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The Crystal Structure of Chloritoid*

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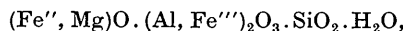
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The crystal structure of chloritoid, a layer silicate mineral, composition



has been determined by two-dimensional Patterson and Fourier syntheses from single-crystal X-ray data obtained with Cu $K\alpha$ and Mo $K\alpha$ radiation. The monoclinic unit cell has $a = 9.52 \pm 0.02$, $b = 5.47 \pm 0.01$, $c = 18.19 \pm 0.04$ Å, $\beta = 101^\circ 39' \pm 25'$, space group $C2/c$. The structure is discussed in relation to the micas and corundum; the twinning of the mineral and its infra-red absorption are also considered. The possibility of other forms of chloritoid is discussed.

Introduction

Chloritoid is a layer silicate mineral with the idealized composition $(\text{Fe}'' , \text{Mg})\text{O} \cdot (\text{Al}, \text{Fe}''')_2\text{O}_3 \cdot \text{SiO}_2 \cdot \text{H}_2\text{O}$. Although usually classified in the brittle mica group of which margarite $(\text{CaAl}_2(\text{Si}_2\text{Al}_2\text{O}_{10})(\text{OH})_2)$ is a typical member, it has a number of distinguishing features suggesting significant structural differences from the true micas. An approximate structure already briefly described (Brindley & Harrison, 1952, henceforth called Paper I) has now been refined by two-dimensional syntheses; the resulting structure is markedly different from that of muscovite.

The only structure analysis of chloritoid previously attempted was by Machatski & Mussgnug (1942) who gave the cell dimensions as $a = 5.4$, $b = 9.4$, $d(001) = 17.85$ Å, and on the basis of $00l$ reflexions proposed a layer structure similar to that of muscovite. This structure was not tested in detail and, as we have previously pointed out, is not confirmed by the present work. Milne (1949) determined the cell parameters, $a = 9.45$, $b = 5.48$, $c = 18.16$ Å, $\beta = 101^\circ 30'$, and gave the space group as Cc or $C2/c$, but he undertook no structure analysis. Hietanen (1951) examined particularly the optical and twinning properties and discussed the possibility of monoclinic and triclinic forms. The differences between Milne's cell parameters, which are confirmed by the present work, and those given by Machatski & Mussgnug may therefore be real; on the other hand, their choice of the axis of length ~ 9.4 Å as the monoclinic b axis may have been based on no more than a comparison with muscovite.

* The major part of this work was carried out whilst the authors were at the Department of Physics, The University of Leeds, England.

Experimental

Chloritoid from Point des Chats, Ile de Groix, Morbihan, France, (British Museum Cat. No. 1908, 362) yielded lamellar crystals suitable for single-crystal study by rotation and Weissenberg methods using Cu $K\alpha$ and Mo $K\alpha$ radiations. Attempts to grind spherical crystals by the method of Bond (1951) were unsuccessful owing to the easy basal cleavage, but with low air pressures crystals roughly ellipsoidal in shape were obtained which were used for a - and b -axis rotations. Lamellar crystals, circular in shape, were used for c -axis rotations. In the earlier work with Cu $K\alpha$ radiation absorption corrections were applied, but in the later work with Mo $K\alpha$ radiation the crystal size and shape rendered an absorption correction unnecessary. The reflected intensities determined by visual comparison methods were corrected for Lorentz and polarization factors. In all, 118 $0kl$, 215 $h0l$ and 90 $hk0$ reflexions were recorded. A one-dimensional $00l$ Fourier synthesis and two-dimensional $0kl$ and $h0l$ Fourier and Patterson syntheses were carried out. An analysis of the $h0l$ intensities agreed well with a centric (010) projection (Howells, Philips & Rogers, 1950). The infra-red absorption spectrum, recorded on a Perkin-Elmer spectrophotometer, provided additional evidence for certain features of the structure.

The unit cell and space group

The unit cell dimensions, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, are:

$$a = 9.52 \pm 0.02, \quad b = 5.47 \pm 0.01, \quad c = 18.19 \pm 0.04 \text{ Å}, \\ \beta = 101^\circ 39' \pm 25'.$$

Systematic absences of the type $h+k$ odd for general

hkl reflexions and $h0l$ with l odd, show the space group to be Cc or $C2/c$. Since the $h0l$ reflexions possess a centric distribution, the former possibility is eliminated.

