Crystal Structure of 2α,3α-Epithio-5α-androstan-17β-yl *p*-Bromobenzoate

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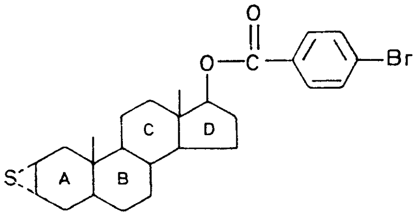
The crystal structure of the title compound (I) has been determined by three-dimensional *X*-ray analysis from diffractometer data. Crystals are orthorhombic, $a = 18{\cdot}924 \pm 0{\cdot}003$, $b = 7{\cdot}099 \pm 0{\cdot}002$, $c = 17{\cdot}825 \pm 0{\cdot}003$ Å, $Z = 4$, space group $P2_12_12_1$. The structure was solved by the programme SEARCHER, for automatic heavy-atom analysis, and refined by block-diagonal least-squares calculations to a final R of 0·073 for 1172 independent observed reflexions. Hydrogen atoms were located from a difference-Fourier synthesis. Rings B and C of the steroid skeleton have the chair and rings A and D the half-chair conformation. The absolute configuration was determined by the anomalous dispersion method.

DURING an investigation on the ring-opening reaction of steroidal oxides by thiocyanic acids, the synthesis and reactions of 2,3- and 3,4-episulphides were investigated.[1] Their crystal structures were determined [2-4] in our search for relationships between the structure and biological activity of steroid molecules. The present crystal structure determination was carried out in order to elucidate the detailed stereochemistry of 2α,3α-epithio-5α-androstan-17β-yl *p*-bromobenzoate (I) particularly in the thiiran ring and ring A. This compound seems to be

(I)



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[1] K. Takeda, T. Komeno, J. Kawanami, S. Ishihara, H. Kadokawa, H. Tokura and H. Itani, *Tetrahedron*, 1965, **21**, 329.

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the first example of steroid which contains the thiiran ring in the skeleton. C.d. and o.r.d. data for 2α,3α-epithio-5α-androstan-17β-ol are reported in ref. 5.

EXPERIMENTAL

Crystals of (I) were prepared [6] as colourless prisms, m.p. 180—181·5 °C (from ethyl acetate), elongated along the *b* axis. Precession and Weissenberg photographs were taken with Cu-K_α (λ = 1·5418 Å) radiation; space group and unit-cell dimensions were determined from precession photographs and from measurements taken on a Hilger and Watts automatic diffractometer.

Crystal Data.—$C_{26}H_{33}BrO_2S$, M = 489·5. Orthorhombic, a = 18·924 ± 0·003, b = 7·099 ± 0·002, c = 17·825 ± 0·003 Å, U = 2394 Å^3, D_m = 1·359 (by flotation), Z = 4, D_c = 1·358. F(000) = 1024. Space group, $P2_12_12_1$, (D_2^4, No. 19) from systematic absences: h00 for h odd, 0k0 for k odd, 00l for l odd. Mo-K_α radiation, λ = 0·7107 Å; μ(Mo-K_α) = 19·25 cm^{-1}.

TABLE 1

Final fractional atomic co-ordinates and anisotropic thermal parameters (× 10^4),* with estimated standard deviations in parentheses

