begins to develop R = 1 ordering near the bottom of the Zuni sequence (Figures 5 and 6).

Samples below the Zuni-Absaroka boundary contain little or no I/S but are instead dominated by C/S. The C/S show R = 1 ordering and corrensite is present in some samples. With increasing depth, the C/S decreases in expandability and may exhibit longer-range ordering. Near the bottom of the hole only chlorite and illite remain (Figures 5 and 6).

The Zuni And Tejas Sequences

The Tejas and Zuni sequences comprise approximately the upper 5700 feet of the hole. Samples from the surface to 4200 feet are similar mineralogically. XRD patterns exhibit the 17 Å 001 and integral reflections characteristic of smectite. The 10 Å 001 and integral reflections indicate illite. In addition to the illite and smectite peaks, all patterns have 7 Å peaks and integral reflections. These peaks can indicate either kaolin or chlorite (Figure 6). Heat treatment and HCl treatment indicates the presence of both kaolin and chlorite in all samples.

Figure 7 contains both a Mixmod calculated pattern and an XRD pattern from a sample 3000 feet below surface. The Mixmod pattern was calculated with

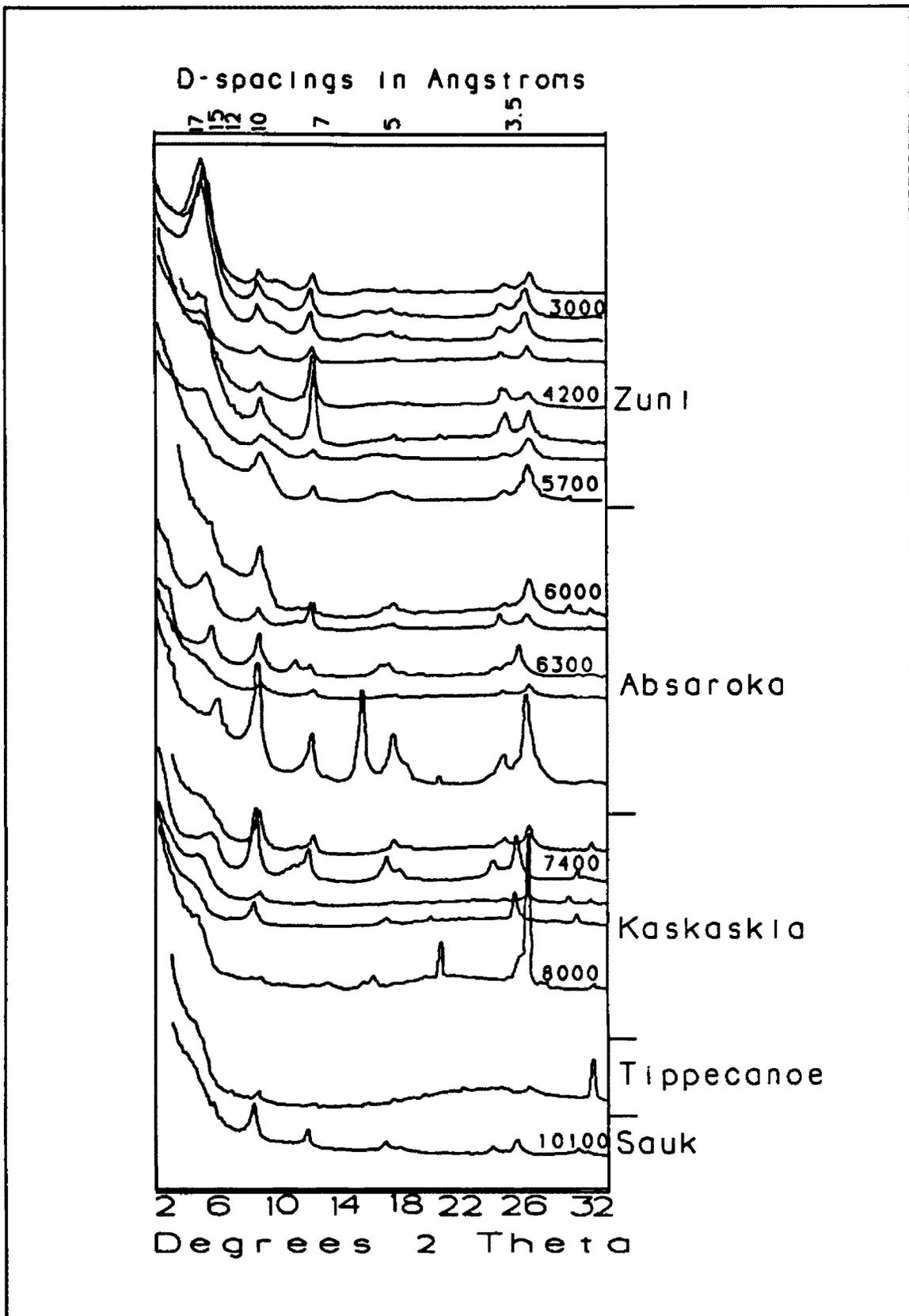


Figure 6. Representative XRD patterns of the clay fraction.

input parameters of:

- 1) 45% I/S with .9 di-mica containing 6% iron, .1 di-smectite containing 50% iron, R = 3 ordered.
- 2) 45% I/S with .8 di-smectite containing 60% iron, .2 di-mica containing 50% iron, R = 0 ordered.
- 3) 7% kaolinite.
- 4) 3% chlorite.

This calculated pattern corresponds closely to the XRD pattern. The close match suggests that the clay mineralogy at 3000 feet is described accurately by the input parameters used to calculate the Mixmod pattern.

Samples from 4200 to 5700 feet are mineralogically similar to those above 4200 feet. However, in this interval the 17 Å peaks diminish in intensity and broaden relative to samples above 4200 feet, indicating a decrease in expandability of the I/S. Patterns from 5500 to 5700 feet display a rise in low angle scattering. New peaks appear in the 11 to 13 Å region. The low angle scatter exhibits faint peaks. The position and appearance of 11 - 13 Å peaks suggest that the I/S becomes ordered and decreases in expandability in these samples. Interference from other phases and limited sample availability restrict better mineralogical characterization of these samples.

Figure 8 contains both a Mixmod calculated pattern and an XRD pattern from a sample 5700 feet below surface. The Mixmod pattern was calculated with input parameters of:

