values returned (Table 7) very nearly to those obtained at the end of the previous cycle.

There appeared to be no good reason to carry the calculations further. All the results indicated that there are probably few atoms in the c sites and their exclusion from the calculation is not the main cause of the negative thermal parameters. Examination of Table 3 now indicated that the source of the difficulty was error in the observed data. There are a number of calculated amplitudes which are considerably larger than the observed which lead to a larger factor by which the observed amplitudes are multiplied to put them on an absolute scale. This, however, makes a sizable number of the observed amplitudes especially at high angles too large relative to the calculated amplitudes which in turn tends to reduce the thermal parameters. When the *true* values of these parameters are small, the calculations can, as in this case, produce negative values. This case shows also that the scale and thermal parameters tend to absorb measurement errors. Because as stated earlier, the correlations between thermal or scale and positional parameters are small it is probable that the effect on the positional parameters is small.

Finally we give the X-ray density of the compound with ideal composition $Pd_{17}Se_{15}$, 8·34 g.cm.$^{-3}$.

The IBM 704 programs used in the calculations other than those of the least squares refinement were those of Dr R. G. Treuting. The drawing of the structure (Fig. 2) was made by Mr H. J. Seubert.

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The Crystal Structure of Yoderite

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Yoderite is a hydrous magnesium iron aluminosilicate discovered by D. McKie in 1956. The approximate empirical formula is $[Mg_{2.0}Ca_{0.2}(Fe''Fe''')_{0.5}Al_{5.3}]Si_4O_{17.6}(OH)_{2.4}$. There are weak subsidiary reflections with unitary structure factors ≤ 0.03 (McKie, 1959). The average structure, ignoring these, has been determined and refined by two-dimensional Fourier methods. It is monoclinic with space group $P2_1/m$ and cell dimensions

$$a = 8·035 \pm 0·003, \quad b = 5·805 \pm 0·001, \quad c = 7·346 \pm 0·002 \text{ Å}; \quad \beta = 105° 38' \pm 4'.$$

Chains of AO_6 octahedra, sharing edges, run parallel to the y-axis, linked by isolated SiO_4 tetrahedra and AO_5 trigonal bipyramids in which A sites are mainly occupied by Al and Mg. The packing of oxygen atoms resembles (but is not identical with) that in kyanite with which the mineral intergrows. Two of the 2·4 hydrogen atoms per unit cell have been located. Consideration of the causes of the subsidiary reflections is left to a later paper.

Introduction and summary of previous work

Yoderite is a hydrous magnesium iron aluminosilicate of composition $Mg_2Al_{5.3}Fe_{0.5}Ca_{0.2}Si_4O_{20}H_{2.4}$. It was

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discovered by McKie in 1956 occurring in a quartz-yoderite-kyanite-talc schist in grains containing relicts of kyanite in a fixed orientation. McKie (1959) made an X-ray study of the mineral and showed that it had a primitive monoclinic lattice with cell dimensions similar to those of the triclinic kyanite lattice. The

Table 1. *Cell dimensions of yoderite,*
and comparison with kyanite

(1) Yoderite	(2) Kyanite	Angles between (1) and (2)
$a = 8 \cdot 035 \pm 0 \cdot 003$ Å†	$b = 7 \cdot 72$ Å	$0°$
$b = 5 \cdot 805 \pm 0 \cdot 001$	$c = 5 \cdot 56$	$0°$
$c = 7 \cdot 346 \pm 0 \cdot 002$	$a = 7 \cdot 09$	$11° \, 2'$
$\beta = 105° \, 38' \pm 4'$		

Volume of cell $= 329 \cdot 6$ Å^3

Yoderite cell content is $Mg_{2 \cdot 0}Al_{5 \cdot 3}Fe_{0 \cdot 5}Ca_{0 \cdot 2}Si_4O_{20}H_2$; kyanite cell content is $4(Al_2SiO_5)$.

† Errors are limits estimated from experimental graphs of $\sin^2 \theta$ against a^*, b^*, c^*, and $\cos \beta^*$ calculated from reflections with different θ values.

cell dimensions of yoderite determined in the present work are in agreement with McKie's, and are compared with those of kyanite (Naray Szabo, Taylor & Jackson, 1929) in Table 1. Fig. 1 shows the relative orientation of the two lattices in intergrowths. The y axis of yoderite ($b = 5 \cdot 8$ Å) corresponds to the z axis of kyanite ($c = 5 \cdot 6$ Å), which represents the length of the two AlO_6 octahedra sharing a common edge. For this reason, McKie suggested that yoderite, like kyanite, should be made up of chains of octahedra sharing edges, linked by SiO_4 tetrahedra and AlO_6 octahedra.
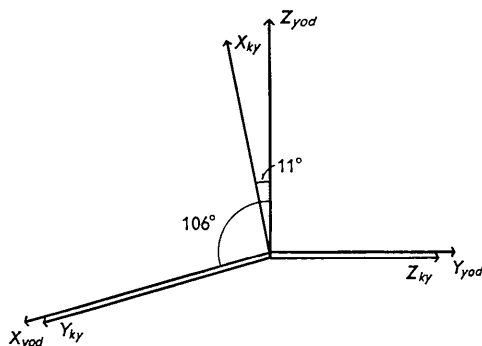

Fig. 1. Axial directions of kyanite and yoderite drawn to show the relative orientation of the two lattices in intergrowth. $X_{ky}Z_{yod}Z_{ky}Y_{yod}$ are coplanar.

