-4 (10), S_{12} -4 (2), S_{13} -1 (2), $\bar{S}_{22}$ 1 (11), $\bar{S}_{23}$ -3 (2), $\bar{S}_{33}$ 3 (11) 10^{-4} rad. Å; T_{11} 77 (10), $\bar{T}_{12}$ -11 (7), T_{13} -11 (7), T_{22} 222 (9), T_{23} -2 (6), T_{33} 193 (9) 10^{-4} Å² (relative to an origin at $x/a=0\cdot2514$, $y/b=-0\cdot0871$, $z/c=-0\cdot1070$ which makes S symmetric). The generalized index $R_g=[\sum\varDelta^2/\sum U^2]^{1/2}$ for the agreement between the observed and calculated orthogonal U_{ij} was 0·079.

The asymmetric unit consists of one molecule, with approximately $\bar{4}2m$ (D_{2d}) symmetry; chemically equivalent bond lengths are equal within experimental er-

Table 5. *Non-bonded distances* (Å)

Intramolecular			Moleules related by centre of symmetry	
As(1)–As(2)	3·61		As(1)–As(4)	3·60
As(1)–As(3)	3·62		As(1)–Se(2)	3·49
As(1)–Se(3)	3·85		As(1)–Se(4)	3·88
As(1)–Se(4)	3·83		As(2)–Se(1)	3·62
As(2)–As(4)	3·59		As(2)–Se(2)	3·77
As(2)–Se(2)	3·80		As(3)–Se(1)	3·42
As(2)–Se(4)	3·82		As(4)–Se(2)	3·47
As(3)–As(4)	3·60		Se(1)–Se(2)	4·05
As(3)–Se(1)	3·84		Se(2)–Se(2)	3·99
As(3)–Se(3)	3·83		Se(2)–Se(4)	3·93
As(4)–Se(1)	3·80			
As(4)–Se(2)	3·83		Molecules related by *n* glide	
Se(1)–Se(2)	3·49			
Se(1)–Se(3)	3·48		As(2)–As(4)	3·55
Se(1)–Se(4)	4·94		As(2)–As(3)	3·50
Se(2)–Se(3)	4·93		As(2)–Se(4)	3·62
Se(2)–Se(4)	3·51		As(3)–As(4)	3·50
Se(3)–Se(4)	3·47		As(3)–Se(4)	3·73
			Se(3)–Se(4)	3·68
Intermolecular (less than 4·4 Å)			As(4)–Se(4)	4·33
			Se(1)–Se(4)	4·18
Molecules related by 2_1 axis			Se(2)–Se(4)	4·29
As(1)–Se(3)	3·71			
Se(1)–Se(3)	3·93		Molecules related by cell translation	
Se(2)–Se(3)	3·83		As(3)–Se(1)	4·03
			As(3)–Se(3)	3·64

ror. The vectors As(1)–As(4) and As(2)–As(3) make angles of 89·7 and 89·9° with the normal to the mean plane through the four selenium atoms, and an angle of 90·2° with each other. The As–As bond length is within the range found in α-As₄S₄ (2·59 Å), β-As₄S₄ (2·59 Å) α-As₄S₃ (2·45 Å, Whitfield, 1970), and $As_4S_6^{2-}$ (2·58 Å, Porter & Sheldrick, 1971). The intermolecular distances are slightly longer than those in realgar, whose structure has been discussed in detail by Ito, Morimoto & Sadanaga (1952).

A projection of the structure perpendicular to [001] is shown in Fig. 2.

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(±)-4α,8α,14β-Trimethyl-18-nor-5α,13β-androst-9(11)-en-3,17-dione, a Synthetic Precursor of Fusidic Acid*

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Abstract. Crystals of the title compound are monoclinic, $C2/c$, $a=19\cdot562$ (6), $b=11\cdot915$ (4), $c=15\cdot623$ (4) Å, $\beta=107\cdot84$ (4)°, 24°C; $C_{21}H_{30}O_2$, $M=314\cdot47$, $Z=8$, $D_x=1\cdot208$ g cm⁻³; the racemic material was prepared by Dauben, Ahlgren, Leitereg, Schwarzel & Yoshioka [*J. Amer. Chem. Soc.* (1972). **94**, 8593–8594] and crystallized from a cyclohexane–methanol solution. Unusual stereochemistry is exhibited by the 8α configuration and by the 13β,14β *cis* C/D ring junction.

