

EPIGENETIC CHLORITE CRYSTALS IN FAULTED  
LOWER DEVONIAN MUDSTONES, KINGSTON,  
NEW YORK<sup>1,2</sup>

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ABSTRACT

Tiny chlorite crystals that appear to be localized on fault surfaces of mudstones were discovered in Lower Devonian (Helderberg) rocks of the central Hudson Valley, New York. Both the *Ib* and *I Ib* chlorite polytypes and  $2M_1$  trioctahedral mica were identified using X-ray powder film patterns. Indirect methods indicate that the chlorite composition is approximately  $(\text{Fe}_3\text{Al}_{1.2-1.4}\text{R})_6(\text{Al}_{1.2-1.4}\text{Si})_4\text{O}_{10}(\text{OH})_8$  where *R* signifies undetermined octahedral cations. Abundant inclusions of chlorite and a small amount of titaniferous material occur in the chlorite crystals; no 1:1 phyllosilicate mineral was detected as either an interstratified or single phase. Single crystal study of an apparent *I Ib* chlorite shows that the layer sequence is irregular and distorted from that of the ideal *I Ib* polytype. The mineral assemblage of *Ib* chlorite, distorted *I Ib* chlorite, and  $2M_1$  trioctahedral mica apparently formed by local isochemical metamorphism in a low-to-intermediate temperature range. Crystal growth was initiated by faulting and continued after fault movement ceased.

INTRODUCTION

During a mineralogical study of Lower Devonian (Helderberg) rocks of the central Hudson Valley, Borst (1966) discovered tiny chlorite crystals on fault surfaces in mudstones. This occurrence seems to be unique in that very small chlorite crystals have not, to the writers' knowledge, been reported previously from this environment even though massive chlorite commonly occurs in fault zones.

Bailey and Brown (1962) discussed the factors which apparently govern the geologic occurrence of chlorite polytypes and expressed a need for additional studies of chlorite occurrences where close control could be established for both mineral characteristics and field relationships. For several reasons the chlorite crystals of the mudstones seemed well suited for a detailed study of this type: (1) The crystals are epigenetic and occur in fresh drill core and roadcut samples; hence, the effects

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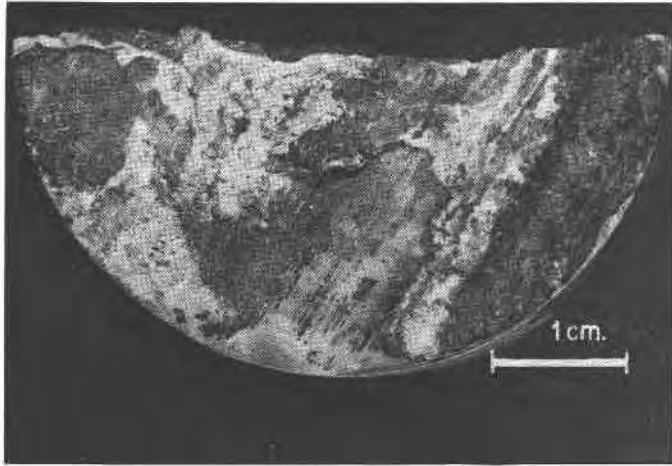


FIG. 1. Photograph of a fault surface where chlorite crystals were found. Note the slickensiding.

of provenance, weathering and depositional environment on their formation are not of concern; (2) The crystals are suitable for X-ray study by single crystal methods; (3) Both *Ib* and *IIf* chlorite and *2M*, trioctahedral mica appeared to have formed in the same environment; and (4) The mineralogy, stratigraphy and textural features of the host strata are well documented. One disadvantage was the lack of sufficient chlorite for a wet chemical analysis. Thus, chemical composition had to be determined by indirect methods.

#### OCCURRENCE AND DISTRIBUTION OF CHLORITES

The chlorite crystals were discovered during the binocular examination of cores of mudstones and carbonate rocks from Ravena, New York, 15 miles south of Albany, and Kingston, New York, 30 miles farther south. At Ravena, crystals were found in mudstones of the New Scotland Formation at core depths ranging from 84 to 187 feet. Core from several drill holes contained crystals. They were usually found on fault surfaces (Fig. 1) where slickensides and the break-up and cementation of the mudstone were apparent; however, there were a few sites where crystals appeared to be embedded in homogeneous rock. There was no preferred orientation of the crystals. Not every mudstone surface which exhibited slickensiding or fragmentation contained crystals. Both the *Ib* and *IIf* polytypes were identified by X-ray powder patterns.