McKie observed extra X-ray reflections between main layer lines on heavily exposed y- and z-axis oscillation photographs, resembling the reflections given by intermediate plagioclase felspars. These reflections are all very weak (unitary structure factors $\leq 0 \cdot 3$) and they occur in positions which may be described by indices subject to one of the following restrictions:

$$h \text{ integral, } k = n \pm 0 \cdot 16, \, l \text{ integral}$$

or

$$h \text{ integral, } k = m \pm 0 \cdot 42, \, l = p \pm 0 \cdot 5$$

where n, m and p are integers.

It was decided to begin by neglecting the subsidiary reflections and to determine the 'average' structure using main X-ray reflections only. The

existence of subsidiary reflections suggested a larger true cell, characterized by small regularly recurring deviations from an average arrangement of atoms with the small unit cell defined above. Fourier syntheses, omitting the weak reflections, should then show a superposition of the electron densities of all the varieties of subcell, and could be considered to be syntheses of the 'average' structure. This 'average' structure is described here, while the interpretation of the weak reflections in terms of detailed differences between subcells will be left to a later paper.

Description of material used

A sample of selected small grains of yoderite was kindly provided by Mr McKie. The results of a chemical analysis are given in Table 2. The approximate formula, in the form useful for the present paper, is $A_8Si_4O_{20}H_2$, where A represents the average of all the divalent and trivalent cations. Regularly shaped single crystals approximately $0 \cdot 15$ mm. in all dimensions were chosen to give the best compromise between the conflicting demands for small absorption, small secondary extinction and strong reflection intensity.

Table 2. *Composition of yoderite*
(Analysis by A. J. Radford, quoted by McKie (1959))

Ion	No. in formula unit	Estimated error	Symbol used in paper
Mg^{2+}	$2 \cdot 02$	$0 \cdot 02$	A
Al^{3+}	$5 \cdot 35$	$0 \cdot 02$	A
$Fe^{2+}Fe^{3+}$	$0 \cdot 49$	$0 \cdot 03$	A
Ca^{2+}	$0 \cdot 17$	$0 \cdot 02$	A
Si^{4+}	$4 \cdot 00$	$0 \cdot 02$	Si
O^{2-}	$17 \cdot 64$	$0 \cdot 02$	O
OH^-	$2 \cdot 36$	$0 \cdot 02$	O

Others, $Ti^{4+}Mn^{2+}$ etc.

Space group

Yoderite has Laue symmetry $2/m$ and the only systematic absences are for $0k0$ reflections with k odd. The space-group is thus $P2_1$ or $P2_1/m$. To decide on the presence or absence of a centre of symmetry, reflections in the $hk0$ and $0kl$ zones were subjected to the statistical test suggested by Ramachandran & Srinivasan (1959). Using reflections with $\sin \theta/\lambda \leq 1 \cdot 00$, the values of their test ratio N_2/N_1 were $0 \cdot 58$ and $0 \cdot 39$ for $hk0$ and $0kl$ projections respectively, as compared with the theoretical values of $0 \cdot 776$ for a centro-symmetric structure and $1 \cdot 96$ for a non-centro-symmetric structure. Thus the projections are centro-symmetric. As an additional check, crystals were tested for pyro- and piezo-electric effects, with negative results. The centro-symmetric space group $P2_1/m$ was assumed thereafter, and later refinement of the structure confirmed this assumption.

Collection of intensities

X-ray reflections $hk0$, $h0l–h6l$, $0kl–4kl$, were recorded on Weissenberg photographs with filtered Mo $K\alpha$

radiation; the multiple-film technique (Robertson, 1943) was used. Intensities recorded on the film were measured visually by comparison with an intensity scale, made using the well-shaped 040 reflection from the crystal.

Corrections and errors

The intensities were corrected for Lorentz and polarization effects and for absorption, using programs written by S. Darlow and M. Wells for EDSAC II. No attempt was made to correct for extinction or the measuring error introduced in the intensities by the increasing resolution of the $\alpha_1\alpha_2$ doublet with sin θ. Some tests using F_c instead of F_o for strong low-angle reflections suggested that the effect of extinction on coordinates was negligible within the accuracy here aimed at. After the average temperature factor had been determined, F_o values were scaled to F_c by a scaling factor independent of θ. One set of films was remeasured after an interval of some weeks and showed that intensity measurements were reproducible to about 8%. Values of F_o and F_c are recorded (Fleet, 1962) and can be made available on request.

