maximum deviation from the least-squares plane of 0·004 Å. The four substitutents S(1), C(4), C(8) and C(14) are at 0·065 (1), 0·013 (3), −0·090 (3) and −0·083 (4) Å from the best plane. C(14)—N(2), C(2)—C(4), N(1)—C(8) are 1·458 (4), 1·491 (4) and 1·435 (4) Å. The average angles C—N—C, N—C—C are 110·2 (3) and 107·2 (3)°. The N—C—N angle is 105·3 (2)°.

Thiotetrafuranose ring. This compound represents one of the first structural studies of a thiofuranose nucleoside and as expected the substitution of S for O affects markedly the conformation of this ring. The average C—C bond length is 1·522 (5) Å and the distances S(2)—C(4) and S(2)—C(7) are equal and correspond to the S—C value given for a single bond. The average C—C—C, C—C—S and C—S—C endocyclic angles are 106·9 (2), 105·6 (2) and 94·8 (2)°. The thiofuranose ring is not planar. In terms of ring-puckering coordinates (Cremer & Pople, 1975) the amplitude phase magnitudes are q = 0·427 (4) Å and φ = 98·2 (4)° for the sequence S(2)—C(4)—C(5)—C(6)—C(7) and the resulting conformation is intermediate between twist ${}^{2}_{3}T$ and envelope E_3. The phenyl–imidazole, and thiofuranose–imidazole dihedral angles are 46·7 (1) and 52·9 (1)°. These values are rather lower (values range from 60 to 80°) and could indicate a large contribution of crystal forces to the phenyl–imidazole and thiofuranose–imidazole subrotations (Moreno, López-Castro & Márquez, in the press). Table 2 shows the main torsion angles.

The crystal packing, as viewed along the c axis is shown in Fig. 2; it is maintained by van der Waals contacts.

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Structure of an Optically Pure Steroidal Methanesulfinate*

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Abstract. 3-Oxo-*A*-norandrostane-5β,17β-diol 17-acetate 5-[(*R*)-methanesulfinate], $C_{21}H_{32}O_5S$, M_r = 396·55, monoclinic, $P2_1$, a = 12·475 (4), b = 7·203 (1), c = 11·510 (4) Å, β = 90·80 (2)°, V = 1034·2 Å^3, Z = 2, D_m = 1·282, D_x = 1·273 Mg m^{-3}, Cu $K\alpha$, $\lambda(K\alpha_1)$ = 1·54056 Å, μ = 1·582 mm^{-1}, $F(000)$ = 428, T = 293 K, R = 0·029 for 2239 reflexions with $I \geq 2{\cdot}0\sigma(I)$. The ring junction *A*/*B* is *cis*. The geometry around the S atom, with the lone pair of electrons, is tetrahedral with the *R* chirality. The molecules are linked by C—H···O=S hydrogen bonds into chains along **b**.

Introduction. Derivatives of *A*-norandrostane-5,17α-diol were synthesized (Edwards & Grue-Sorensen, 1985) in order to study the behaviour of α-acyl carbocations in steroids (Edwards & Paryzek, 1983) and explore the limits to apparent S_N2 displacement at tertiary C atoms adjacent to carbonyl groups (Edwards

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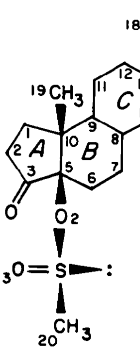
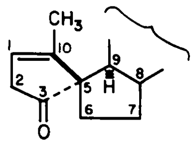
& Grieco, 1974). The 5-methanesulfonate could only be prepared by oxidation of the methanesulfinate (1) (Creary & Geiger, 1982). The configuration at C(5) had been deduced from the observation of contraction of ring *B* in the methanesulfonate. The spiro compound (2) which was formed would result from migration of the 9,10 bond antiparallel to the 5β C—O bond being ruptured.

It was considered useful to check this configuration and determine by X-ray analysis the chirality of S in the title methanesulfinate.