Although more than two thousand feet of Lower Devonian core was searched by binocular examination at Kingston, chlorite crystals were

found in only seven pieces. Only the *I1b* polytype was identified. As at Ravena, the crystals were in faulted mudstones of the New Scotland Formation.

A roadcut encompassing the New Scotland Formation is located near Broncks Lake, between Ravena and Kingston. This section has been described by Rickard (1962) who processed several samples as a matter of routine laboratory procedure. In a sample from the lower part of the overlying Becraft Formation he found very small crystals which were *I1b* chlorites. Examination of his untreated sample showed that these crystals also came from mudstones.

*Associated Trioctahedral Mica.* A few black crystals were noted among those isolated by Rickard. X-ray powder patterns of three of these crystals showed them to be  $2M_1$  trioctahedral mica, as determined using the criteria of Yoder and Eugster (1954). Table 1 summarizes the geographic occurrence of these crystals.

TABLE 1. GEOGRAPHIC OCCURRENCE OF CHLORITE POLYTYPES AND ASSOCIATED MICA.

| Ravena              | Broncks Lake        | Kingston            |
|---------------------|---------------------|---------------------|
| <i>I1b</i> chlorite | <i>I1b</i> chlorite | <i>I1b</i> chlorite |
| <i>Ib</i> chlorite  | $2M_1$ mica         |                     |

#### PHYSICAL DESCRIPTION OF THE CHLORITE CRYSTALS

The crystals have prominent basal and prism faces and range from those with a hexagonal habit to those of irregular outline. The binocular appearance of most is one of a "bookish" crystal with relatively smooth crystal faces. However, the scanning electron micrographs of Figure 2 show that prism faces are not smooth but have a rough appearance because of the irregular stacking of thin laminae. At a magnification of  $5,000\times$  this irregularity is especially striking and in micrographs taken at  $20,000\times$ , individual lamina less than  $2,500 \text{ \AA}$  thick are resolved.

The maximum diameter of crystals from the Ravena and Kingston core is about  $\frac{1}{2}$  mm; those from the Broncks Lake roadcut are usually twice as large. Since crystals were hand-picked only from core, the size difference may be due to the separation technique of Rickard which could have preserved only the larger crystals.

The crystals range from colorless to dark brown although most are a medium brown. They have a pearly luster, are extremely soft, and are easily powdered by gently scratching with a steel scalpel. Density as

determined by the sink-and-float method is approximately 2.90, however, this value is probably high due to the presence of titaniferous inclusions.

#### INCLUSIONS IN THE CHLORITE CRYSTALS

One of the most interesting aspects of the chlorite occurrence is the large amount of included material in the crystals. Point counting showed that most crystals contain from 30 to 60 percent inclusions; a few contain more or less than this range. In grain mounts the inclusions appear

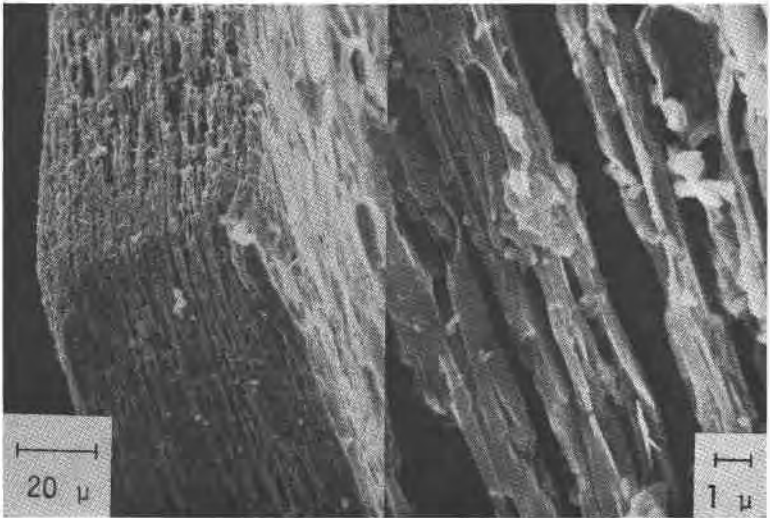


FIG. 2. Scanning electron micrographs showing the prism faces of a chlorite crystal at 500 $\times$  (left) and 5,000 $\times$  (right).

acicular, prismatic, or platy and often exhibit euhedral outlines (hexagonal, monoclinic?). Other material occurs as fine-grained aggregates devoid of crystalline form. Figure 3 illustrates some inclusions in a crystal.