Atom	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Br	0·6039(1)	1·4175(4)	−0·00488(1)	66(1)	763(10)	52(1)	34(3)	7(1)	54(2)
S	0·5357(2)	0·8620(11)	0·8194(2)	57(2)	732(27)	43(1)	−52(7)	7(1)	−38(6)
O(1)	0·6097(5)	0·8734(17)	0·2619(4)	44(3)	396(36)	34(3)	−7(12)	−7(3)	−51(10)
O(2)	0·5384(7)	0·6889(19)	0·1939(6)	98(7)	461(47)	49(5)	−90(16)	−22(5)	−51(13)
C(1)	0·5903(8)	1·0013(22)	0·6814(7)	50(6)	246(12)	30(4)	30(15)	8(5)	26(13)
C(2)	0·5915(8)	1·0159(27)	0·7665(7)	42(6)	458(63)	31(5)	−18(18)	−2(4)	−1(16)
C(3)	0·6290(9)	0·8688(29)	0·8091(7)	66(7)	474(65)	24(4)	−2(21)	0(5)	−61(16)
C(4)	0·6708(9)	0·7222(29)	0·7691(7)	66(7)	398(16)	26(5)	−25(20)	−13(5)	26(16)
C(5)	0·6468(6)	0·6924(26)	0·6870(8)	20(4)	434(60)	39(5)	−19(14)	−2(4)	−44(16)
C(6)	0·6943(9)	0·5529(23)	0·6510(9)	70(8)	155(40)	52(6)	−46(17)	18(6)	38(15)
C(7)	0·6707(9)	0·5125(21)	0·5668(9)	72(8)	130(38)	54(7)	0(15)	9(6)	−42(15)
C(8)	0·6702(8)	0·6944(20)	0·5229(7)	48(6)	144(37)	29(4)	−15(13)	0(4)	22(11)
C(9)	0·6200(7)	0·8404(18)	0·5636(7)	41(5)	123(32)	35(5)	−29(12)	16(4)	−13(11)
C(10)	0·6458(8)	0·8853(23)	0·6444(7)	48(6)	212(41)	33(5)	4(15)	9(4)	4(14)
C(11)	0·6120(7)	1·0086(20)	0·5152(6)	27(4)	242(41)	27(4)	−5(13)	5(4)	−28(11)
C(12)	0·5858(7)	0·9650(26)	0·4357(7)	39(5)	419(58)	32(5)	20(16)	−9(4)	53(15)
C(13)	0·6335(6)	0·8296(24)	0·3971(7)	18(4)	389(54)	28(4)	−14(13)	−2(3)	−41(14)
C(14)	0·6393(7)	0·6465(23)	0·4476(8)	36(5)	317(51)	41(5)	−5(14)	−0(5)	47(17)
C(15)	0·6758(9)	0·5113(23)	0·3939(9)	62(7)	197(44)	59(7)	40(16)	15(6)	19(17)
C(16)	0·6430(9)	0·5659(27)	0·3139(9)	66(8)	305(53)	56(7)	−49(20)	8(6)	−44(19)
C(17)	0·6032(8)	0·7410(24)	0·3255(8)	43(5)	282(47)	39(5)	−25(16)	14(5)	−8(14)
C(18)	0·7071(7)	0·9145(30)	0·3805(7)	41(5)	446(60)	27(4)	−8(19)	14(4)	6(17)
C(19)	0·7178(7)	0·9725(25)	0·6453(7)	35(5)	378(58)	34(5)	−48(16)	−8(4)	−5(16)
C(1′)	0·5786(8)	0·9687(26)	0·1416(9)	47(6)	314(54)	56(7)	−10(16)	−13(6)	−50(17)
C(2′)	0·5996(7)	1·1462(29)	0·1579(6)	23(4)	679(81)	19(3)	72(19)	4(3)	10(15)
C(3′)	0·6073(9)	1·2897(24)	0·1026(9)	48(6)	289(51)	59(7)	64(17)	−18(6)	−43(17)
C(4′)	0·5961(8)	1·2353(28)	0·0285(9)	40(6)	411(58)	59(8)	40(18)	−12(6)	−54(19)
C(5′)	0·5736(10)	1·0656(32)	0·0113(7)	79(9)	559(72)	17(4)	80(25)	3(5)	−36(17)
C(6′)	0·5657(10)	0·9273(35)	0·0670(8)	73(8)	677(87)	34(6)	−3(25)	−2(6)	−116(22)
C(7′)	0·5705(9)	0·8261(25)	0·2026(7)	64(7)	334(57)	31(5)	−36(18)	4(5)	−32(15)

* In the form $T = \exp\{-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)\}$.

Three-dimensional intensity data were collected on a Hilger and Watts automatic four-circle Y 290 diffractometer controlled by a PDP 8 computer. Integrated intensities were measured for $\theta < 27\cdot5°$ by the θ—2θ scan technique with Mo-K_α radiation and a scintillation counter (with a zirconium filter and pulse-height analyser). Each reflexion was integrated in 80 steps of intervals of 0·01°. In this way 3135 independent reflexions were recorded of which 1172 were considered observed, having $I > 3\sigma(I)$, and were used in the subsequent analysis. All intensities were corrected for Lorentz and polarization factors, and structure amplitudes were derived. No absorption corrections were applied since the specimen was considered to be sufficiently small (0·33 × 0·43 × 0·38 mm).