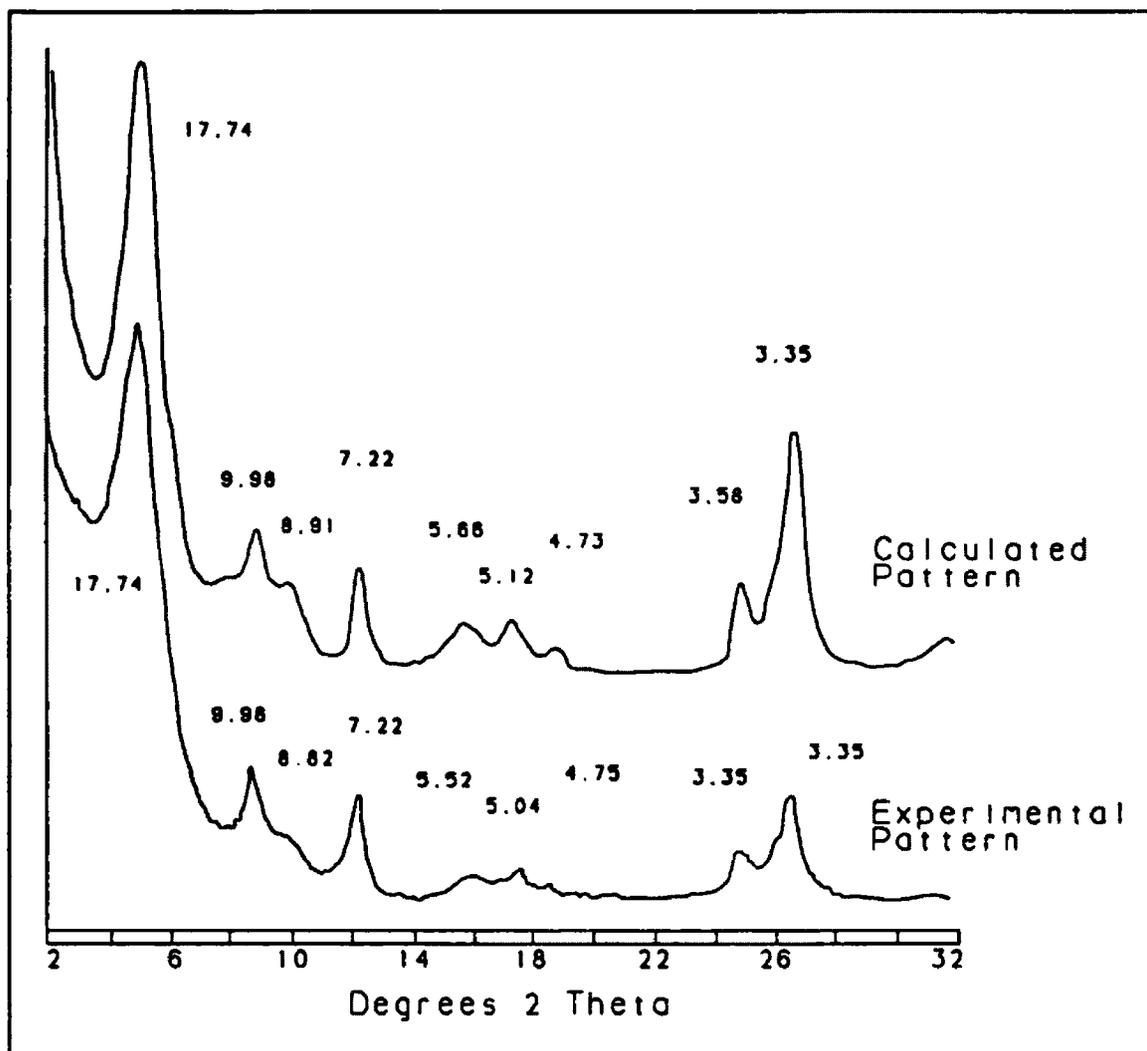


Figure 7. XRD and calculated patterns at 3000 feet below surface.

- 1) 62% I/S with .9 di-mica containing 6% iron, .1 di-smectite containing 50% iron, R = 3 ordered.
- 2) 34% I/S with .45 di-smectite containing 60% iron, .55 di-mica containing 50% iron, R = 1 ordered.
- 3) 4% chlorite

This calculated pattern corresponds closely to the XRD. The close match suggests that the clay mineralogy at 5700 feet is described accurately by the input parameters used to calculate the Mixmod pattern.