The identification of the inclusions was a problem. Apparent optical properties are a small  $2V$ , negative sign, low birefringence,  $\beta$  greater than 1.610, parallel extinction, light green color under uncrossed nicols, and pale brown or abnormal blue interference colors. These data suggest that the inclusions are chlorite residing in a chlorite host. If the included material is a mineral other than chlorite, diagnostic lines should appear on X-ray powder films because the inclusions are abundant and crystalline. No lines other than those for the mineral chlorite were found on

films except for one pattern which contained a  $d$ -value of  $10 \text{ \AA}$ , the (001) spacing of mica. Spectrographic analysis showed that the crystals contain about 1 percent titanium and a titaniferous phase was identified as a recrystallization product of an aliquot of chlorite heated at  $1050^\circ\text{C}$ . This indicates that some of the included material is titaniferous.

#### X-RAY STUDY

*Powder Film Patterns.* Several crystals were powdered and X-rayed from 10 to 75 hours with either  $\text{CuK}\alpha$  or  $\text{FeK}\alpha$  radiation using the Straumanis technique and a 114.6 mm Debye-Scherrer camera. Platinum

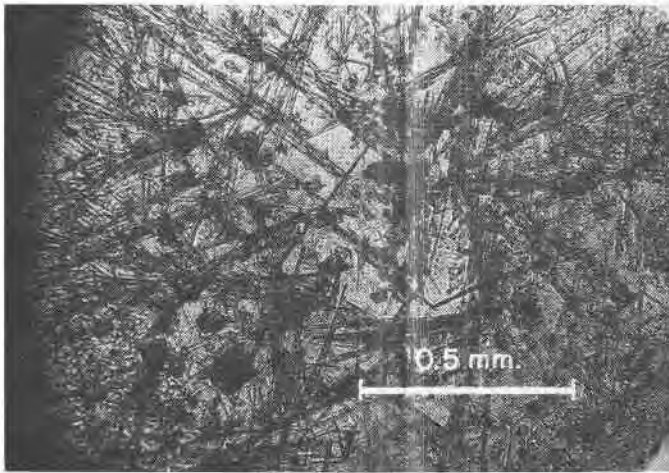


FIG. 3. Photomicrograph of inclusions in a chlorite crystal.

lines provided an internal standard. Two crystals were identified as *Ib* chlorite using the criteria of Bailey and Brown (1962). Comparison of the powder pattern of the other crystals, Table 2, with the pattern of a *IIB* chlorite listed by Bailey and Brown suggests that these crystals are *IIB* chlorites.

*Investigation of Chlorite Layer Type.* Several chlorite crystals were studied by the precession method. Examination of  $b^*c^*$  zero level photographs taken about each of the three possible  $b$  axes showed that the (020) reflection was on a line perpendicular to the  $c^*$  axis on only one of the photographs. This axis was assumed to be the unique  $b$  axis. Photographs of seven crystals showed a distribution of intensities for  $h0l$  reflections which was interpreted as that of a *Ib* chlorite (monoclinic-shaped unit cell,  $\beta = 97\text{--}98^\circ$ ). The  $0kl$  reflections allowed the stacking sequence to be

TABLE 2. X-RAY DIFFRACTION POWDER DATA FOR NATURAL CHLORITES.