Deduction of trial structure

The volume per oxygen atom in yoderite is about 16·4 Å³, which suggested an approximately close-packed oxygen array. (In kyanite, with cubic-close-packed oxygens, the volume is 14·2 Å³ per oxygen.) Evidence from strong reflections and from the systematic weakness of $0kl$ reflections with k odd, used together with packing considerations, led to two possible oxygen packing arrays. An $hk0$ Patterson

synthesis showed peaks corresponding to a chain of octahedra running parallel to the diad axis with the shared edges approximately parallel to z^*: the relevant vectors are indicated on the Patterson diagram in Fig. 2. A successful trial structure made up of such chains, linked by SiO_4 tetrahedra and AO_5 trigonal bipyramids, could be fitted using one of the oxygen arrays. This trial structure is shown below in Fig. 3 with the corresponding projection of kyanite alongside
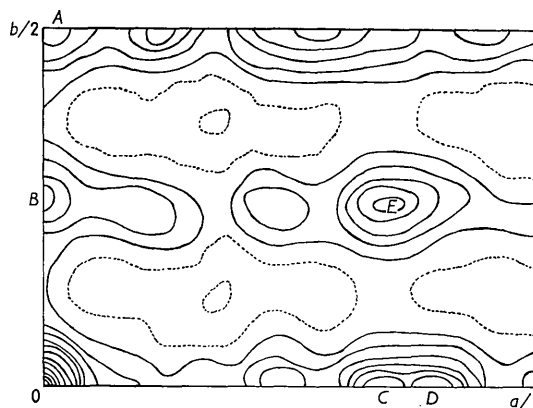

Fig. 2. $hk0$ Patterson projection of yoderite. Peaks represent the following vectors:

A: vector between two adjacent octahedra in a chain;

B: vector between an A_1 atom in an octahedral site and the oxygen of the shared edges in its own chain;

C: vector between an A_1 atom and one of the oxygens at the vertex of its octahedron;

D: vector between an A_1 atom in one chain and the nearest A atom in the adjacent chain;

E: vector between an A_1 atom and oxygens in a shared edge of the other octahedral chain.

Projection $\perp_r$ to [010]
Yoderite

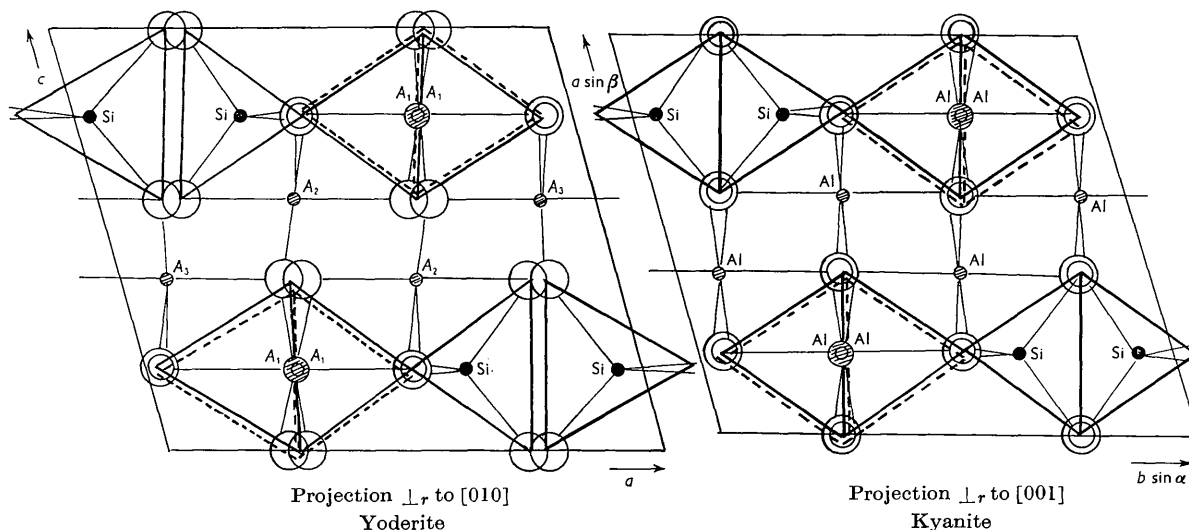Projection $\perp_r$ to [001]
Kyanite

Fig. 3. Idealized projection of yoderite $\perp_r$ to [010] with the corresponding projection of kyanite alongside for comparison. The polyhedra outlined by heavy and dashed lines form slabs of structure (extending indefinitely perpendicular to the paper and to right and left) which are almost identical in the two materials; but in kyanite the continuity of the oxygen close-packing is maintained from one slab to the next, while in yoderite one slab is displaced relative to the next by $\frac{1}{4}b_{yod}$ or $\frac{1}{4}c_{ky}$ from this position.

for comparison. It gave good agreement between observed and calculated structure factors.