The Absaroka and Kaskaskia Sequences

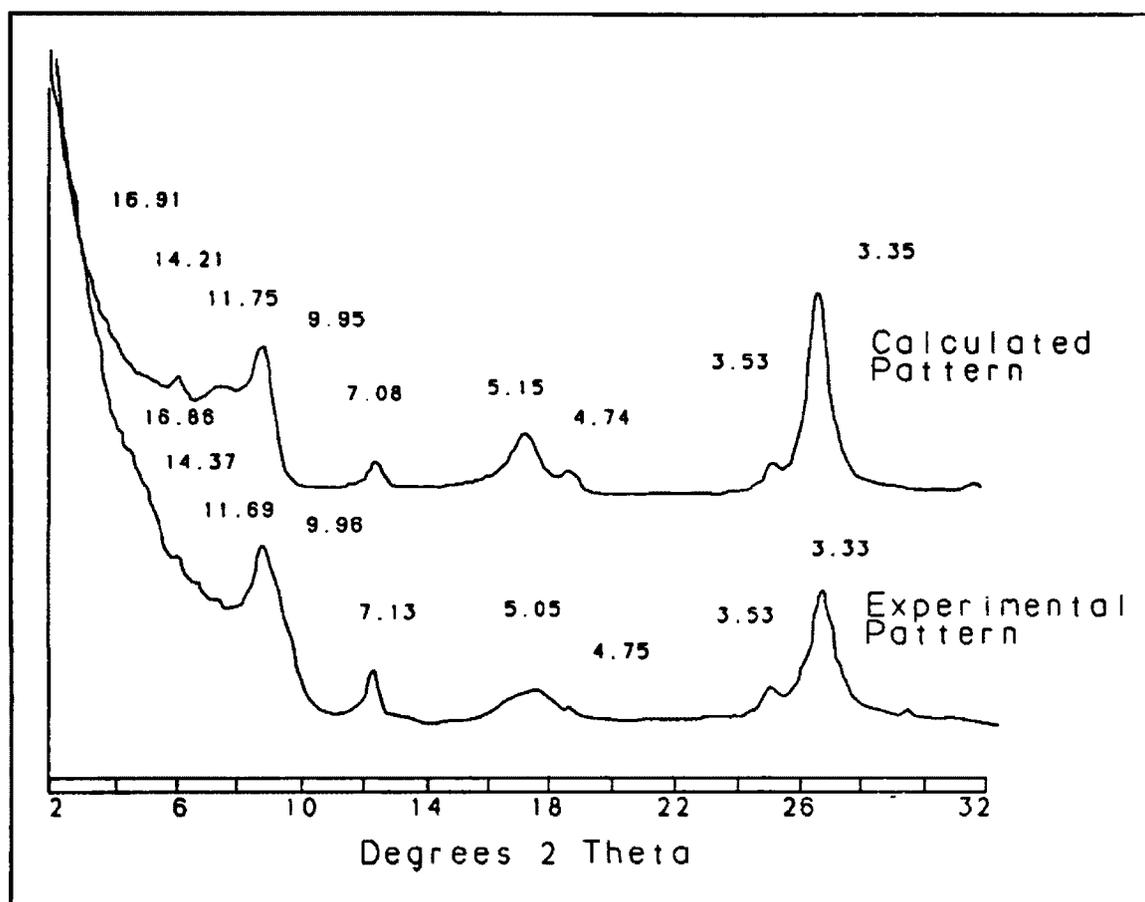


Figure 8. XRD and calculated patterns from 5700 feet below surface.

Figures 5 and 6 show that samples from the Absaroka and Kaskaskia sequences are mineralogically different from those of the Tejas and Zuni sequences. As in the Tejas and Zuni Sequences, 10 Å illite peaks are present in the Absaroka and Kaskaskia Sequences. HCl, heat treatment and the appearance

of a peak at 4.73 Å indicate that the 7 Å phase is entirely due to chlorite.

Smectite is absent from the Absaroka and Kaskaskia samples. However, a 32 Å super-lattice peak and integral reflections are present. The 32 Å super-lattice peak is poorly defined in some samples but the 16 Å peak is present in all samples