As the available material was insufficient for chemical analysis, the unit-cell content was obtained from an analysis given by Milne (1949, analysis 5) of chloritoid from the same locality. The unit cell content in atoms is

Si 7.92, Al 15.08, Fe'' 6.96, Mg 1.20, OH 13.60,
O 40.00.

The structure determination

Atomic scattering factors appropriate to silicate structures given by Bragg & West (1928) were used. The scattering factor for Fe was reduced by 2 electron units for Cu $K\alpha$ radiation to correct for dispersion.

(a) The layer sequence normal to (001)

As stated in Paper I, it was impossible to obtain satisfactory agreement between observed and calculated values of $|F(00l)|$ on the basis of Machatski & Mussgnug's structure, but on the assumption that the signs of the calculated values were correct a one-dimensional 00l Fourier synthesis was calculated. A new atomic distribution resulted giving calculated values of $F(00l)$ in good agreement with observed values. The Fourier projection and atomic distribution obtained are compared with a similar projection for muscovite in Fig. 1.

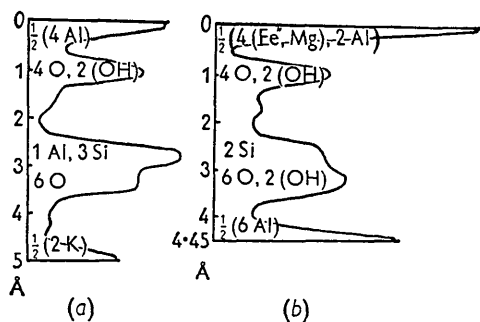


Fig. 1. One-dimensional Fourier projections on the normal to (001). (a) Muscovite; (b) chloritoid.

(b) The (010) projection

Observed values of $F^2(h0l)$ were used in computing a Patterson projection parallel to b , the shortest unit-cell dimension. The layer sequence found normal to (001), Fig. 1(b), enabled a partial identification of the vector peaks to be made. This, with the assumption that the cations at the origin of Fig. 1(b) and the layer of (O, OH) ions on either side of them form a normal octahedral arrangement as in the micas, was used to derive the structural arrangement shown in Fig. 1(a), Paper I, which conforms to the space group and agrees qualitatively with the Patterson projec-

tion. For details of this derivation see Harrison (1954, pp. 52-5). $h0l$ structure factors calculated from the arrangement made the residual discrepancy $R (= (\sum|F_o - F_c|) \div \sum|F_o|)$ equal to 0.32. Further confirmation was obtained from a Fourier projection on (010).

(c) Completion of the trial structure by the application of space-group limitations and coordination principles

In Fig. 1(a) Paper I, the layer denoted by L_1 resembles the normal trioctahedral arrangement found, for example, in brucite, $Mg(OH)_2$, where the Mg ions occupy interstices between two close-packed layers of (OH) ions. The arrangement of layer L_2 , however, in which Al ions lie between oxygens at the bases of SiO_4 tetrahedra, appears to be new. The relation of this structure to that of mica can be described as follows. Chloritoid contains only half as many Si atoms as there are (Si, Al) atoms in the micas. If alternate (Si, Al) atoms are removed from the silicate layer of mica, the arrangement shown in Fig. 2(a) is

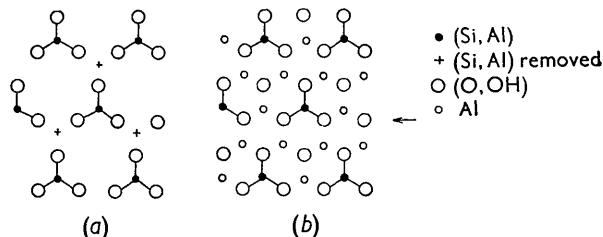


Fig. 2. (a) Removal of alternate (Si, Al) atoms from the silicate layer of mica type. (b) An arrangement of Al atoms which links together the (SiO_4) groups and (O, OH)'s.