Introduction. Fusidic acid has been shown to be effective in combating infections caused by staphylococci. The total synthesis of the tetracyclic nucleus of fusidic

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acid has been accomplished by Dauben, Ahlgren, Leitereg, Schwarzel & Yoshioka (1972) yielding a (±) material whose n.m.r. spectrum is essentially identical with that of the (+) material obtained from fusidic acid. The crystal and molecular structure determination of the synthetic (±) material, $4\alpha,8\alpha,14\beta$-trimethyl-18-nor-$5\alpha,13\beta$-androst-9(11)-en-3,17-dione, $C_{21}H_{30}O_2$, was undertaken to establish its structure and stereochemistry. The structural formula is shown in Fig. 1.

A single crystal of approximate dimensions $0.30 \times 0.16 \times 0.13$ mm $[\mu(\text{Mo } K\alpha)=0.81$ cm$^{-1}]$ was used. Weissenberg photographs revealed the diffraction conditions hkl: $h+k=2n$ and $h0l$: $l=2n$ $(h=2n)$ which are indicative of the space groups $C2/c$ and Cc. Diffraction data were measured at 24 °C with a Picker FACS-I automatic diffractometer using graphite monochromatized Mo $K\alpha$ radiation. The cell parameters were determined by a least-squares fit to the angular settings of 12 reflections in the range $35° < 2\theta < 39°$ $[\lambda(\text{Mo } K\alpha_1) = 0.70926$ Å]. Intensities for the hemisphere with k positive were collected for $2\theta < 40°$ using the $\theta-2\theta$ scan method. Assuming $2/m$ symmetry, equivalent reflections were averaged to give 1622 unique reflections of which 495, with $I < 3\sigma(I)$ were given zero weight; the standard deviations and weights for the 1127 reflections used in the least-squares refinement were calculated as described by Gibler, Adams, Fischer, Zalkin & Bartlett (1972) with $q=0.05$.

The crystal structure was solved using the program *MULTAN* (Germain, Main & Woolfson, 1971). It was refined by full-matrix least squares to $R_1 = \sum|\Delta F|/\sum|F_o| = 0.0336$ and $R_2 = \{\sum w(\Delta F)^2/\sum w(F_o)^2\}^{1/2} = 0.0412$; $R_1 = 0.0539$ including zero-weighted data. The scattering factors of Doyle & Turner (1968) were used for oxygen and carbon, those of Stewart, Davidson & Simpson (1965) for hydrogen, and the anomalous scattering factors of Cromer & Liberman (1970) for all atoms. Three additional cycles of full-matrix least-squares refinement using the polar complex hydrogen scattering factors as described by Templeton, Olson, Zalkin & Templeton (1972) led to $R_1 = 0.0338$, $R_2 = 0.0409$, R_1 (including zero-weighted data) $= 0.0538$, and the parameters listed in Table 1. A structure-factor table is available.*

Discussion. The molecular structure is closely related to that of fusidic acid reported as the methyl ester 3-*p*-bromobenzoate by Cooper & Hodgkin (1968). A major difference between the stereochemistry of the tetracyclic nucleus in the two structures is the 13β-hydrogen in the present compound leading to a *cis* C/D ring junction; this is apparently of no consequence in establishing a chemical relationship between the two compounds as the configuration at C(13) can be

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inverted by the enol-keto equilibrium at C(17) during the anticipated synthesis of fusidic acid from the present compound.