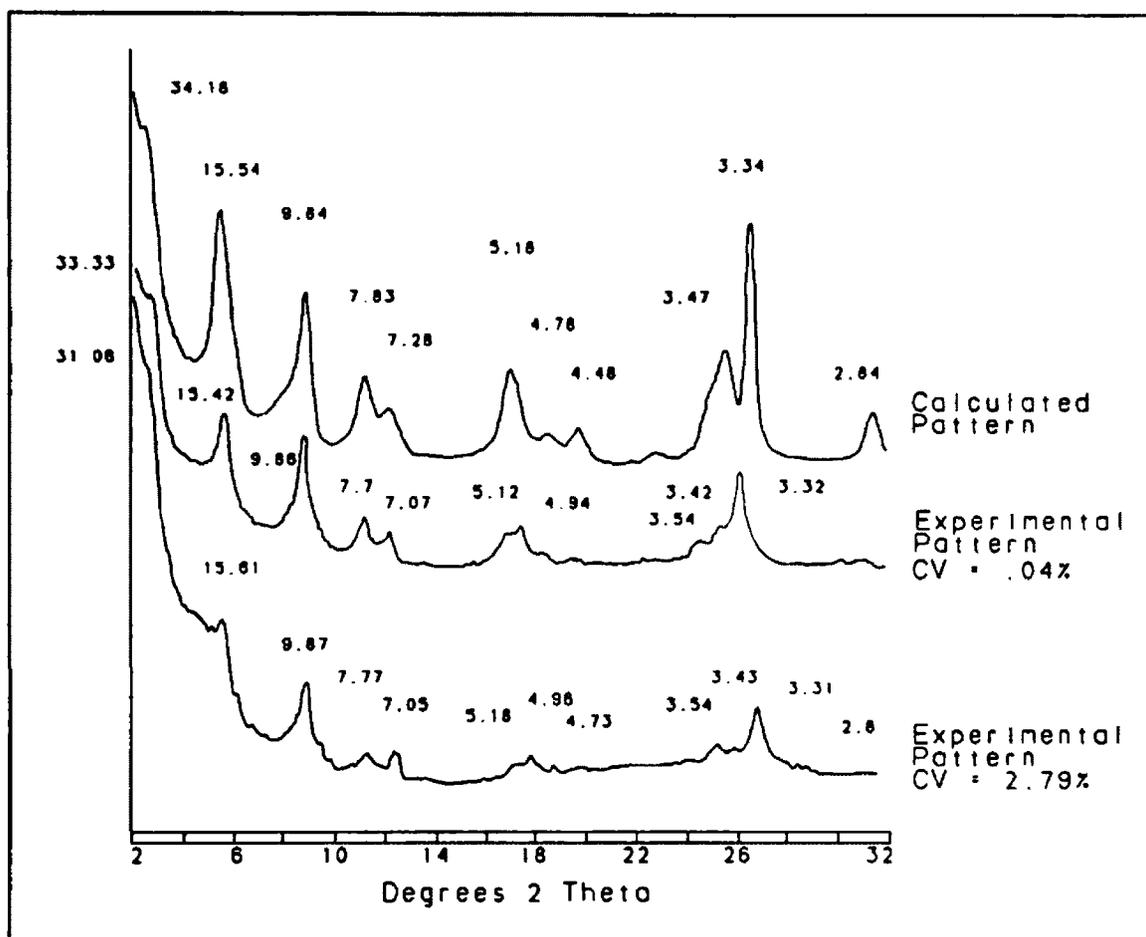


Figure 9. XRD and calculated patterns from 6300 feet below surface.

from 6000 to 7400 feet. These peaks are characteristic of C/S and corrensite.

Figure 9 contains both a Mixmod calculated pattern and two XRD patterns from a sample 6300 feet below surface. The two XRD patterns presented in figure 9 are from different cuts of a sample at a depth of 6300 feet. Although the

XRD patterns are similar, the minor differences can be attributed to the fact that chip sampling covers a stratigraphic range. Using the CV index one is C/S and the other is corrensite. The Mixmod pattern was calculated with input parameters of:

- 1) 50% I/S with .85 di-mica containing 10% iron, .15 di-smectite containing 10% iron, R = 0 ordered.
- 2) 35% corrensite with .62 tri-trichlorite, .38 tri-chlorite, containing 5% iron in 2:1 layers and 90% iron in the hydroxy layers, R = 1 ordered.
- 3) 15% chlorite

This calculated pattern corresponds closely to the XRD pattern. The close match suggests that the clay mineralogy at 6300 feet is described accurately by the input parameters used to calculate the Mixmod pattern.

Figure 10 contains both a Mixmod calculated pattern and an XRD pattern from a sample 7400 feet below surface. The Mixmod pattern was calculated with input parameters of:

- 1) 80% I/S with .85 di-mica containing 10% iron, .15 di-smectite containing 10% iron, R = 0 ordered.
- 2) 15% corrensite with .75 tri-trichlorite, .25 tri-smectite, containing 5% iron in 2:1 layers and 90% iron in the hydroxy layers, R = 1 ordered.
- 3) 5% pure chlorite parameters used to calculate the Mixmod pattern.

This calculated pattern corresponds closely to the XRD pattern. The close match suggests that the clay mineralogy at 7400 feet is described accurately by the input parameters used to calculate the Mixmod pattern.