| <i>I</i> Ib chlorite, from Bailey and Brown (1962) |              |                 | Chlorite from Kingston, New York <sup>a</sup> |   |
|--|--------------|-----------------|---|---|
| <i>hkl</i>   | <i>d</i> (Å) | <i>I</i>        | <i>d</i> (Å)                                  | <i>I</i> / <i>I</i> <sub>0</sub> <sup>b</sup> |
| 001  | 14.15        | 8               | 14.22   | m   |
| 002  | 7.05         | 10              | 7.18 <sup>c</sup>                             | s   |
| 003  | 4.72         | 6               | 4.75  | m   |
| 0 2; 1 1   | 4.60         | 2               | —   | —   |
| 004  | 3.54         | 10              | 3.54  | s   |
| 005  | 2.83         | 4               | 2.83  | vw  |
| 20 $\bar{1}$                                       | 2.66         | 1 $\frac{1}{2}$ | 2.66  | vw  |
| 200  | —            | —               | —   | —   |
| 20 $\bar{2}$                                       | 2.59         | 5               | 2.59  | w   |
| 201  | 2.54         | 8               | 2.55  | w   |
| 20 $\bar{3}$                                       | 2.44         | 7               | 2.45  | w   |
| 202  | 2.38         | 4               | 2.38  | w   |
| 20 $\bar{4}$                                       | 2.555        | 4               | 2.255   | vw  |
| 203  | —            | —               | —   | —   |
| 20 $\bar{5}$                                       | 2.06         | 1 $\frac{1}{2}$ | —   | —   |
| 007  | —            | —               | —   | —   |
| 204  | 2.00         | 6               | 2.00  | w   |
| 20 $\bar{6}$                                       | 1.88         | 2 $\frac{1}{2}$ | 1.89  | vw  |
| 205  | 1.82         | 2 $\frac{1}{2}$ | —   | —   |
| 1 5; 2 4; 3 1                                      | 1.74         | 1               | —   | —   |
| 207  | 1.715        | $\frac{1}{2}$   | —   | —   |
| 206  | 1.66         | 1 $\frac{1}{2}$ | —   | —   |
| 208  | 1.565        | 3               | 1.570   | vw  |
| 060  | 1.538        | 7               | 1.545   | m   |
| 062  | 1.503        | 2 $\frac{1}{2}$ | 1.510   | w   |
| 063  | 1.462        | $\frac{1}{2}$   | 1.450   | w   |
| 209  | —            | —               | —   | —   |
| 0, 0, 10 }<br>064 }<br>208 }                       | 1.414        | 1               | 1.418   | vw  |
|  | 1.392        | 2 $\frac{1}{2}$ | —   | —   |

<sup>a</sup> Film, Copper Radiation, Nickel Filter, Camera Diameter 114.6 mm;  $a=5.28_6\text{\AA}$ ,  $b=9.28_0\text{\AA}$ ,  $c=14.28_6\text{\AA}$ ,  $\beta=97-98^\circ$ .

<sup>b</sup> Visually Estimated: s, strong; m, medium; w, weak; vw, very weak.

<sup>c</sup> Measured at the low-angle edge of a diffuse band.

determined as 1-layered. Precession and Weissenberg photographs of the  $a^*c^*$  zero level are shown in Figure 4, top and bottom respectively. The reflections have been indexed and the calculated structure amplitudes of Bailey and Brown (1962) added.