Structure Analysis.—The structure was solved by the SEARCHER programme for automatic heavy-atom analysis, written [7,8] for the CDC 3600 computer (later modified for CDC 6600 computer). Initial co-ordinates for the bromine atom were easily derived from a three-dimensional Patterson synthesis as x/a = 0·10, y/b = 0·09, z/c = 0·05. The atomic co-ordinates of 29 light atoms, which were treated as carbon atoms, were found from the first cycle of the SEARCHER programme. The second cycle yielded the last atom in the molecule. Total time for the automatic heavy-atom analysis was only *ca.* 19·14 min with a CDC 6600 computer (14·23 min for the 1st cycle, 4·91 min for the 2nd cycle).

The co-ordinates of the 30 atoms from the second SEARCHER cycle were refined [9] by full-matrix least-squares calculations. After two cycles, with isotropic temperature factors, R was 0·116. At this stage, a three-dimensional difference-Fourier synthesis was calculated and the positions of the 33 hydrogen atoms found. Five cycles of block-diagonal least-squares refinement were carried out with anisotropic temperature factors, the function which minimized being $\Sigma w(F_o - F_c)^2$ with unit weights; hydrogen atoms were included in the structure-factor calculations with isotropic temperature factors (*B*

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[5] K. Kuriyama, T. Komeno, and K. Takeda, *Tetrahedron*, 1966, **22**, 1039.

[6] S. Ishihara, Ref. 1.

[7] H. Koyama and K. Okada, 1970, SEARCHER, A Fortran program for the automatic heavy-atom analysis of organic compounds, Shionogi Research Laboratory, Osaka, Japan.

[8] H. Koyama and K. Okada, *Acta Cryst.*, 1970, *B*, **26**, 444.

[9] W. L. Busing, K. O. Martin, and H. A. Levy, ORFLS, A Fortran crystallographic least-squares program, Oak Ridge National Laboratory, Tennessee, Report ORNL-TM-305, 1962.

fixed at 2·44 Å^2). In the final calculations the hydrogen co-ordinates were also permitted to vary (a shift factor 0·35 was applied), and at this stage R was 0·073 for the 1172 reflexions.

Observed and calculated structure amplitudes are listed in Supplementary Publication No. SUP 20796 (4 pp.).* Atomic scattering factors used in all calculations were taken from ref. 10 for non-hydrogen and from ref. 11 for hydrogen atoms. Final positional and thermal parameters with their estimated standard deviations are given in Table 1.

Absolute Configuration.—In the final stage of the analysis the absolute configuration of the molecule was determined by the anomalous dispersion method.[12] The differences between $I_0(hkl)$ and $I_0(h\bar{k}l)$ were measured visually from rotation photographs rotated around the b axis, taken with Cr-K_α radiation. Structure factors were calculated for all the hkl and $h\bar{k}l$ reflexions, with a scattering factor for the bromine atom of the form $f_{\mathrm{Br}} = f_{\mathrm{Br}} + \Delta f'_{\mathrm{Br}} + i\Delta f''_{\mathrm{Br}}$, where $\Delta f'_{\mathrm{Br}} = -0{\cdot}6$ and $\Delta f''_{\mathrm{Br}} = 2{\cdot}7$. The results (Table 2) indicate that the parameters of Table 1 represent the true absolute configuration.

TABLE 2

Determination of the absolute configuration (Cr-K_α radiation)

hkl	$\lvert F_c(hkl)\rvert^2$	$\lvert F_c(h\bar{k}l)\rvert^2$	$I_0(hkl) \lessgtr I_0(h\bar{k}l)$
2,1,7	707	441	>
3,1,3	302	605	<
3,1,4	306	585	<
3,1,7	349	620	<
6,1,4	635	806	<
7,1,4	156	8	>
7,1,9	353	696	<
8,1,2	676	542	>
9,1,5	40	64	<
11,1,1	196	289	<
11,1,3	243	106	>
13,1,4	595	718	<
7,2,1	571	1874	<

DISCUSSION

The configuration of the molecule is illustrated[13] in Figure 1 where each atom is represented as an ellipsoid to conform with the thermal parameters listed in Table 1. The steroid skeleton consists of the thiiran ring and the basic 5α-androstane moiety with rings A, B, C, and D *trans*-connected. Atoms C(18), C(19), and O(1) are on the β side of the ring system of the steroid skeleton, while the atom S is on the α side.