The Tippecanoe and Sauk Sequences

The lower Kaskaskia, Tippecanoe, and Sauk sequences contain illite and

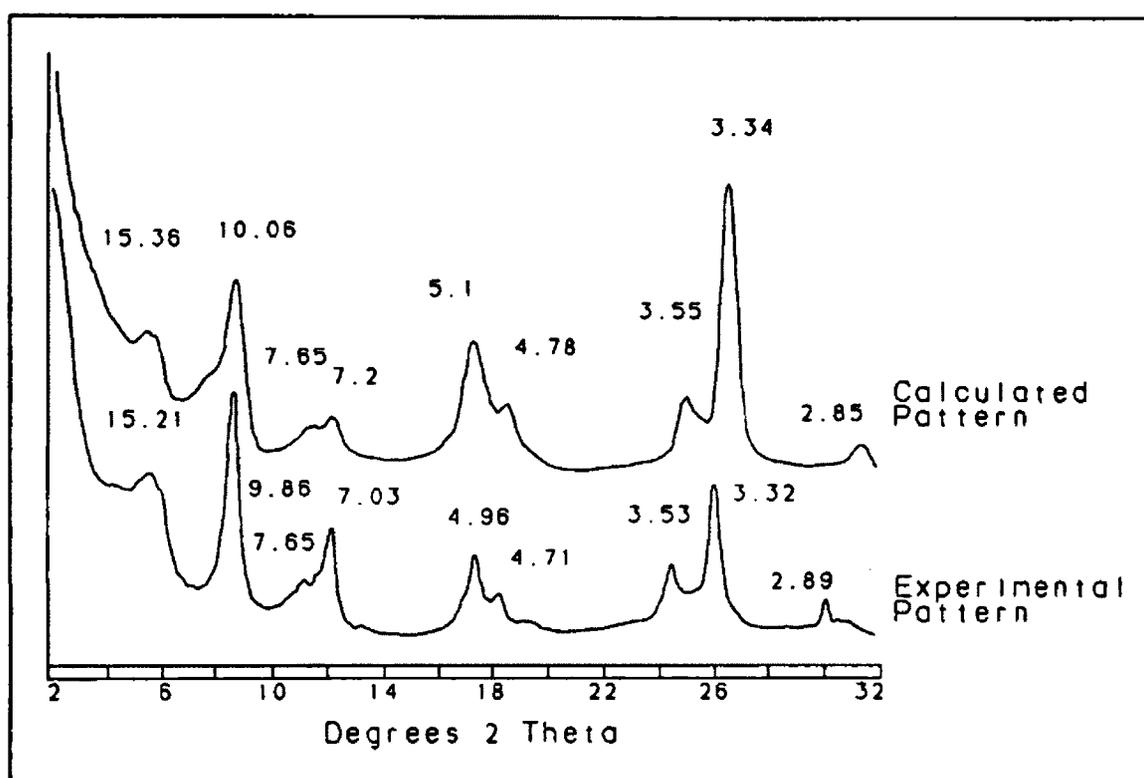


Figure 10. XRD and calculated patterns from 7400 feet below surface.

chlorite (Figure 6). A broad low angle hump in the 18\AA region indicates some expandability, probably associated with the illite and/or chlorite. This low angle hump diminishes and disappears with depth indicating a continuing loss of expandability, leaving only the illite and chlorite peaks at greater depths.

DISCUSSION

Figure 6 shows that mixed-layer clay minerals from this well in the Williston basin show a general trend of decreasing proportions of expandable layers with increasing depth. This trend transcends sequence boundaries as well as a substantial mineralogical discontinuity at the Zuni-Absaroka sequence boundary. In a general sense, this trend is similar to that of the Gulf Coast of the U. S. (Hower et al, 1976; Boles and Franks, 1979; Howard, 1987), the Salton Sea (Jennings and Thompson, 1986), and many other studies of burial diagenesis in clay-rich sedimentary basins.

In the Tejas and Zuni sequences, the shallowest in the basin, highly expandable I/S becomes progressively more illitic, as well as R = 1 ordered, with increasing depth. At the base of the Zuni sequence the I/S attains a maximum of 55% illite layers and is R1 ordered.

Immediately below the Zuni-Absaroka sequence boundary, the mixed-layer mineral is 62% non-expandable and R1 ordered. However, the non-expandable component of the mixed layer phase is chlorite rather than illite. That is, the mixed-layer mineral is a chlorite/smectite (C/S) rather than I/S. With increasing depth, the C/S becomes progressively more chlorite-rich, attaining 75 % chlorite layers at a depth of 7400 feet, and continuing to increase in proportion of chlorite below that depth.

Thus, although the proportion of expandable layers in the mixed-layer phase decreases in a regular fashion throughout the well, a major mineralogical

discontinuity occurs at the Zuni-Absaroka sequence boundary. Here, the mineralogy changes from I/S to C/S. Such a continuity in the trend of decreasing proportion of expandable layers in mixed-layer minerals, coupled with a transition from I/S to C/S, has not been reported previously.