Zero level intensity data were collected about the  $X$ ,  $Y$ , and  $Z$  axes

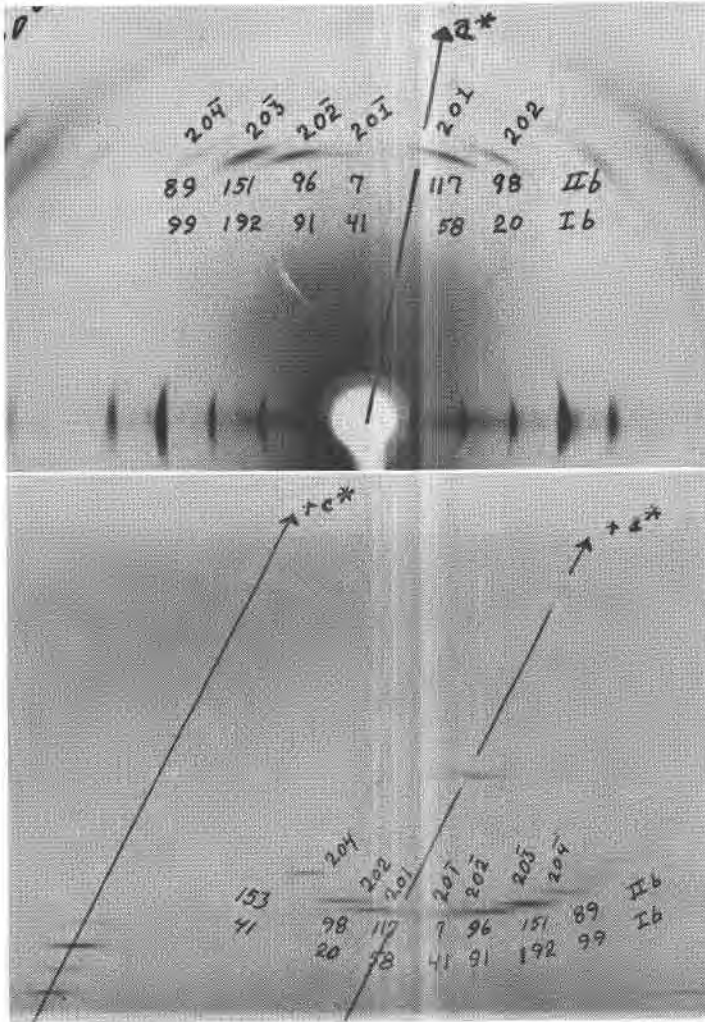


FIG. 4. Precession (top) and Weissenberg (bottom) photographs showing the  $a^*c^*$  zero level. Reflections have been indexed and the calculated structure amplitudes of Bailey and Brown (1962) for Ib and IIb chlorite added.

from multiple-film-pack integrating Weissenberg exposures. Following corrections for Lorentz-polarization and absorption, the  $h0l$  intensity distribution agreed best with the calculated structure amplitudes of Bailey and Brown for a IIb chlorite suggesting that the initial identification of the crystal as a Ib polytype was incorrect. At this point a portion of the crystal was sacrificed to make a film pattern, Table 2, which

is similar to the pattern of a natural *IIB* chlorite listed by Bailey and Brown.

*Structural Regularity.* Brindley and Gillery (1956) showed that the octahedral iron content of chlorite can be determined from the relative intensities of basal reflections. According to this method, the corrected 00 $l$  Weissenberg data of the crystal of Table 2 indicated that the chlorite contained about 6 Fe atoms per 6 octahedral sites. However, the octahedral iron content based on the  $b$  cell dimension was 3 Fe atoms. This discrepancy was similar to that reported by Brindley and Gillery (1954) for an interstratified kaolin-chlorite mineral. To test for the presence of interstratified 1:1 type layers with chlorite (2:1:1) layers, the intensities of the basal reflections were used to calculate Fourier transforms by the direct method of McEwan *et al.* (1961). Using the structure factors of Cole and Lancucki (1966), transforms were calculated for mixtures of 1:1 and 2:1:1 layers from 0 to 90 percent chlorite assuming either trioctahedral or dioctahedral 1:1 layers. No rational interpretation could be made of the resultant curves suggesting that no 1:1 layers were interlayered with the chlorite.

A 1-dimensional electron density map was constructed assuming a chlorite composition of 50 percent iron in the octahedral sheets and setting the intensities of unobserved reflections at  $F_{\min}/3$ . This curve, Figure 5 (upper), compares favorably with respect to peak shapes and heights, with the curve calculated by Brindley and Ali (1950) for a magnesian chlorite, Figure 5 (lower). This also suggests that no 1:1 layers are present in the chlorite.

Tuddenham and Lyon (1959) studied the infrared absorption spectra of chlorite and trioctahedral 1:1 phyllosilicates and found that the absorption band at 3850–3600  $\text{cm}^{-1}$  is weak or absent for chlorite but relatively strong for the 1:1 layered mineral. As shown in Figure 6, no band was observed at 3850–3600  $\text{cm}^{-1}$  for an aliquot of chlorite examined by infrared analysis, strengthening the conclusion that 1:1 layers are not interlayered with the chlorite.

Since an analysis based on interstratification did not explain the 00 $l$  intensity distribution, 2-dimensional refinement of the data was initiated. The crystal was assumed to be centered ( $C\bar{1}$ ) and the coordinates listed by Bailey and Brown (1962) for the *IIB-4* polytype were used. The  $XOZ$  electron density map indicates that the hydroxide sheet is distorted and the  $OYZ$  map shows an extra silicon atom in the 2:1 layer. Difference maps based on the contributions of just the 2:1 layer or the hydroxide sheet confirm these findings. The extra atom is shifted by  $+b/3$  or  $+b/6$  relative to the silicon atoms that belong in the *IIB-4* polytype.