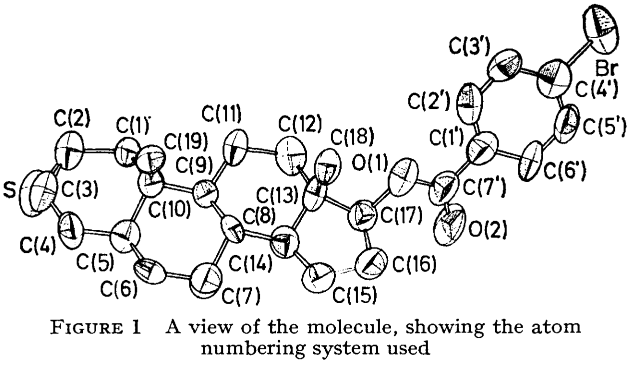

FIGURE 1 A view of the molecule, showing the atom numbering system used

TABLE 3

Interatomic distances (Å) and angles (°), with standard deviations in parentheses

(*a*) Bond distances

S–C(2)	1·788(17)	C(13)–C(17)	1·533(20)
S–C(3)	1·775(19)	C(13)–C(18)	1·546(20)
C(1)–C(2)	1·519(18)	C(14)–C(15)	1·521(23)
C(1)–C(10)	1·488(21)	C(15)–C(16)	1·601(24)
C(2)–C(3)	1·474(25)	C(16)–C(17)	1·468(25)
C(3)–C(4)	1·488(26)	C(17)–O(1)	1·478(18)
C(4)–C(5)	1·548(20)		
C(5)–C(6)	1·482(23)	O(1)–C(7′)	1·333(18)
C(5)–C(10)	1·565(23)	O(2)–C(7′)	1·158(22)
C(6)–C(7)	1·591(23)	C(1′)–C(2′)	1·352(26)
C(7)–C(8)	1·510(21)	C(1′)–C(6′)	1·384(22)
C(8)–C(9)	1·581(19)	C(1′)–C(7′)	1·493(23)
C(8)–C(14)	1·503(20)	C(2′)–C(3′)	1·424(24)
C(9)–C(10)	1·554(19)	C(3′)–C(4′)	1·393(23)
C(9)–C(11)	1·480(18)	C(4′)–C(5′)	1·313(30)
C(10)–C(19)	1·496(21)	C(5′)–C(6′)	1·403(27)
C(11)–C(12)	1·534(18)	Br–C(4′)	1·896(18)
C(12)–C(13)	1·486(21)		
C(13)–C(14)	1·584(22)		

(*b*) Valency angles

C(2)–S–C(3)	48·8(8)	C(12)–C(13)–C(17)	115·0(11)
C(2)–C(1)–C(10)	118·0(12)	C(12)–C(13)–C(18)	112·4(14)
S–C(2)–C(1)	118·4(12)	C(14)–C(13)–C(17)	99·3(12)
S–C(2)–C(3)	65·1(9)	C(14)–C(13)–C(18)	111·4(11)
C(1)–C(2)–C(3)	118·2(14)	C(17)–C(13)–C(18)	109·7(11)
S–C(3)–C(2)	66·0(9)	C(8)–C(14)–C(13)	110·4(12)
S–C(3)–C(4)	123·9(14)	C(8)–C(14)–C(15)	121·8(13)
C(2)–C(3)–C(4)	120·2(12)	C(13)–C(14)–C(15)	101·0(11)
C(3)–C(4)–C(5)	113·1(14)	C(14)–C(15)–C(16)	103·3(13)
C(4)–C(5)–C(6)	108·8(13)	C(15)–C(16)–C(17)	106·1(13)
C(4)–C(5)–C(10)	110·0(14)	O(1)–C(17)–C(13)	110·2(12)
C(6)–C(5)–C(10)	112·4(12)	O(1)–C(17)–C(16)	112·7(12)
C(5)–C(6)–C(7)	110·9(13)	C(13)–C(17)–C(16)	105·8(13)
C(6)–C(7)–C(8)	109·6(12)		
C(7)–C(8)–C(9)	109·0(11)	C(17)–O(1)–C(7′)	113·6(12)
C(7)–C(8)–C(14)	105·7(12)	C(2′)–C(1′)–C(6′)	117·1(16)
C(9)–C(8)–C(14)	108·9(11)	C(2′)–C(1′)–C(7′)	120·3(14)
C(8)–C(9)–C(10)	111·7(11)	C(6′)–C(1′)–C(7′)	122·5(17)
C(8)–C(9)–C(11)	108·8(10)	C(1′)–C(2′)–C(3′)	123·2(13)
C(10)–C(9)–C(11)	114·0(11)	C(2′)–C(3′)–C(4′)	116·2(16)
C(1)–C(10)–C(5)	106·1(11)	Br–C(4′)–C(3′)	119·2(14)
C(1)–C(10)–C(9)	107·6(11)	Br–C(4′)–C(5′)	118·8(12)
C(1)–C(10)–C(19)	114·1(13)	C(3′)–C(4′)–C(5′)	121·6(16)
C(5)–C(10)–C(9)	105·8(12)	C(4′)–C(5′)–C(6′)	120·7(14)
C(5)–C(10)–C(19)	110·2(12)	C(1′)–C(6′)–C(5′)	120·7(20)
C(9)–C(10)–C(19)	112·3(11)	O(1)–C(7′)–O(2)	127·6(15)
C(9)–C(11)–C(12)	114·1(12)	O(1)–C(7′)–C(1′)	110·4(14)
C(11)–C(12)–C(13)	111·2(11)	O(2)–C(7′)–C(1′)	121·6(14)
C(12)–C(13)–C(14)	108·0(11)		