Bond lengths are listed in Table 2 and bond angles in Table 3; these values have not been corrected for thermal motion. The C–H bond lengths before the refinement with the polar complex hydrogen scattering factors averaged approximately 0.09 Å shorter than the final values listed in Table 2. The observed bond lengths do not appear to differ significantly from the

Table 1. *Atomic parameters*

Fractional coordinates are $\times 10^4$, except those for hydrogen atoms, which are $\times 10^3$.

	x	y	z	B
O(1)	3904 (2)	3918 (3)	−399 (2)	
O(2)	−474 (2)	4822 (3)	1085 (2)	
C(1)	1241 (2)	3548 (3)	1487 (3)	
C(2)	550 (2)	3693 (4)	1743 (4)	
C(3)	174 (2)	4775 (3)	1424 (2)	
C(4)	639 (2)	5810 (3)	1586 (3)	
C(5)	1325 (2)	5610 (3)	1307 (2)	
C(6)	1789 (2)	6675 (3)	1453 (3)	
C(7)	2595 (2)	6466 (3)	1641 (3)	
C(8)	2775 (2)	5419 (2)	1182 (2)	
C(9)	2394 (1)	4391 (3)	1397 (2)	
C(10)	1747 (2)	4558 (3)	1748 (2)	
C(11)	2632 (2)	3385 (3)	1272 (2)	
C(12)	3282 (2)	3142 (3)	982 (3)	
C(13)	3794 (2)	4132 (3)	1078 (2)	
C(14)	3613 (2)	5212 (2)	1515 (2)	
C(15)	4042 (2)	6129 (3)	1189 (3)	
C(16)	4178 (3)	5701 (4)	337 (4)	
C(17)	3952 (2)	4498 (4)	251 (3)	
C(19)	2025 (2)	4656 (4)	2785 (2)	
C(30)	214 (3)	6836 (4)	1128 (4)	
C(31)	2502 (2)	5605 (4)	157 (2)	
C(32)	3885 (2)	5101 (4)	2537 (3)	
H(1)	111 (1)	345 (2)	76 (2)	2.7 (7)
H(2)	152 (2)	280 (3)	184 (2)	6.2 (10)
H(3)	18 (2)	304 (3)	139 (2)	5.7 (10)
H(4)	70 (2)	366 (3)	250 (3)	6.2 (11)
H(5)	81 (2)	596 (2)	232 (2)	3.4 (7)
H(6)	112 (2)	545 (2)	55 (2)	3.8 (7)
H(7)	159 (2)	726 (3)	88 (2)	5.5 (10)
H(8)	168 (2)	710 (3)	205 (2)	5.3 (9)
H(9)	283 (2)	638 (3)	235 (2)	3.7 (9)
H(10)	279 (2)	718 (3)	134 (2)	4.7 (9)
H(11)	237 (1)	270 (2)	138 (2)	1.7 (6)
H(12)	310 (2)	289 (3)	25 (2)	5.3 (9)
H(13)	363 (2)	242 (4)	147 (3)	7.3 (10)
H(14)	433 (2)	387 (2)	150 (2)	3.4 (7)
H(15)	373 (2)	694 (3)	103 (2)	4.6 (8)
H(16)	454 (2)	627 (3)	169 (2)	5.2 (10)
H(17)	384 (2)	612 (3)	−32 (2)	5.2 (10)
H(18)	471 (2)	579 (3)	38 (3)	6.9 (12)
H(19)	−28 (3)	693 (4)	131 (3)	8.4 (13)
H(20)	13 (3)	676 (4)	37 (4)	10.6 (17)
H(21)	53 (2)	763 (4)	143 (3)	6.9 (11)
H(22)	231 (2)	537 (3)	301 (2)	5.2 (11)
H(23)	159 (2)	470 (3)	312 (2)	5.3 (8)
H(24)	241 (2)	391 (3)	310 (2)	6.9 (10)
H(25)	192 (2)	573 (2)	−4 (2)	3.0 (7)
H(26)	278 (2)	637 (3)	−1 (2)	5.8 (9)
H(27)	260 (2)	484 (3)	−21 (2)	4.7 (9)
H(28)	442 (2)	475 (3)	275 (2)	5.4 (9)
H(29)	357 (2)	443 (3)	281 (2)	5.5 (9)
H(30)	389 (2)	586 (3)	289 (3)	6.6 (11)

Table 1 (cont.)