Refinement

Initially, refinement was carried out by means of $F_o - F_c$ syntheses for $hk0$, $h0l$, $0kl$ reflections (Lipson & Cochran, 1953). When calculating the scattering factors, it was assumed that all atoms were half-ionized and that the A cations were average atoms of composition $\frac{1}{8}(5 \cdot 3f_{Al} + 2 \cdot 0f_{Mg} + 0 \cdot 5f_{Fe} + 0 \cdot 2f_{Ca})$. Scattering curves for these were constructed by interpolation from those in the literature quoted by Forsyth & Wells (1959), and an expression of the form $f(x) = A \exp(-ax^2) + B \exp(-bx^2) + C$ was fitted to them by the least-squares process they suggested. The constants so found are given in Table 3. Isotropic temperature factors were applied to the different classes of atom, and F_o values were scaled to F_c values using one scaling factor only. Finally, a number of cycles of refinement with full three-dimensional data were carried out on EDSAC II using a differential Fourier program written by Wells (1961).

Table 3. Scattering factor constants calculated for half-ionized atoms

(After Forsyth & Wells, 1959)

Half-ionized atom	A	a	B	b	C
O^-	4·322	6·983	3·243	36·48	1·434
Si^{2+}	7·616	2·857	2·252	63·64	2·132
A ion	7·689	2·699	3·602	6·730	1·229

The coordinates of the atoms in the 'average' structure at the final stage of refinement are shown in Table 4 and the values of the isotropic 'temperature' factors in Table 5. Final R-factors, and the total number of observed reflections are given in Table 6. Difference maps showed anisotropic regions

Table 4. Atomic coordinates of yoderite

(average structure)

The atoms lie in one of two positions:
(1) 2-fold special position $(x, \frac{1}{4}, z)$, $(\bar{x}, \frac{3}{4}, \bar{z})$
(2) 4-fold general position
   (x, y, z), $(\bar{x}, \bar{y}, \bar{z})$, $(\bar{x}, \frac{1}{2} + y, \bar{z})$, $(x, \frac{1}{2} - y, z)$

Atom	No. of related sites	Fractional coordinate x	y	z
Si_1	2	0·0624	$\frac{1}{4}$	0·7979
Si_2	2	0·3397	$\frac{3}{4}$	0·8026
A_1	4	0·2951	0·0054	0·1779
A_2	2	0·3894	$\frac{1}{4}$	0·6297
A_3	2	0·0554	$\frac{1}{4}$	0·3547
O_1	4	0·0571	0·9817	0·2162
O_2	4	0·4545	0·9770	0·7818
O_3	2	0·1455	$\frac{1}{4}$	0·6161
O_4	2	0·2206	$\frac{1}{4}$	0·9907
O_5	2	0·2988	$\frac{1}{4}$	0·3607
O_6	2	0·1686	$\frac{3}{4}$	0·6338
O_7	2	0·2818	$\frac{3}{4}$	0·0037
O_8	2	0·3633	$\frac{3}{4}$	0·3631

Table 5. Isotropic 'temperature' factors

(broadening factors)

Atoms	B
Si_1, Si_2	0·23 Å²
A_1, A_2, A_3	0·21
O_{1-8}	0·84

Table 6. Degree of refinement achieved

Type of reflection	No. of independent reflections measured	Final R factors
$hk0$	101	9·9%
$h0l$	197	11·1%
$0kl$	73	13·2%
hkl	1116	11·1%

near some atoms giving B values as high as 1·10 Å² for the major axis of the temperature ellipsoid, but no attempt has been made to apply anisotropic 'temperature' factors in refinement. It must be emphasized that empirically determined 'temperature' factors, whether isotropic or anisotropic, are in fact only broadening factors, and one cannot distinguish from this evidence how far they are due to thermal vibrations and how far to disorder or to disregarded kinds of order.

Errors

Values of standard deviations of coordinates were calculated at the end of the refinement using the formula:

$$\sigma(x_n) = \left\{ \overline{\left(\frac{\partial(\varrho_o - \varrho_c)}{\partial x} \right)^2} \right\}^{\frac{1}{2}} \Big/ C_n$$

(Cochran, 1951); it was assumed that $\sigma(x_n)$ was the same in every direction, and C_n (the curvature at the atomic peak) was estimated from the postulated scattering factor curves modified by 'temperature' factors. The values for C_n and $\sigma(x_n)$ are recorded in Table 7. It should be stressed that the model of electron-density distribution used to calculate the curvature is not identical with that believed to exist in the average structure of yoderite, where we are dealing with a distribution representing the average of a number of sharp atoms centred at different positions. While the F_c's are rather insensitive to the difference between the two models, the curvatures may be extremely sensitive and are probably smaller for the multiple peak than for the Gaussian peak. The calculated S.D.'s of Table 7 are, therefore, likely to

Table 7. Curvature of atoms and standard deviations of atomic coordinates and bond lengths

Atoms	Curvature at atomic peak (e.Å⁻⁵)	Mean $\sigma(x_n)$ (Å)	Error in bond length (Å)	
Si_1, Si_2	−1384	0·0013	$\sigma(Si-O)$	0·0037
A_1, A_2, A_3	−999	0·0018	$\sigma(A-O)$	0·0038
O_{1-8}	−529	0·0034	$\sigma(O-O)$	0·0048

be too small and this must be remembered when examining the significance of bond-length differences.