obtained. If (O, OH) ions are placed in the centres of the hexagonal holes of the oxygen layers and Al atoms are located in the layer occupied by K atoms in mica, then the continuous Al-(O, OH) layer is obtained which is shown in Fig. 2(b). When this layer is viewed in the direction of the arrow, it is seen to comply with the (010) projection in Fig. 1(a), Paper I. Comparison of the (010) projection of space group $C2/c$ with that of the structure requires centres of symmetry in the layer L_1 and twofold rotation and screw axes in the layer L_2 . The limitations imposed by these symmetry elements enable the structure of the whole unit cell to be defined uniquely. Full details of this procedure are given in Harrison (1954, pp. 55-8). The idealized arrangement viewed along the a axis is shown in Fig. 1(b), Paper I. The $hk0$ structure factors calculated from this arrangement, when compared with observed values, made the residual discrepancy R equal to 0.31.

In Fig. 1, Paper I, some anions have been labelled O and others (OH) on the basis of a consideration of the electrostatic valencies. Anions in layer L_1 not attached to Si appear to be (OH) ions whilst those in layer L_2 are O ions. If, as is suggested in the discussion

which follows, a hydrogen bond occurs between an (OH) ion in L_1 and the O ion directly above or below it in L_2 , with H more closely attached to the ion nominally called (OH), then the criterion of Pauling (1948) that in natural minerals no ion is unsatisfied by more than one-sixth of an electron unit, is fulfilled,

Refinement of the trial structure

The structure was refined by $h0l$ and $0kl$ Fourier syntheses, using structure factors obtained from measurements with Mo $K\alpha$ radiation.

After two $h0l$ syntheses the value of R for the $h0l$ structure factors decreased to 0.213 when reflexions too weak to be observed were given a value equal to half the minimum observable value, and 0.201 when they were neglected. After one refinement the corresponding values for the $0kl$ structure factors were 0.170 and 0.143. For the hko structure factors they were 0.30 and 0.25.

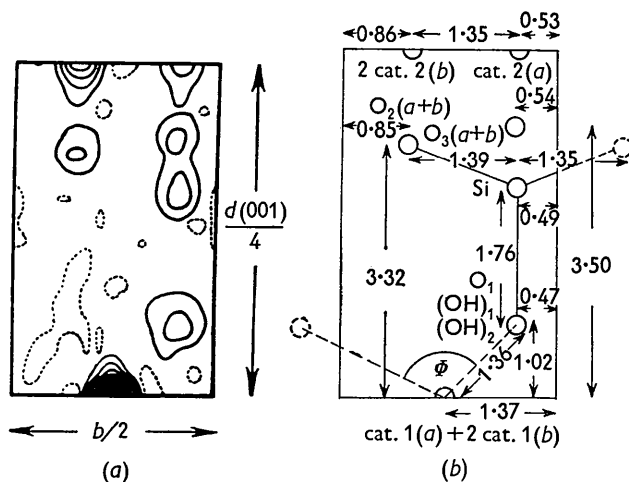


Fig. 3. (a) The $0kl$ Fourier synthesis (contours at equal but arbitrary intervals; zero contour broken). (b) Interatomic distances in the $0kl$ Fourier synthesis.

The final $0kl$ and $h0l$ syntheses are shown in Figs. 3(a) and 4(a) and the principal interatomic distances in Figs. 3(b) and 4(b). A comparison of the peak heights of the cations denoted (1) and (2) shows the latter to be primarily Al, the former the remaining Fe'', Mg and Al. Peaks (1) are not of uniform height, indicating a preference of Fe'' for particular positions, and have been divided into two types, (a) and (b); the atomic compositions fitting the relative peak heights are given in Table 1. The final calculated structure factors are

Table 1. Final atomic parameters expressed in terms of the Wyckoff notation

Coordinates of equivalent positions $(0, 0, 0; \frac{1}{2}, \frac{1}{2}, 0)$

- 4: Special cation (1a) (c) $\frac{1}{2}, \frac{1}{2}, 0; \frac{1}{2}, \frac{1}{2}, \frac{1}{2}$.
 cation (2a) (e) $0, y, \frac{1}{2}; 0, \bar{y}, \frac{1}{2}$,
 where $y = 0.403_6$.
- 8: General (f) $x, y, z; \bar{x}, \bar{y}, \bar{z}; \bar{x}, y, \frac{1}{2}-z; x, \bar{y}, \frac{1}{2}+z$,
 where x, y, z for the atoms are:

Atoms	x	y	z
Cation (1b)	0.085 ₅	0.750 ₆	0.000 ₆
O ₁	0.069 ₅	0.085 ₃	-0.065 ₉
0.85(OH) ₁	0.105 ₆	0.585 ₃	-0.050 ₀
0.85(OH) ₂	0.233 ₀	0.585 ₃	-0.052 ₀
Si	0.033 ₀	0.090 ₃	-0.156 ₀
O _{2a}	0.105 ₀	0.347 ₅	-0.186 ₁
O _{2b}	0.105 ₀	0.847 ₅	-0.186 ₁
O _{3a}	-0.143 ₂	0.098 ₁	-0.196 ₆
O _{3b}	-0.143 ₂	0.598 ₁	-0.196 ₆
Cation (2b)	0.250 ₀	0.342 ₁	-0.250 ₀

where cation (1a) = 0.75 Al + 0.25 Fe'';
 cation (1b) = $\frac{1}{2}$ (0.3 Mg + 1.5 Fe'');
 cation (2a) = cation (2b) = Al.

compared with the observed values in Harrison (1954, pp. 141-9).

Some estimate of the accuracy of the atomic positions is possible by comparing the z coordinates obtained from the a - and b -axis projections respectively. Errors of approximately 0.01 Å are suggested for the atomic positions corresponding to the larger peaks (the cations and oxygen atoms O₂ and O₃) and of the

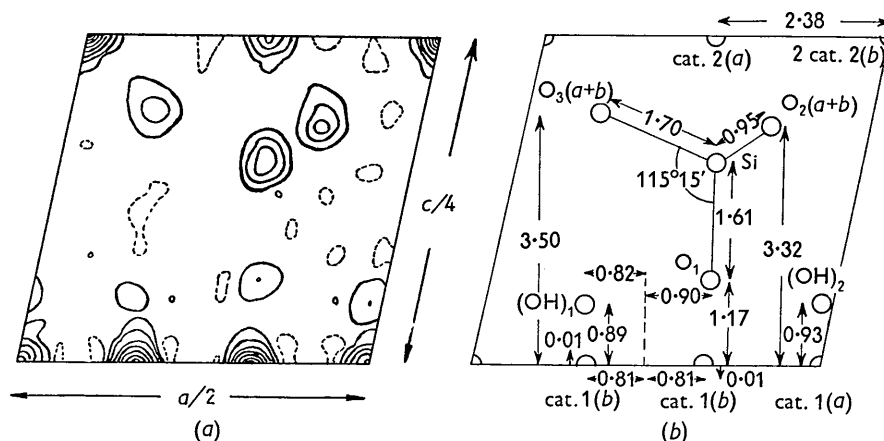


Fig. 4. (a) Final $h0l$ Fourier synthesis (contours at equal but arbitrary intervals; zero contour broken). (b) Interatomic distances in the $h0l$ Fourier synthesis.

order of 0.04 Å for those of the smaller peaks (O₁, (OH)₁ and (OH)₂) which are completely resolved in the *b*-axis projection.

Discussion

(a) The SiO₄ tetrahedron

The overlapping of oxygen peaks in the *a*- and *b*-axis projections, Figs. 3 and 4, makes the determination of the exact shape and size of the SiO₄ group somewhat uncertain. In Fig. 4(b), Si-O₁ = 1.61 Å, in good agreement with the values found in other silicates (see Smith, 1954). Its direction is at right angles to the *a* axis. The Si-O_{3(a+b)} distance of 1.70 Å is large, while the angle O₁-Si-O_{3(a+b)} = 115° 15' is larger than the tetrahedral angle of 109° 28'. However, only one of the atoms O₃ is attached to Si, the other lying at the centre of a hexagonal hole bordered by O atoms attached to Si atoms. If one assumes that O_{3a} is situated normally with respect to Si (i.e. at a distance 1.61 Å and making O₁-Si-O_{3a} = 109° 28'), then O_{3b} must be symmetrically placed with respect to the centre of the composite peak. The resultant positions of O_{3a} and O_{3b} are shown in Fig. 5. The angles α and γ, Fig. 5,