Thermal parameters are in units of Å².
The anisotropic temperature factor has the form:

$$\exp\{-\tfrac{1}{4}\textstyle\sum_i\sum_j(a_i^*a_j^*h_ih_jB_{ij})\}$$

where a_i^* is a reciprocal cell edge and h_i is one of the Miller indices.

	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
O(1)	11·7 (2)	6·9 (2)	6·5 (2)	−0·1 (2)	6·0 (2)	−0·9 (2)
O(2)	2·8 (1)	9·2 (2)	8·2 (2)	−0·2 (1)	1·1 (1)	0·2 (1)
C(1)	3·6 (2)	3·6 (2)	4·6 (2)	−0·4 (2)	1·7 (2)	−0·4 (2)
C(2)	3·6 (2)	4·8 (2)	6·6 (3)	−0·5 (2)	2·3 (2)	−0·2 (2)
C(3)	3·1 (2)	5·5 (2)	4·2 (2)	−0·1 (2)	1·7 (1)	−0·0 (2)
C(4)	3·3 (2)	4·3 (2)	4·7 (2)	0·7 (2)	1·5 (2)	0·3 (2)
C(5)	2·8 (2)	3·3 (2)	3·6 (2)	0·5 (1)	1·1 (1)	0·3 (1)
C(6)	4·1 (2)	3·1 (2)	5·6 (2)	0·5 (2)	2·0 (2)	−0·0 (2)
C(7)	3·5 (2)	2·6 (2)	5·0 (2)	0·0 (1)	1·7 (2)	−0·5 (2)
C(8)	2·9 (2)	2·5 (1)	2·7 (2)	0·0 (1)	0·9 (1)	0·1 (1)
C(9)	2·6 (1)	2·3 (2)	2·3 (1)	0·1 (1)	0·3 (1)	−0·2 (1)
C(10)	2·9 (2)	2·6 (1)	2·8 (2)	0·2 (1)	1·0 (1)	0·1 (1)
C(11)	3·4 (2)	2·6 (2)	4·1 (2)	−0·6 (2)	1·5 (1)	0·0 (1)
C(12)	4·0 (2)	3·5 (2)	5·4 (2)	−0·1 (2)	2·4 (2)	−0·5 (2)
C(13)	3·0 (2)	3·0 (2)	3·7 (2)	0·2 (1)	1·4 (1)	0·0 (1)
C(14)	2·7 (2)	3·0 (2)	3·4 (2)	−0·0 (1)	0·9 (1)	−0·3 (1)
C(15)	4·0 (2)	3·5 (2)	6·8 (3)	−0·3 (2)	2·5 (2)	−0·3 (2)
C(16)	6·1 (3)	4·7 (3)	8·3 (3)	0·3 (2)	5·0 (3)	0·7 (2)
C(17)	5·0 (2)	4·6 (2)	5·2 (2)	0·4 (2)	3·0 (2)	−0·2 (2)
C(19)	4·1 (2)	5·0 (2)	2·9 (2)	0·4 (2)	1·5 (2)	−0·0 (2)
C(30)	4·3 (2)	5·7 (3)	10·2 (4)	2·2 (2)	2·9 (3)	1·3 (3)
C(31)	3·8 (2)	4·5 (2)	3·4 (2)	0·5 (2)	1·1 (1)	1·1 (2)
C(32)	3·2 (2)	5·5 (2)	4·1 (2)	0·2 (2)	0·2 (2)	−0·9 (2)

typical values for steroid structures. Angle distortion in the 5-membered D ring appears to be a real effect of strains in the molecule resulting from the >C=O entity at C(17), the C(9)–C(11) bond, the *cis* C/D ring