Table 8

(a) Cation–anion bond distances

Cation	Type of oxygen (cf. Fig. 4)	Bond length (Å)	σ (bond length) from Table 7 (Å)	Mean cation–oxygen distance (Å)
Si_1	O_1	1·639	0·0037	
	O_1	1·639		$1·638 \pm 0·007$
	O_4	1·627		
	O_3	1·647		
Si_2	O_7	1·664	0·0037	
	O_6	1·582		
	O_2	1·639		$1·631 \pm 0·035$
	O_2	1·639		
A_1	O_2	1·955	0·0038	
	O_8	1·986		
	O_5	1·949		$1·966 \pm 0·024$
	O_7	1·942		
	O_4	1·954		
	O_1	2·011		
A_2	O_8	1·973	0·0038	
	O_5	1·912		
	O_3	1·935		$1·935 \pm 0·023$
	O_2	1·928		
	O_2	1·928		
A_3	O_6	1·824	0·0038	
	O_5	1·944		
	O_3	1·860		$1·870 \pm 0·039$
	O_1	1·862		
	O_1	1·862		

(b) Oxygen–oxygen distances

(i) O–O distances within tetrahedra

Si_1

O_1–O_1 2·690 Å
O_1–O_4 2·695
O_1–O_3 2·657
O_4–O_3 2·653

$\}$ Mean = $2·673 \pm 0·019$ Å

Si_2

O_7–O_6 2·622 Å
O_7–O_2 2·746
O_6–O_2 2·616
O_2–O_2 2·635

$\}$ Mean = $2·655 \pm 0·053$ Å

(ii) O–O distances for oxygens not coordinated round the same atom

O_6–O_8 2·843 Å
O_3–O_6 2·909
All others   > 3·000

(c) Interbond angles within tetrahedra

O_1–Si_1–O_1 110° 18′
O_1–Si_1–O_4 111 20
O_1–Si_1–O_3 107 54
O_1–Si_1–O_4 111 20
O_1–Si_1–O_3 107 54
O_4–Si_1–O_3 108 14

$\}$ Mean = 109° 29′ ± 1° 31′

O_2–Si_2–O_2 107° 0′
O_2–Si_2–O_6 108 32
O_2–Si_2–O_7 112 30
O_2–Si_2–O_6 108 32
O_2–Si_2–O_7 112 30
O_6–Si_2–O_7 107 42

$\}$ Mean = 109° 24′ ± 2° 13′

(The error quoted for the mean is in each case the standard deviation of a single member of the group from the mean.)

Cation-oxygen bond lengths and content of $A_1A_2A_3$ sites

The standard deviations of bond lengths within each kind of polyhedron are shown in the last column of Table 8(a). Most of these are so much greater than the error estimated from $\sigma(x_n)$ as to suggest a real scatter of bond lengths within a polyhedron. Nevertheless, they provide an estimated upper limit to the error in bond lengths, which is useful because of the suspicion mentioned above that the value derived from $\sigma(x_n)$ is too low.

In the octahedron, the average A–O distance* of $1·97 \pm 0·01$ Å is significantly larger than the sum of the Goldschmidt ionic radii for aluminium and oxygen in six-coordination ($\backsimeq 1·89$ Å), but less than that for magnesium and oxygen ($\backsimeq 2·10$ Å). This suggests that some of the Mg atoms and possibly the few Ca and Fe atoms present (cf. Table 2) are occupying A_1 sites.

The A_3–O distance* of $1·87 \pm 0·02$ Å is about that expected for Al–O in 5-coordination; but the A_2–O distance* of $1·94 \pm 0·01$ Å is significantly bigger than this, suggesting that some A_2 sites are occupied by larger cations than aluminium. As there is so little calcium and iron, some magnesium would have to occupy the 5-coordinated site to explain the large A_2–O distance.

Thus, to summarize, it may be said that:

1. A_1 and A_2 sites contain some magnesium atoms as well as aluminium;
2. A_3 sites contain aluminium and little or no magnesium;
3. the few Fe and Ca atoms present probably occupy A_1 or A_2 sites.