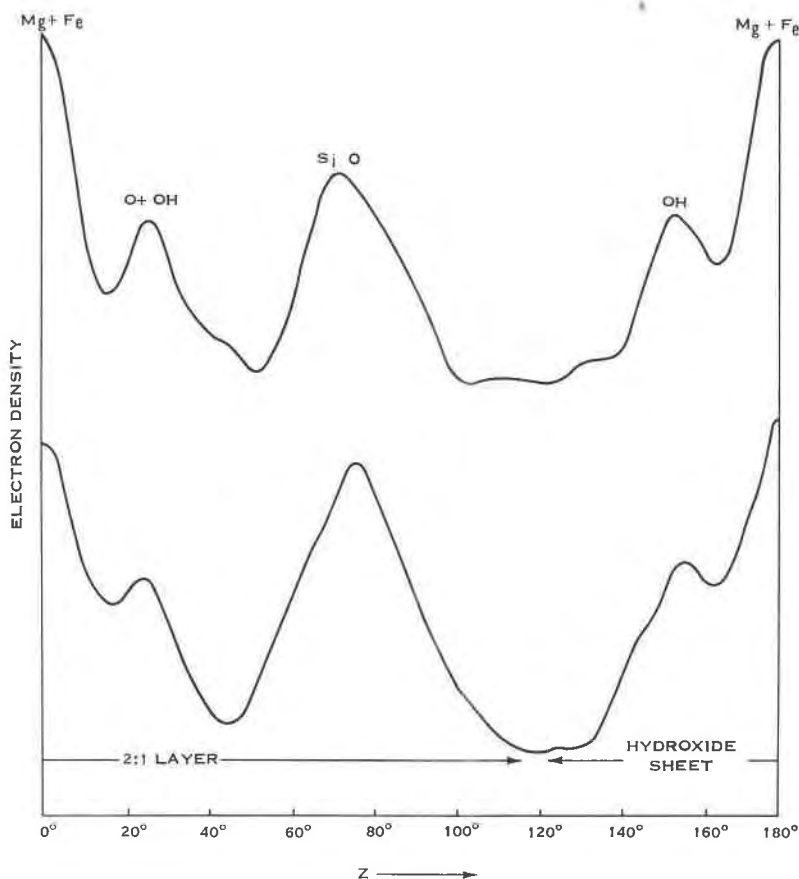


FIG. 5. One-dimensional Fourier synthesis of electron density normal to the *ab* plane of chlorite. Upper line is for the chlorite of this study; lower line is for penninite (after Brindley and Ali, 1960).

Possible interpretations to explain the presence of the atom are (1) twinning, (2) different directions of shift within the 2:1 layers of some *I**b*** layers, and (3) intergrowth of two layer types, such as a *I**a*** or *I**b*** layer that is partly reconstituted to a *I**b*** layer. Professor S. W. Bailey has tested stacking models and concluded that the last two interpretations are feasible (personal communication). No twinning models have been tested. The data indicate that the structure is severely distorted from that of the ideal *I**b*** polytype but additional study will be required to delineate the specific nature of the structural irregularity.