Table 2. *Bond lengths*

O(1)—C(17)	1·208 (4) Å	C(1)—H(1)	1·09 (3) Å
O(2)—C(3)	1·216 (4)	C(1)—H(2)	1·10 (4)
C(1)—C(2)	1·532 (5)	C(2)—H(3)	1·09 (4)
C(1)—C(10)	1·532 (4)	C(2)—H(4)	1·13 (4)
C(2)—C(3)	1·492 (5)	C(4)—H(5)	1·11 (3)
C(3)—C(4)	1·507 (5)	C(5)—H(6)	1·15 (3)
C(4)—C(5)	1·552 (5)	C(6)—H(7)	1·10 (4)
C(4)—C(30)	1·528 (5)	C(6)—H(8)	1·14 (4)
C(5)—C(6)	1·536 (5)	C(7)—H(9)	1·06 (3)
C(5)—C(10)	1·541 (4)	C(7)—H(10)	1·10 (4)
C(6)—C(7)	1·532 (5)	C(11)–H(11)	1·00 (3)
C(7)—C(8)	1·534 (4)	C(12)–H(12)	1·13 (4)
C(8)—C(9)	1·522 (4)	C(12)–H(13)	1·21 (5)
C(8)—C(14)	1·579 (4)	C(13)–H(14)	1·10 (3)
C(8)—C(31)	1·542 (5)	C(15)–H(15)	1·13 (4)
C(9)—C(10)	1·540 (4)	C(15)–H(16)	1·06 (4)
C(9)—C(11)	1·322 (4)	C(16)–H(17)	1·15 (4)
C(10)–C(19)	1·548 (4)	C(16)–H(18)	1·02 (4)
C(11)–C(12)	1·502 (5)	C(19)–H(22)	1·02 (4)
C(12)–C(13)	1·525 (5)	C(19)–H(23)	1·13 (4)
C(13)–C(14)	1·548 (4)	C(19)–H(24)	1·18 (4)
C(13)–C(17)	1·483 (5)	C(30)–H(19)	1·09 (5)
C(14)–C(15)	1·555 (5)	C(30)–H(20)	1·15 (6)
C(14)–C(32)	1·526 (5)	C(30)–H(21)	1·15 (4)
C(15)–C(16)	1·524 (6)	C(31)–H(25)	1·10 (3)
C(16)–C(17)	1·494 (6)	C(31)–H(26)	1·13 (4)
		C(31)–H(27)	1·12 (4)
		C(32)–H(28)	1·08 (4)
		C(32)–H(29)	1·17 (4)
		C(32)–H(30)	1·06 (4)

junction, and the methyl groups substituted at C(8) and C(14). Similar angles in the D ring of fusidic acid were reported by Cooper & Hodgkin (1968).

An *ORTEP* drawing (Johnson, 1965) of the molecule is shown in Fig. 2. This view of the molecule emphasizes the distortion of the tetracyclic nucleus from the relatively planar form found when there are all-*trans* ring junctions and when there is no unsatura-