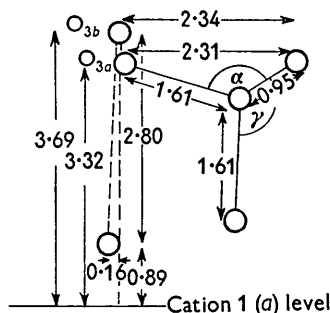


Fig. 5. Interatomic distances associated with the (SiO₄) tetrahedron and hydrogen bond.

(b) The hydrogen bond

To fulfil the Pauling criterion for electrostatic valencies the occurrence of a hydrogen bond between the anions O_{3b} and (OH)₁ was suggested previously. Since the anions each have as nearest neighbours three cations of small radius and charge +2 or +3, the occurrence of a hydrogen bond would be expected, due to the polarizing effect of the cations as described by Bernal & Megaw (1935). In Fig. 5 anion O_{3b} is almost vertically above (OH)₁, being offset from the vertical by 3° 18' ± 1°. The O_{3b}-(OH)₁ distance of 2.80 ± 0.05 Å agrees with the occurrence of a hydrogen bond, since the two anions would hardly approach each other so closely unless they were so linked.

(c) The brucite-type layer

Consideration of the interatomic distances and angles in this layer shows that the octahedral arrangement of atoms is far from regular. The angle Φ, Fig. 3(b), which for a regular octahedron is 90°, in fact is 102° 40' ± 4°. The distance between cation 1(a+2(1b)) and (O₁+(OH)₁+(OH)₂), Fig. 3(b), in a regular octahedral arrangement would equal the mean separation between cations 1(a) and 1(b), Fig. 4(b), differs considerably from it, the two being equal to 1.36 ± 0.04 and 1.59 ± 0.02 Å respectively.

As already mentioned, the peak heights of the final *h*0*l* synthesis indicate that the majority of the Fe^{II} enters the cation 1(b) positions whilst the 1(a) position is filled primarily by Al. A possible explanation for this is that the surrounding six anions for each position are arranged differently in the two cases, the two anions O₁ being adjacent in the two cases, the two anions O₁ being adjacent in the two cases, the two anions O₁ being further from the cation sheet than (OH)₁ and (OH)₂. In such a case, one would expect the smaller cation to prefer the smaller interstice.

In the *b*-axis projection, Fig. 4(b), the cations 1(b) depart from the ideal positions by 0.02 Å in the *a*

126° 15' and 124° 17' respectively, agree well with the value 125° 16' for a regular tetrahedron.

In the *a*-axis projection, Fig. 3(b), the composite peak representing O₁, (OH)₁ and (OH)₂ is 1.76 Å from the Si peak. The projections of the Si-O_{2(a+b)} bonds, namely 1.39 ± 0.02 Å and 1.35 ± 0.02 Å, are not significantly different, and when combined with the projected value 0.95 Å shown in Fig. 4(b) yield Si-O₂ bond distances of 1.68 ± 0.03 Å and 1.65 ± 0.03 Å respectively, and a bond angle of 110° 12' ± 2°. These distances are significantly larger than the value 1.61 Å. However, since oxygens (a) and (b) forming O_{2(a+b)} are not required to be coincident in the *a*-axis projection, they may be displaced by small equal and opposite amounts in the *b* direction, retaining the accepted value from their associated Si atoms, whilst combining to give the unresolved peak of the *a*-axis projection.

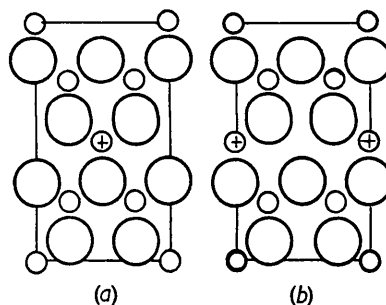


Fig. 6. The Al-O sheet: (a) in chloritoid; (b) in corundum.

direction and 0.01 Å perpendicular to it. It is doubtful whether these small shifts are significant.