The depositional environment of the Tejas and Zuni sequences was that of a shallow marine delta receiving volcanoclastic sediment and detritus from the rising Rocky Mountains to the west (Gerhard, 1982). Dioctahedral smectite was the major clay to form in this weathering environment (Schultz, 1963) and was the main clay supplied to the basin. Progressive illitization of the smectite accompanied burial, as has been the case with burial of other sequences rich in dioctahedral smectite (eg: Hower et al, 1976; Jennings and Thompson, 1986).

In the Absaroka and lower sequences, however, the provenance and depositional environments were quite different from those of the Tejas and Zuni sequences. During those earlier times the Williston Basin was a deep-water basin that periodically evaporated and dried up, similarly to the Mediterranean Sea in late Miocene time (Hsu and Ryan, 1973), and evaporites formed during intervals of maximum evaporation. Clastic sediment was supplied from erosion of the adjoining Precambrian crystalline basement, rather than from a rising, volcanic source. In these very different weathering and sedimentary environments, trioctahedral rather than dioctahedral smectite must have formed in abundance. This trioctahedral smectite is thought to have been the precursor mineral for the C/S that now dominates the clay mineralogy of the lower sequences.

Chang et al (1986) studied the clay mineralogy of four Cretaceous sedimentary basins in Brazil. Three of the basins filled with clay-rich sediment from a tectonically active volcanic source similar to the one that supplied sediment to the Tejas and Zuni sequences of the Williston Basin. The clay fractions of those sediments were rich in dioctahedral smectite. The smectite was progressively illitized with increasing burial depth in those three basins. The fourth basin filled with clay-rich sediment derived by weathering and erosion of crystalline basement rock of the Brazilian Shield. The most abundant clay deposited in this basin was saponite, a trioctahedral smectite. With burial, the saponite progressively converted to mixed-layer chlorite/smectite. The proportion of chlorite layers in the C/S increases with depth, much as it does in the Absaroka and lower sequences of the Williston Basin.

In a similar vein, Bodine (1985) and Bodine and Madsen (1985) have reported that C/S and corrensite become chloritized with increasing burial of Paleozoic evaporites. Additionally, development of corrensite and other types of C/S have been reported in several evaporite-forming environments (Fournier, 1961; Lippman, 1954; Kopp and Fallis, 1974; Hluchy and Reynolds, 1987; Bettison and Schiffman, 1980). Detrital influence may be less important in the formation of trioctahedral clay minerals in evaporite sequences than equilibration with interstitial pore fluids. However, in all known cases, deep burial and the resulting diagenesis converts saponite to C/S, and C/S and corrensite to chlorite.

Thus, I suggest that the mineralogical discontinuity from I/S to C/S coincident with the Zuni-Absaroka sequence boundary, is the result of a change in provenance similar to that reported by Chang et al (1988), or to a change in sedimentary environment, similar to that discussed by Fournier (1961), Lippman (1954), Kopp and Fallis (1974), Hluchy and Reynolds (1987), and Bettison and Schiffman (1980), or to a combination of both. The fact that C/S is ubiquitous throughout the Absaroka and lower sequences, whereas evaporites are sporadic, suggests that provenance rather than sedimentary environment may be the main factor controlling clay mineralogy of those sequences.

The ubiquitous presence of 10 Å illite might indicate an early heating and illitization event. This study revealed no evidence to support this interpretation. A continuous detrital input of fine grained illite is the most likely source. Polytype analysis might indicate the petrogenesis of the illite.

ACKNOWLEDGMENTS

I wish to thank Charles Maio of the Montana Board of Oil and Gas for providing access to the Library of well chips in Billings, Montana. Without this source this study could not have been done. I must also thank Dr. Gray Thompson professor of geology at the University of Montana whose tireless guidance and invaluable editing can only be out done by the inspiration he has provided me throughout my tenure at the University of Montana. Thanks also to Jim Sears and Dick Field, the rest of my committee, their input and diligent

response is deeply appreciated. I wish to acknowledge the Department of Geology at the University of Montana and the University itself for providing an environment in which this type of project can reach fruition. Finally, my family has supported me in so many ways and to them I give my deepest thanks.

All data produced in this study, both raw and processed, have been stored magnetically and is on file, along with the remaining samples and logs, in the Geology Department at the University of Montana and with both Dr. Thompson and myself.

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