Table 3. *Bond angles*

C(2)—C(1)—C(10)	113·1 (3)°	C(12)-C(13)-C(14)	117·0 (3)°
C(1)—C(2)—C(3)	113·2 (3)	C(12)-C(13)-C(17)	116·6 (3)
C(2)—C(3)—O(2)	121·8 (3)	C(14)-C(13)-C(17)	106·1 (3)
C(4)—C(3)—O(2)	122·0 (3)	C(8)—C(14)-C(13)	109·8 (2)
C(2)—C(3)—C(4)	116·2 (3)	C(8)—C(14)-C(15)	113·1 (3)
C(3)—C(4)—C(5)	110·9 (3)	C(8)—C(14)-C(32)	110·6 (3)
C(3)—C(4)—C(30)	111·3 (4)	C(13)-C(14)-C(15)	102·7 (3)
C(5)—C(4)—C(30)	112·6 (3)	C(13)-C(14)-C(32)	109·6 (3)
C(4)—C(5)—C(6)	110·8 (3)	C(15)-C(14)-C(32)	110·7 (4)
C(4)—C(5)—C(10)	113·0 (3)	C(14)-C(15)-C(16)	107·9 (3)
C(6)—C(5)—C(10)	113·2 (3)	C(15)-C(16)-C(17)	105·8 (3)
C(5)—C(6)—C(7)	114·9 (3)	C(13)-C(17)-O(1)	125·5 (3)
C(6)—C(7)—C(8)	114·4 (3)	C(16)-C(17)-O(1)	124·6 (3)
C(7)—C(8)—C(9)	110·7 (3)	C(13)-C(17)-C(16)	109·8 (3)
C(7)—C(8)—C(14)	109·7 (3)	C(2)—C(1)—H(1)	110 (1)
C(7)—C(8)—C(31)	108·2 (3)	C(2)—C(1)—H(2)	108 (2)
C(9)—C(8)—C(14)	109·3 (2)	C(10)-C(1)—H(1)	107 (1)
C(9)—C(8)—C(31)	107·9 (3)	C(10)-C(1)—H(2)	108 (2)
C(14)-C(8)—C(31)	111·1 (3)	H(1)—C(1)—H(2)	110 (3)
C(8)—C(9)—C(10)	119·1 (2)	C(1)—C(2)—H(3)	107 (2)
C(8)—C(9)—C(11)	118·6 (3)	C(1)—C(2)—H(4)	108 (2)
C(10)-C(9)—C(11)	122·3 (3)	C(3)—C(2)—H(3)	105 (2)
C(1)—C(10)-C(5)	107·8 (3)	C(3)—C(2)—H(4)	109 (2)
C(1)—C(10)-C(9)	109·8 (3)	H(3)—C(2)—H(4)	115 (4)
C(1)—C(10)-C(19)	109·2 (3)	C(3)—C(4)—H(5)	106 (1)
C(5)—C(10)-C(9)	109·4 (2)	C(5)—C(4)—H(5)	108 (1)
C(5)—C(10)-C(19)	112·0 (3)	C(30)-C(4)—H(5)	108 (1)
C(9)—C(10)-C(19)	108·6 (2)	C(4)—C(5)—H(6)	105 (1)
C(9)—C(11)-C(12)	126·0 (3)	C(6)—C(5)—H(6)	107 (1)
C(11)-C(12)-C(13)	114·3 (3)	C(10)-C(5)—H(6)	107 (1)
C(5)—C(6)—H(7)	110 (2)	C(4)—C(30)-H(19)	111 (2)
C(5)—C(6)—H(8)	104 (2)	C(4)—C(30)-H(20)	108 (2)
C(7)—C(6)—H(7)	111 (2)	C(4)—C(30)-H(21)	109 (2)
C(7)—C(6)—H(8)	111 (2)	H(19)-C(30)-H(20)	114 (5)
H(7)—C(6)—H(8)	106 (3)	H(19)-C(30)-H(21)	102 (4)
C(6)—C(7)—H(9)	108 (2)	H(20)-C(30)-H(21)	113 (5)
C(6)—C(7)—H(10)	105 (2)	C(10)-C(19)-H(22)	114 (2)
C(8)—C(7)—H(9)	109 (2)	C(10)-C(19)-H(23)	115 (2)
C(8)—C(7)—H(10)	105 (2)	C(10)-C(19)-H(24)	110 (2)
H(9)—C(7)—H(10)	115 (3)	H(22)-C(19)-H(23)	102 (4)
C(9)—C(11)-H(11)	119 (1)	H(22)-C(19)-H(24)	106 (4)
C(12)-C(11)-H(11)	115 (1)	H(23)-C(19)-H(24)	109 (3)
C(11)-C(12)-H(12)	109 (2)	C(8)—C(31)-H(25)	108 (1)
C(11)-C(12)-H(13)	108 (2)	C(8)—C(31)-H(26)	109 (2)
C(13)-C(12)-H(12)	108 (2)	C(8)—C(31)-H(27)	111 (2)
C(13)-C(12)-H(13)	106 (2)	H(25)-C(31)-H(26)	111 (3)
H(12)-C(12)-H(13)	113 (3)	H(25)-C(31)-H(27)	107 (3)
C(12)-C(13)-H(14)	108 (2)	H(26)-C(31)-H(27)	112 (3)
C(14)-C(13)-H(14)	106 (1)	C(14)-C(32)-H(28)	111 (2)
C(17)-C(13)-H(14)	101 (1)	C(14)-C(32)-H(29)	113 (2)
C(14)-C(15)-H(15)	112 (2)	C(14)-C(32)-H(30)	114 (2)
C(14)-C(15)-H(16)	110 (2)	H(28)-C(32)-H(29)	101 (3)
C(16)-C(15)-H(15)	108 (2)	H(28)-C(32)-H(30)	108 (4)
C(16)-C(15)-H(16)	109 (2)	H(29)-C(32)-H(30)	109 (4)
H(15)-C(15)-H(16)	109 (3)		
C(15)-C(16)-H(17)	115 (2)		
C(15)-C(16)-H(18)	110 (2)		
C(17)-C(16)-H(17)	105 (2)		
C(17)-C(16)-H(18)	112 (2)		
H(17)-C(16)-H(18)	108 (4)		