The hydrogen atoms

Chemical analysis (cf. Table 2) shows that yoderite contains 2·36 hydrogen atoms per unit cell. The lines of argument followed in deducing their positions are similar to those used for afwillite (Megaw, 1952). These rely on evidence from (i) the electrostatic valency, (ii) O–O distance, (iii) cation–O distance.

Table 9. Electrostatic valency table

Oxygen	Neighbours closer than 2·50 Å	Approximate electrostatic valency*
O_1	A_1, Si_1, A_3	2·06
O_2	A_1, A_2, Si_2	2·01
O_3	A_3, A_2, Si_1	2·15
O_4	A_3, Si_2	1·60
O_5	A_1, A_1, Si_1	1·94
O_6	A_1, A_1, Si_2	1·94
O_7	A_1, A_1, A_2, A_3	2·07
O_8	A_2, A_1, A_1	1·47

* The valencies are calculated assuming A_1, A_2 and A_3 sites are occupied in accordance with the conclusions drawn in the text above.

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* The error limit quoted is the (rounded-off) S.D. of the mean for the polyhedron, derived from the S.D. of individual bonds listed in Table 8(a).

(i) Table 9 shows the electrostatic valencies of the oxygen atoms calculated on the assumption that neighbours closer than 2·5 Å are electrostatically linked (all atoms being taken as fully ionized). The results show:

1. that all atoms except O_4 and O_8 have a valency consistent with O^{2-} occupation;
2. that O_4 and O_8 could be OH^- ions;
3. that all atoms are sufficiently negative to rule out the possibility of H_2O groups.

The suggestion that O_4 is an OH^- ion is not, however, confirmed by the evidence from O–O and cation–O distances.

(ii) All O–O distances between oxygens not coordinated around the same cation are greater than 3·00 Å and may be taken as van der Waals contacts, except for O_6–O_8 which is 2·843 ± 0·005 Å and O_3–O_6 which is 2·909 ± 0·005 Å (cf. Table 8). Of these atoms, O_3 and O_6 have electrostatic valencies $\simeq 2$ (cf. Table 9), while O_8 has electrostatic valency 1·47, and is thus the most likely O to be linked to a hydrogen atom. If each O_8 is attached to one hydrogen, 2 of the 2·36 hydrogens in each cell are accounted for.

(iii) Cation–oxygen distances are likely to be shorter for O^{2-} than for OH^-. From Table 8 it can be seen that O_8 is the only oxygen showing greater bond lengths than the mean for each polyhedron in which it occurs. In A_1 sites, A_1–O_8 is 1·986 ± 0·004 Å (error calculated from $\sigma(x_n)$, which is not significantly greater than the mean of the remaining A_1–O distances 1·962 ± 0·025 Å (error from S.D. of a bond length); but in A_2 sites A_2–O_8 is 1·973 ± 0·004 Å, which is very significantly greater than the mean of

the remaining A_2–O distances, 1·926 ± 0·009 Å (errors as before). It may therefore be concluded that 1 of the 1·18 hydrogens in each asymmetric unit is linked to O_8 and forms a hydrogen bond between O_8 and O_6. Nothing can be said about the remaining 0·18 hydrogens.

Description of the structure

The average structure of yoderite has been shown to consist of chains of AO_6 octahedra running parallel to the y-axis, linked to each other by isolated SiO_4 tetrahedra and by two kinds of AO_5 trigonal bipyramids. One kind of AO_5 bipyramid contains only Al atoms (as far as the present work can distinguish); the other contains some of the larger cations, as do the octahedra. There is an OH^- group at a corner common to two octahedra and one AO_5 group, and a hydrogen bond links this to one corner of a SiO_4 tetrahedron. Fig. 4 is a projection of the structure down [010] and shows the positions of the hydrogen bonds. Fig. 5 is the final $h0l$ Fourier synthesis.