(d) The Al-O layer

The arrangement of Al atoms in the close-packed O layer of chloritoid (Fig. 6*a*) is very similar to that found in corundum, Al_2O_3 (Fig. 6*b*). The only difference lies in the alternative positions of the Al atom marked by a cross. The repeat dimensions of the Al-O layer in corundum (9.50 Å and 5.48 Å, Bragg, 1937, p. 94) correspond closely with the dimensions for chloritoid ($a = 9.52$ Å, $b = 5.47$ Å).

The infra-red absorption spectrum

The infra-red absorption spectrum of chloritoid (Fig. 7) shows the sharp band at a wavelength just less than 3μ associated with hydroxyl ions and the composite band between 10 and 12μ generally associated with the silicon-oxygen arrangement. The wavelength and shape of the absorption band associated with (OH) in many substances appear to depend on the degree of interaction between the (OH) ion and neighbouring O and (OH) ions. When there is no interaction, the band is sharp and occurs at 2.75μ . With an increase in bonding and a resultant decrease in the distance between the neighbouring ions, the wavelength in-

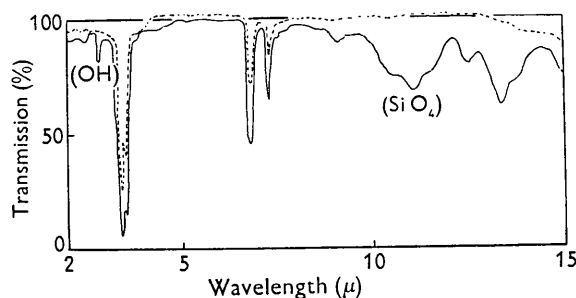


Fig. 7. Infra-red absorption spectrum of chloritoid. Full line: chloritoid; broken line: mineral oil suspensand.

creases and the band may broaden (cf. Rundle & Parasol, 1952). The absorption band for chloritoid at 2.86μ corresponds on Rundle & Parasol's classification with an oxygen separation of 2.85 Å, compared with the value of 2.80 ± 0.05 Å obtained from the structure investigation.

Launer (1952) examined the infra-red absorption of a variety of silicates, and attempted a correlation between the wavelength and width of the composite 9– 12μ band and the type of silicate structure. He finds that the region of strongest absorption tends to shift towards shorter wavelength as the ratio of silicon to oxygen increases. The absorption band for chloritoid from 10 to 12μ is of the type which Launer associates with isolated (SiO_4) groups.

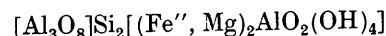
Comparison of chloritoid with mica minerals

Though the structure of chloritoid was approached from the standpoint of a mica, it should be more appropriately considered as an alternation of layers of

brucite-type and corundum-type, linked by Si atoms in fourfold coordination and by hydrogen bonds. The ideal structural formula may be written



to show the SiO_4 groups, or alternatively



to demonstrate the oxide and hydroxide layers linked by Si atoms.

The increase in the values of a and b for chloritoid when compared with mica-type minerals of similar Fe'' content, e.g. biotite ($b = 9.25$ Å (Walker, 1949)) and bavalite ($b = 9.35$ Å (Engelhardt, 1942)), is now explained, since whereas in mica the continuous hexagonal silicate layers set the scale of the dimensions, in chloritoid, where distinct (SiO_4) tetrahedra replace the continuous $(\text{Si}_4\text{O}_{10})$ layers, the values of a and b are governed by the corundum-type layers.

The decrease in c for chloritoid when compared with that of the micas ($c = 20.04$ Å (Jackson & West, 1930)), follows from the close-packing of the O atoms at the bases of the (SiO_4) tetrahedra resulting in spaces with octahedral coordination in contrast to the direct superposition of O atoms in muscovite giving spaces containing K atoms with 12-fold coordination.

Chloritoid is particularly distinguished from the mica minerals by its high hardness and difficult basal cleavage. These differences can be related to the structures, for in mica the talc-like layers are bound by comparatively weak electrostatic forces to the K ions, which in chloritoid are replaced by the strongly bound corundum layer. It cannot be expected that chloritoid will cleave at this level. The bonding forces of the brucite-type layer are weaker, but the cleavage most probably occurs between the brucite and corundum-type layers, where there are only two Si-O bonds plus two H-bonds per unit cell level.