tion in or adjacent to the rings (see, for example, Geise & Romers, 1966).

The molecules are arranged in the crystal with their longest dimension parallel to **a**, and they are packed side by side in a triangular fashion into layers parallel to **b** and **c**. There are no abnormally close intermolecular contacts. The shortest intermolecular distances between carbon and oxygen atoms are 3·36 Å for C–O, 3·55 Å for C–C, and 4·35 Å for O–O.

Fig. 1. Structural formula.

Fig. 2. Molecular structure. Thermal ellipsoids were drawn at the 50% probability level and hydrogen atoms were given an arbitrary temperature parameter.

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Barium Chloride Silicate with an Open Framework: Verplanckite
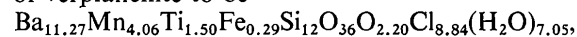
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Abstract. Verplanckite is hexagonal, $P6/mmm$, $a = 16\cdot398$ (10), $c = 7\cdot200$ (4) Å, $[(Mn,Ti,Fe)_6(OH,O)_2(Si_4O_{12})_3]Ba_{12}Cl_9\{(OH,H_2O)_7\}$, $Z = 1$, $D_x = 3\cdot33$ g cm^{-3}. Material from the type locality was used, Esquire No. 7 mine, Big Creek, Fresno County, California (Specimen No. M18580, Field Museum of Natural History). The structure is recognized as an example of a new kind of compound which consists of a wide open framework, wholly or partly a silicate, filled by barium and chlorine atoms and OH or H$_2$O groups. The voids in verplanckite (free diameter 7·2 Å) approach the dimensions of the openings found in the most open zeolites.

Introduction. Two small crystals of verplanckite were isolated from a specimen of sanbornite-bearing rock (Alfors, Stinson, Matthews & Pabst, 1965). One crystal was used to determine by electron microprobe anal-

ysis the chemical composition (normalized to 12 for Si) of verplanckite to be $Ba_{11\cdot27}Mn_{4\cdot06}Ti_{1\cdot50}Fe_{0\cdot29}Si_{12}O_{36}O_{2\cdot20}Cl_{8\cdot84}(H_2O)_{7\cdot05}$, where the water content represents the difference between the total weight percents of all other constituents and 100%. The agreement between our analysis and the previous one (Alfors & Putnam, 1965) is fair, except for the water content, which we find to be smaller, and the chlorine content, which we find to be larger.

From the second crystal (approximately cylindrical shape $0\cdot06 \times 0\cdot06 \times 0\cdot2$ mm) we obtained 6557 non-unique intensities on a computer-controlled four-circle diffractometer using crystal-monochromatized Ag $K\alpha$ radiation (wavelength $\lambda = 0\cdot56083$ Å). The reduction and averaging of the intensity data resulted in 679 unique structure factors (maximum $\sin\theta/\lambda = 0\cdot60$ Å^{-1}), of which 293 were considered to be of zero intensity. No absorption correction was applied to the