Relation to kyanite

The striking resemblance between yoderite and kyanite may be seen in Fig. 3 where the idealized [010] projection of yoderite is shown alongside the corresponding kyanite projection. Both structures are made up of octahedral chains sharing edges, but in kyanite these chains are linked by AO_6 octahedra and not AO_5 trigonal bipyramids. The oxygen arrangement in yoderite is related to the cubic close-packed array of kyanite. The packing is identical within slabs of the structure bounded by planes $z = +\frac{1}{2}$, $z = -\frac{1}{2}$ (referred
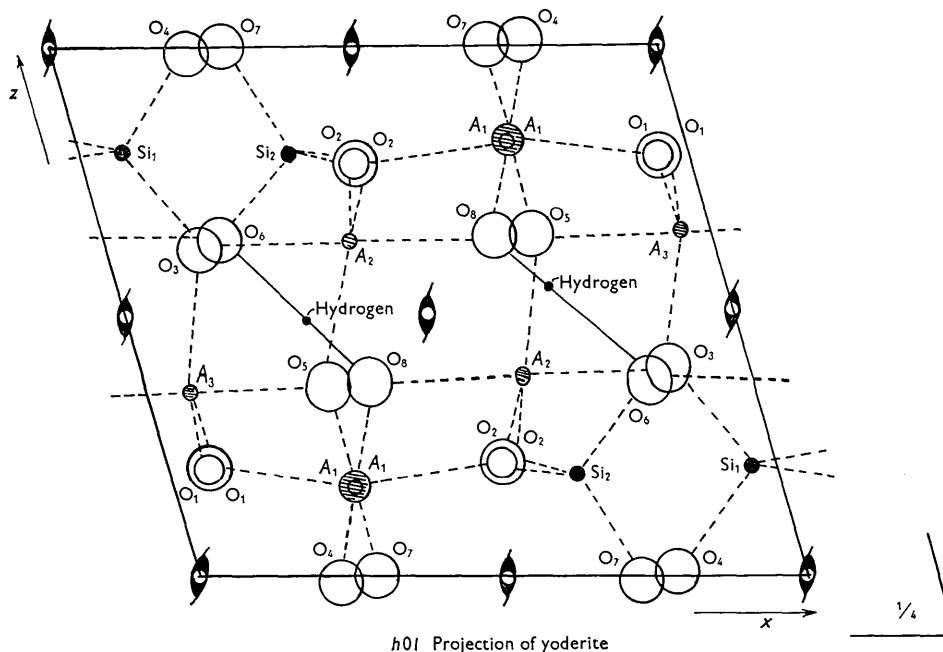

$h0l$ Projection of yoderite
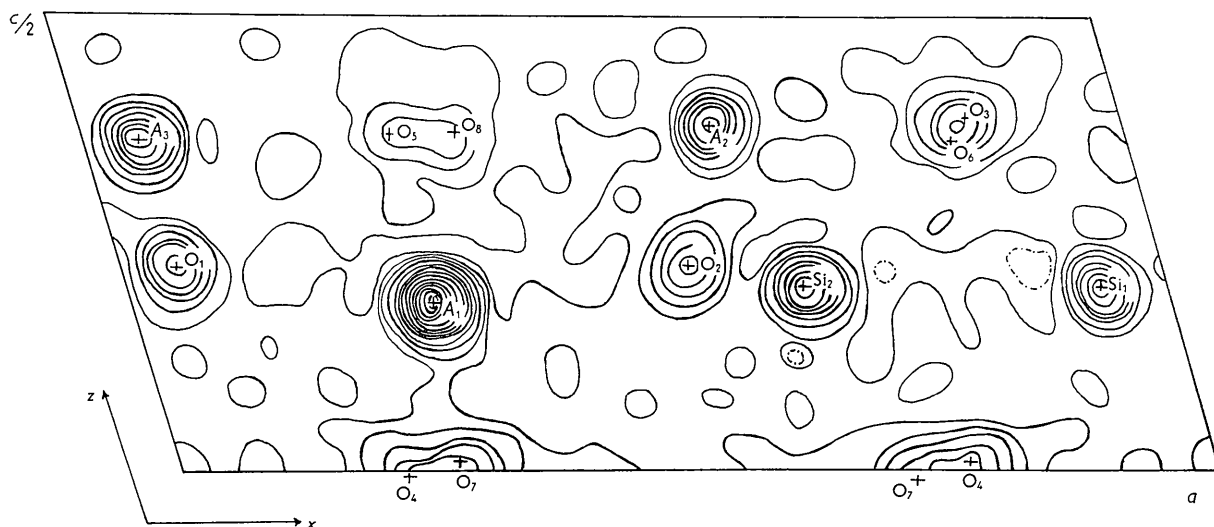
Fig. 4. The structure of yoderite projected $\perp_r$ to [010].

Fig. 5. (010) electron-density projection of yoderite. Contours are at intervals of 3 e.Å^{-2}: zero contour is shown as a chain

to yoderite axes); but to produce the yoderite oxygen packing from that of kyanite, successive kyanite slabs must be displaced upwards by $\frac{1}{4}c_{kyanite}$ relative to the preceding slab (cf. Fig. 3). The increased volume per oxygen atom (16·5 Å^3 in yoderite as opposed to 14·2 Å^3 in kyanite) is partly a result of the substitution of larger ions for some Al^{3+} in yoderite, and partly a result of less good oxygen packing at the interface between the slabs.

Discussion

It must be emphasized that the structure described in this paper is an average structure only. The low R-factors and flat difference maps are evidence that it is a very good approximation to the final answer. Indeed, the only features on the maps which suggest departures from the average are anisotropic regions around some of the oxygen atoms, which might have been ascribed to thermal vibrations with root mean square amplitudes of less than 0·1 Å.