Twinning in chloritoid

The twinning observed in chloritoid is always lamellar with the composition plane parallel to the basal cleavage.

Theoretically all twinning laws are possible which maintain an arrangement in the immediate neighbourhood of any atom close to the composition plane essentially similar to that which would occur if normal growth took place. Thus the composition plane in chloritoid is limited to a position where in the untwinned structure the sequence of atomic sheets on either side of the composition plane is the same, i.e. it must lie at the level of the cations in the brucite-type layer or the corundum-type layer.

Consideration of the former position shows that only three directions are permissible as twin axes, $[100]$, $[130]$ and $[\bar{1}30]$, whilst in the latter $[010]$, $[110]$ and $[\bar{1}\bar{1}0]$ are the only possibilities. The $[130]$ and $[\bar{1}30]$ axes are related by the space-group symmetry, as are

[110] and $[\bar{1}\bar{1}0]$, whilst [010], being the twofold axis of the space group, reproduces the crystal without twinning. The unique twin axes are therefore [100], [130] and [110].

All these have been observed experimentally, [110] by Barth & Balk (1934), [130] by Milne (1949) and Hietanen (1951), all in optical measurements, and [100] in the present work. In addition, Hietanen lists three further axes, [120], [210] and [310], the result of optical measurements on chloritoid from Rawlinsville, Lancaster Co., Pennsylvania, U.S.A., which, if correct, suggest the existence of at least one other structural form of chloritoid.

We are indebted to Dr F. A. Bannister of the British Museum for supplying the sample of chloritoid on which this investigation was made, to Dr H.L. Lovell of the College of Mineral Industries, The Pennsylvania State University, U.S.A., for recording the infra-red absorption spectrum, and to Dr W. H. Taylor of the Cavendish Laboratory, Cambridge, England, who made available computational facilities for the final (*okl*) synthesis.

One of us (F. W. H.) wishes to acknowledge the receipt of a maintenance grant from the Department of Scientific and Industrial Research, during the tenure of which this work was carried out.

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Short Communications

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 500 words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible; and proofs will not generally be submitted to authors. Publication will be quicker if the contributions are without illustrations.

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An analogue computer for double Fourier series summation for X-ray crystal-structure analysis. By G. SURYAN, *Department of Physics, Indian Institute of Science, Bangalore 3, India*

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The need for computational aids in the determination of crystal structures by means of X-ray diffraction studies has been keenly felt by all workers in the field and probably there are as many devices as there are workers, with varying degrees of accuracy, usefulness, complexity and cost. Of these the X-RAC machine built by Pepinsky (1952) has generally been acclaimed the best and would have found wide application but for its expense. It is the object of this note to show that a system which is relatively much less expensive than the X-RAC machine could be devised, based on the general principles of the synchronous magnetic recorder devised by the author (Suryan, 1950, 1953*a*, *b*), without any particular loss in performance.

The problem is to find the sum $\varrho(x, y)$ of a double Fourier series of given coefficients F_{hk} and phases α_{hk} of the form $\sum \sum F_{hk} \cos/\sin 2\pi(hx/a + \alpha_{hk}) \cos/\sin 2\pi(ky/b)$

over the domain $x = 0$ to $x = a$ and $y = 0$ to $y = b$ at close enough intervals, and to present the results preferably in the form of a contour map of the function $\varrho(x, y)$. Electrical sine waves are the most suitable analogues to the cos/sin functions, and the analogy can be effected by the transformation of the x, y domain into time by means of two transformations $x/a = pt$ and $y/b = p't$ ($p \gg p'$), corresponding to a fast and slow scan of the x, y domain respectively. Then one set of harmonically related electrical waves may represent the cos/sin $2\pi(hx/a)$ and a set of cosine resolvers suitably ganged through a gearbox may perform the function of generating the second set cos/sin $2\pi(ky/b)$. To utilize these analogues in their most primitive form one would require a large number of oscillators and much larger number of amplitude controls and a multitude of adding amplifiers etc., all going to make the equipment prohibitively expensive