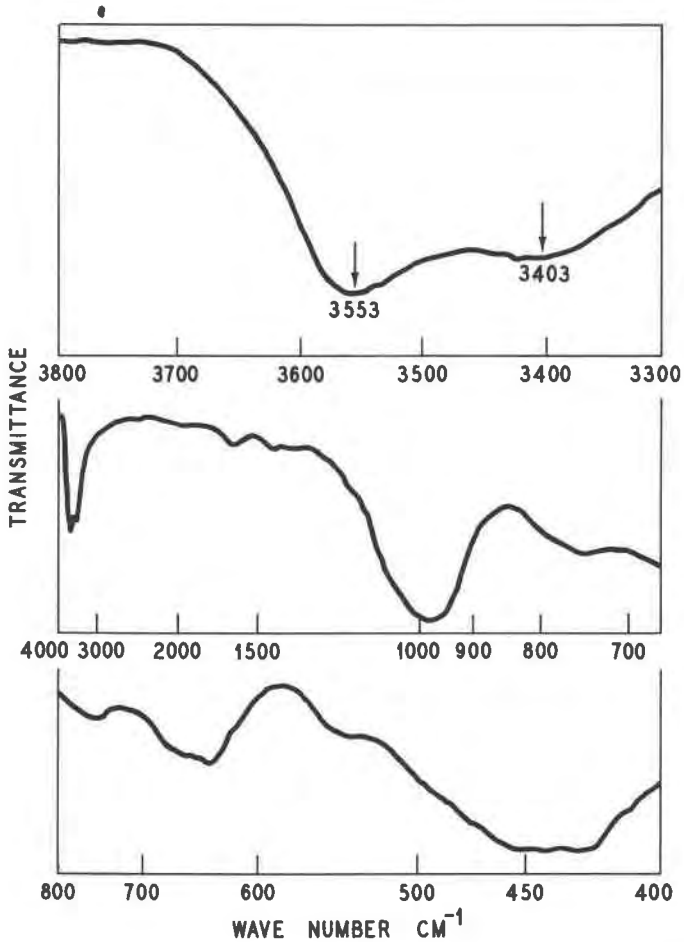


FIG. 6. Infrared absorption spectra of chlorite in the region of 4000–400  $\text{cm}^{-1}$ .

#### CHEMICAL COMPOSITION OF CHLORITE CRYSTALS

*Identification of Recrystallization Products.* An aliquot of crystals was gently ground by hand, heated in air for two hours at 1050°C, and the resultant crystalline phases identified using an X-ray powder camera. The presence of spinel, hematite, and an amorphous phase, probably silica, indicates that the chlorite is iron-rich (Shirozu, 1962). The identification of pseudobrookite among the recrystallization products is undoubtedly due to titaniferous inclusions.

*Cell Dimensions.* Several studies of chlorites have resulted in equations relating the  $b$  cell dimension to the octahedral Fe content and the  $d(001)$  basal spacing to the tetrahedral Al content (Brindley and Gillery, 1956; Brindley and McEwan, 1953; Hey, 1954; Radoslovich, 1962; Shirozu, 1958; see summary in Brown, 1961). Using values of  $b=9.28 \text{ \AA}$  and  $d(001)=14.16 \text{ \AA}$  obtained from the pattern of Table 2, the chlorite appears to be iron-rich, Table 3, which agrees with the results of the recrystallization run. Estimates of aluminum range from 1.0 to 1.4 atoms per 4 tetrahedral sites. Albee (1962) used values of  $d(001)$  to obtain an esti-

TABLE 3. ESTIMATES OF OCTAHEDRAL IRON AND TETRAHEDRAL ALUMINUM CONTENT OF THE CHLORITES BASED ON CELL DIMENSIONS.

| Fe Atoms<br>(per 6 octahedral sites) | Al Atoms<br>(per 4 tetrahedral sites) | Reference                    |
|--------------------------------------|---------------------------------------|------------------------------|
| 1.9                                  | 1.4                                   | Shirozu<br>(1958)            |
| 2.6                                  | —                                     | Radoslovich<br>(1962)        |
| 2.9                                  | 1.0                                   | Hey<br>(1954)                |
| 3.0                                  | —                                     | Brindley & MacEwan<br>(1953) |
| —                                    | 1.1                                   | Brindley & Gillery<br>(1956) |

mate of the total (tetrahedral plus octahedral) aluminum content of chlorite. Using his method, the crystal contains a total of 2.6 Al atoms per half unit-cell. Assuming a tetrahedral content of 1.2 atoms, the octahedral Al content would be 1.4 atoms per 6 octahedral sites.

*Optical Properties.* Refractive indices were measured on inclusion-free areas of the crystals with the petrographic microscope using fresh standard immersion oils and the Becke line method of Emmons and Gates (1948). The accuracy of this method is reported to be  $\pm .002$ . The  $\beta$  and  $\gamma$  indices of all crystals studied could not be differentiated and measured 1.627. Using a spindle stage an  $\alpha$  index of 1.626 was determined. Birefringence was approximately .002 (very weak),  $2V$  was estimated at 30 degrees or less, and with the exception of one crystal, the optic sign was negative. Using the graph of Albee (1962), the refractive indices

indicate that the chlorites contain about 3 Fe atoms per 6 octahedral sites.

*Infrared Absorption Analysis.* A KBr disk was prepared for analysis by adding 0.6 mg of chlorite crystals to 100 mg of KBr, gently hand grinding under alcohol to prevent alteration of the mineral (Tuddenham and Lyon, 1959), and pressing in an oil press at 10,000 lbs/in<sup>2</sup>. Absorption spectra were recorded using the Perkin Elmer Infracord and Model 137 spectrophotometers. To obtain high resolution spectra in the region of 3800–3300 cm<sup>-1</sup>, the disk was examined with the Cary Model 90 spectrophotometer.