Intramolecular bond distances and angles are given in Table 3, together with their estimated standard deviations computed from the least-squares residuals. Mean estimated standard deviations are *ca.* 0·021 Å and 1·2°. Most observed bond distances and angles are comparable to those found in other 5α-androstane type steroids.[14-16] Two or three differences which appear to be significant in terms of the estimated standard

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* For details see Notice to Authors No. 7 in *J.C.S. Dalton*, 1972, Index issue.

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[15] C. M. Weeks, A. Cooper, and D. A. Norton, *Acta Cryst.*, 1971, *B*, **27**, 1562.

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deviations are more likely to indicate a slight underestimation of errors. The mean values of C–C single-bond distances in the steroid skeleton are 1·525 Å. The C–S bonds (1·775 and 1·788 Å) in the thiiran-ring are shorter than for a C–S single-bond distance (1·817 Å), but similar distances were observed in the recently determined structures of sulphur-containing heterocyclic compounds.[17-20] The bond distances in the *p*-bromobenzoate group are as expected: C–C ring system 1·378, C–C single bond 1·493, C–O 1·333, C=O 1·158, and C–Br 1·896 Å. These distances are in good agreement with those found in other *p*-bromobenzoate groups.[21-23] The C–H distances range from 0·63 to 1·24 Å, the mean of 0·96 Å being shorter than the internuclear separations (1·08 Å) obtained spectroscopically.[24]

The bond angles in the thiiran ring are as expected. The mean interbond angle in rings A, B, and C is 111·4°, which is slightly, but not significantly, greater than the normal tetrahedral angle of 109·4°. The angles in the five-membered ring D are very similar to the corresponding angles in the other steroids.[25-27] Maximum distortion of bond angles occurs at C(13) in ring D. The angle C(14)–C(13)–C(17) of *ca.* 99° is an indication of strain imposed at the C/D junction.

TABLE 4

Equations of planes and displacements (Å) of the atoms from mean planes; X, Y, Z are orthogonal co-ordinates (Å)

Plane (I): C(1)—(4)
$0{\cdot}8082X + 0{\cdot}5883Y - 0{\cdot}0238Z = 12{\cdot}9368$
C(1) −0·01, C(2) 0·02, C(3) −0·02, 3(4) 0·01, C(5) −0·44, C(10) 0·36

Plane (II): C(6), C(7), C(9), C(10)
$0{\cdot}8749X + 0{\cdot}3374Y - 0{\cdot}3473Z = 0{\cdot}8066$
C(5) −0·69, C(6) −0·01, C(8) 0·01, C(7) 0·81, C(9) −0·01, C(10) 0·01

Plane (III): C(8), C(11), C(12), C(14)
$0{\cdot}8366X + 0{\cdot}3661Y - 0{\cdot}4117Z = 8{\cdot}5236$
C(8) 0·02, C(9) −0·68, C(11) −0·02, C(12) 0·02, C(13) 0·71, C(14) 0·02

Plane (IV): C(15)—(17)
$0{\cdot}8195X + 0{\cdot}5324Y - 0{\cdot}2117Z = 10{\cdot}9273$
C(15) 0·00, C(16) 0·00, C(17) 0·00, C(13) 0·53, C(14) −0·25

Plane (V): C(1′)—(6′)
$0{\cdot}9554X - 0{\cdot}2734Y - 0{\cdot}1112Z = 8{\cdot}2962$
C(1′) 0·00, C(2′) 0·00, C(3′) −0·02, C(4′) 0·02, C(5′) −0·01, C(6′) 0·00, C(7′) 0·01, O(1) 0·51, O(2) −0·28, Br −0·03

The displacements of atoms from some least-squares mean planes are listed in Table 4. The benzene ring is planar, and ring A of the steroid skeleton has a half-chair conformation. The atoms C(1)—(4) are nearly coplanar and atoms C(5) and C(10) are displaced by −0·44 and 0·36 Å to opposite sides of this plane. Rings B and C are in the normal chair form, while ring D has a conformation somewhere between a half-chair and a β envelope.