The existence of the subsidiary reflections, here neglected, shows that this is not the whole story. These indicate the existence of a larger true cell, made up of a regular periodic repetition of subcells. Work in progress suggests that the sequence of subcells is characterized by a particular Al/Mg ordering associated with periodic differences in atomic coordinates. In the average structure, the superposition of atoms with different coordinates appears as a spread electron density; the anisotropic regions on the difference maps surround atoms whose positions in different subcells lie within about ±0·1 Å from their mean. A model has been found which gives good agreement for the positions of the weak reflections, and fair agreement for their intensities. It is hoped to describe the results in a future paper.

It is worth emphasizing that, without the evidence

of the subsidiary reflections, there is very little in the 'average' structure to show that it is not a perfect structure with some anisotropic temperature factors. If the subsidiary reflections had been unobservable, the regular ordering over periods greater than a subcell would not have been detected. The only hint that something has been neglected is the value of the oxygen temperature factor (Table 5). All temperature factors may have been artificially reduced by neglecting the effect of primary extinction, but the oxygen temperature factor remains higher than is usual in some recent well-refined silicate structures. This observation has an important application to structures which allow the existence of antiphase domains; such domains, if of sufficiently small volume, might broaden out the subsidiary reflections so much that they escaped detection, and therefore the necessity for a larger true repeat unit might remain unsuspected. These remarks serve as a warning that structures which appear perfect, but which show large isotropic temperature factors or unexplained anisotropic regions around atoms on difference maps, may well possess a true cell which is a multiple of that attributed to them in carrying out the analysis.

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Notes and News

Announcements and other items of crystallographic interest will be published under this heading at the discretion of the Editorial Board. The notes (in duplicate) should be sent to the General Secretary of the International Union of Crystallography (D. W. Smits, Mathematisch Instituut, University of Groningen, Reitdiepskade 4, Groningen, The Netherlands).

International Union of Crystallography

International Tables for X-ray Crystallography

Volume III of the *International Tables for X-ray Crystallography*, which completes the trilogy originally planned in 1946, has now been published at the price of £5. 15s. for the quarto-sized volume of xvi + 362 pages. As with the previous Volumes, working crystallographers may obtain a personal reduced-price copy; such orders must be made directly with the publishers. UNESCO coupons may be used where soft currency might cause difficulties of exchange. The low prices are only possible because the editors and authors have all given their time freely. What profits there are will go to the International Union of Crystallography and help to maintain its many activities; but about 2300 copies will have to be sold before the cost price of printing and publishing is recovered. There seems little doubt that this will happen within about 2 years. A total edition of 4000 has been printed. Crystallographers may do a real service to the Union by sending reviews to Journals that might otherwise overlook this publication, as well as by making sure that the library of their institution or industry has an adequate number of copies. Order forms and copies of the Prospectus containing a brief list of contents and authors, may be obtained from the Kynoch Press, Birmingham 6, England.

Further *Errata Slips* for Volumes I and II of the International Tables for X-ray Crystallography are now ready and will be included in all copies of Volume III. Extra copies may be obtained on application to the publishers, the Kynoch Press.

Suggestions for corrections, for further Volumes or Supplements, should be sent to the Chairman of the Editorial Commission who, after July 1962, will be Prof. M. J. Buerger, M.I.T., Cambridge, Mass., U.S.A., or to the Editors most concerned.

International Union of Crystallography

Report of Executive Committee for 1961

Personal notes

The Executive Committee regrets to report that in 1961 the community of crystallographers lost three senior members: Professor William Thomas Astbury (4 June), Sir Kariamanikkam Srinivasan Krishnan (14 June) and Professor Carl Hermann (12 September); and it wishes to express its sympathy with the British, the Indian and the German crystallographers in their loss of these scientists. Professor Krishnan was a member of the Executive Committee during the triennium 1951–1954, and participated in this capacity in the work of the Union. The Union is also indebted to Professor Hermann, who was editor of the original *Internationale Tabellen zur Bestimmung von Kristallstrukturen*, the precursor of the present *International Tables for X-ray Crystallography*. Obituaries devoted to Professors Astbury, Krishnan and Hermann were published in *Nature* (1961), **191**, 331, 541; **192**, 604. Reference may also be made to an obituary published in *ICSU Review* (1961), **3**, 211, in which Professor Krishnan's role in various international organizations has been recalled.

On 18 December the 70th birthday of the Vice-President of the Union, Academician N. V. Belov, was officially celebrated at the Soviet Academy of Sciences; and he was awarded the order of Lenin.

Meetings

By invitation of the Science Council of Japan, the Union participated, through its Commission on Electron Diffraction, in an International Symposium on Electron and Neutron Diffraction, which was held as Part II of an International Conference on Magnetism and Crystallography in Kyoto, Japan, from 25 to 30 September. During thirteen morning, afternoon and evening sessions 118 invited and contributed papers were read. Two of these sessions were actually joint sessions with Part I of the Conference, and devoted to neutron-diffraction studies of magnetic materials. The Conference was attended by about 600 Japanese and 225 foreign scientists; and the approximate ratio of those mainly interested in Parts I and II respectively amounted to 3 to 1.