The values of 3553 cm<sup>-1</sup> and 3403 cm<sup>-1</sup>, Figure 6, are closest to those of the most iron-rich chlorite studied by Hayashi and Oinuma (1967) which contained 2.9 Fe atoms per 6 octahedral sites. This result is consistent with the iron content determined from refractive indices and agrees well with the higher estimates of iron based on the *b* cell dimension. Hayashi and Oinuma (1965) also investigated the relation between the amount of octahedral Al, Mg+Mn, and Fe, and the absorption bands near 560–540 cm<sup>-1</sup>, 692–620 cm<sup>-1</sup>, and 765–744 cm<sup>-1</sup>. According to their graphs, the values of 549 cm<sup>-1</sup>, 640 cm<sup>-1</sup>, and 752 cm<sup>-1</sup> of Figure 6 indicate that the chlorite contains more octahedral Fe+Mg than Al. The band at 549 cm<sup>-1</sup> indicates an octahedral Al content of 1.2 atoms per 6 sites which agrees reasonably well with the estimate of 1.4 atoms based on cell dimensions.

A more precise measurement of tetrahedral Al will be required before it can be established whether the chlorite should be called a ripidolite or a brunsvigite, according to the classification of Foster (1962).

#### CHLORITE GENESIS

The following observations permit inferences to be made concerning the origin of the chlorite crystals:

- (1) The crystals are tiny, delicate and often euhedral suggesting that they have formed in place.
- (2) The crystals are localized on fault surfaces in the mudstones. Their absence elsewhere in mudstones suggests that they are not detrital.
- (3) Some of the larger crystals are bent as if they had been subjected to stress following their formation. However, the absence of broken or grossly deformed crystals and the presence of crystals in the calcite cement that heals fault zones suggest that crystal growth generally took place after fault movement ceased.

It appears that the chlorite crystals have grown by local isochemical metamorphism, all necessary elements being available in the host rock.

The mudstones consist of quartz, calcite, 2M dioctahedral mica and chlorite. Whether detrital chlorite, mica, or some other phase served as nuclei is problematical. No nuclei are identifiable as such.

It is difficult to deduce what temperature-pressure conditions existed in the fault zones, particularly in view of the fact that shearing stresses were involved. The minimum temperature of chlorite formation suggested from hydrothermal studies would be not less than 400°C (Yoder, 1952; Roy and Roy, 1955). However, the high iron content of the chlorite could be expected to cause a lowering of this value (Yoder and Eugster, 1954; Turnock, 1962). The best indicator, at least in a qualitative sense, of the temperature range that prevailed appears to be the epigenetic mineral assemblage. The results of synthesis studies suggest that the trioctahedral 1:1 phyllosilicate forms at low temperatures and converts to chlorite at high temperatures (Nelson and Roy, 1958; Gillery, 1959). Bailey and Brown (1962) indicate that *Ib* chlorite is more likely to occur in geologic environments of a lower energy level than the *IIb* polytype of chlorite-grade metamorphic rocks. Yoder and Eugster (1954) have shown that  $2M_1$  trioctahedral mica is either a stable low temperature form or a metastable phase that crystallizes early at a low temperature. Since the epigenetic minerals identified are *Ib* chlorite,  $2M_1$  trioctahedral mica, and a structurally distorted *IIb* chlorite, the mineral assemblage appears to be one that would crystallize in a low-to-intermediate temperature range.

#### CONCLUSIONS

1. Chlorite crystals found in Lower Devonian faulted mudstones formed by local isochemical metamorphism, all the necessary elements being available in the host rock.
2. Crystal growth apparently was initiated by faulting and continued, in a low-to-intermediate temperature range, after fault movement ceased.
3. The chlorite crystals contain abundant inclusions. X-ray and optical data indicate that most of the included material is chlorite residing in a chlorite host. No 1:1 phyllosilicate mineral was detected as either an interstratified or single phase. A small amount of the included material is titaniferous.
4. The composition of the chlorite crystals as determined by indirect methods is approximately  $(\text{Fe}_3\text{Al}_{1.2-1.4}\text{R})_6(\text{Al}_{1.2-1.4}\text{Si})_4\text{O}_{10}(\text{OH})_8$  where *R* signifies undetermined octahedral cations.
5. Single crystal study shows that the layer sequence of an apparent *IIb* chlorite is irregular and considerably distorted from that of the ideal *IIb* polytype.

## ACKNOWLEDGMENTS

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