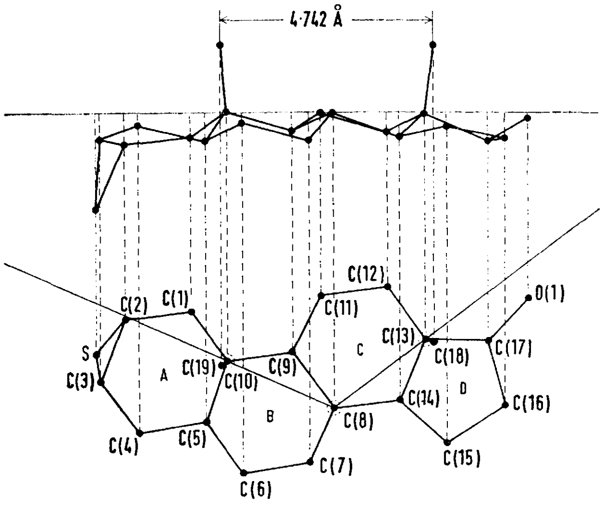

FIGURE 2 Projection of the steroid nucleus on the C(10), C(8), and C(13) plane

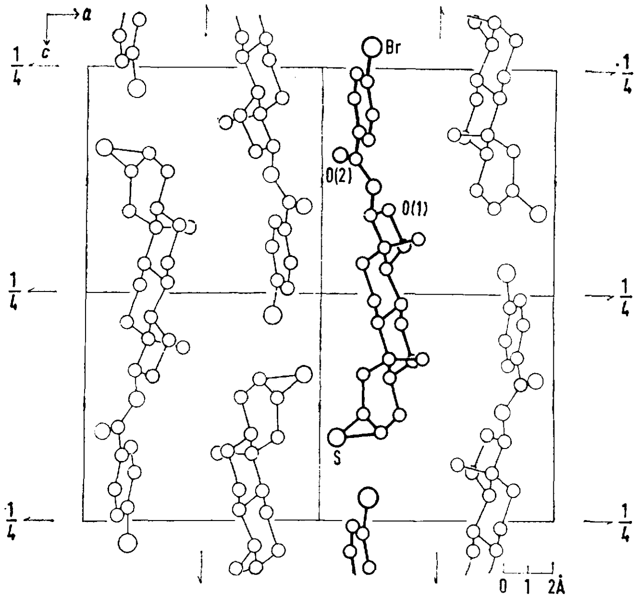

FIGURE 3 The structure viewed down the *b* axis

In order to obtain detailed information on the molecular shape and the conformational features we have calculated the displacements from the planes through C(8), C(10), and C(13) in the steroid skeleton (Figure 2). The presence of the thiiran ring does not significantly affect the overall planarity of the steroid

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skeleton. Indeed, the distance of 4·742 Å between the C(18) and C(19) axial methyl groups is comparable to that found in other steroids.[4,28] However, it may be concluded that introduction of the thiiran ring has some influence on the conformation of ring A. The dihedral angle between the thiiran ring and the least-squares plane through atoms C(1)—(4) is *ca.* 67°. The torsion angle H(2)–C(2)–C(3)–H(3) is *ca.* 27°. The width of the steroid skeleton, calculated as the distance between C(3) and O(1), is 9·744 Å.

The packing of molecules in the unit-cell is illustrated in Figure 3. The average plane of the steroid skeleton is nearly parallel to the *bc* plane of the cell. All intermolecular distances were calculated, and the most significant contacts (<3·60 Å) are given in Table 5. There is no hydrogen bonding in the crystal, the shortest contact being S · · · C(1^{I}) 3·501 Å. Other intermolecular contacts are greater than the sum of the van der Waals radii.

TABLE 5

Intermolecular distances (<3·60 Å)

S · · · C(1^{I})	3·501	C(19) · · · C($2'^{II}$)	3·562
C(3′) · · · O(2^{III})	3·518		

Roman numerals as superscripts refer to the following equivalent positions, relative to the reference molecule at x, y, z:

I $1 - x\ y - \frac{1}{2}, \frac{3}{2} - z$ III $x, 1 + y, z$
II $\frac{3}{2} - x, 2 - y, \frac{1}{2} + z$

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