To the best of our knowledge this is the first pure chiral methanesulfinate to be characterized. The potential exists for preparing from this compound simpler chiral methanesulfinates as standards for optical activity.



(1)



(2)

Experimental. Crystal density by flotation in carbon tetrachloride and hexane solution. Prismatic crystal 0·25 × 0·27 × 0·29 mm. Enraf–Nonius CAD-4 diffractometer, Ni-filtered Cu radiation. Cell parameters by least squares on θ measurements of 23 reflexions with $37 < \theta < 51°$ and α and γ constrained to 90°. Intensities measured for hkl and $\bar{h}kl$ by ω–2θ scans to $2\theta = 150°$; h −15→15, k 0→9, l 0→14; $\Delta\omega = (0{\cdot}8 + 0{\cdot}14\tan\theta)°$ extended by 25% at each side for the background; horizontal aperture width $(5{\cdot}0 + 0{\cdot}4\tan\theta)$ mm; ω-scan speed 0·53 to 3·35° min^{-1}. Three standard reflexions, measured every hour of exposure time, decreased by 7% throughout the experiment. 2297 independent reflexions measured, 2239 observed with $I \geq 2{\cdot}0\sigma(I)$. Corrections for background, scale, and Lorentz and polarization, but not for absorption. Structure determination by the application of *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and Fourier maps. H positions from difference maps. Refinement by block-diagonal least squares, 9 × 9 per atom (4 × 4 for H), minimizing $\sum w(|F_o| - |F_c|)^2$ with $w^{-1} = 1 + (|F_o|/20)^2$. The y coordinate of S was fixed for origin definition. Convergence at $R = 0{\cdot}029$ for the observed reflexions, $wR = 0{\cdot}031$, $S = 0{\cdot}28$. $(\Delta/\sigma)_{\text{ave}} = 0{\cdot}05$, $(\Delta/\sigma)_{\max} = 0{\cdot}55$ [for H(22,1)]. Residual electron density within −0·24 and 0·12 e Å^{-3}. Reflexion $\bar{2}01$, which showed extinction effect, was excluded from later stages of refinement. Scattering-factor curves from *International Tables for X-ray Crystallography* (1974) and Stewart, Davidson & Simpson (1965) for H. Computations with *NRC* programs (Ahmed, Hall, Pippy & Huber, 1973).

Table 1. *Atomic coordinates* (× 10^5 *for* S; × 10^4 *for* O *and* C) *and equivalent isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

$$B_{\text{eq}} = \tfrac{8}{3}\pi^2 \textstyle\sum_i \sum_j U_{ij} a^*_i a^*_j \mathbf{a}_i . \mathbf{a}_j .$$

	x	y	z	B_{eq}
S	91973 (4)	58664	89105 (4)	3·7
O(1)	10318 (1)	2819 (3)	7119 (2)	4·8
O(2)	8223 (1)	4808 (3)	8179 (1)	3·7
O(3)	9750 (2)	4443 (3)	9618 (1)	5·7
O(4)	6347 (1)	−117 (3)	1178 (1)	4·3
O(5)	6286 (2)	−3007 (3)	1856 (2)	5·9
C(1)	7720 (2)	1052 (4)	7781 (2)	4·1
C(2)	8927 (2)	772 (4)	7831 (2)	4·5
C(3)	9393 (2)	2521 (3)	7328 (2)	3·5
C(5)	8467 (2)	3884 (3)	7072 (2)	3·1
C(6)	8697 (2)	5295 (3)	6138 (2)	3·6
C(7)	8780 (2)	4367 (3)	4949 (2)	3·7
C(8)	7792 (2)	3203 (3)	4664 (2)	3·0
C(9)	7615 (2)	1719 (3)	5613 (2)	3·1
C(10)	7512 (2)	2572 (3)	6850 (2)	3·3
C(11)	6678 (2)	412 (4)	5302 (2)	4·1
C(12)	6742 (2)	−432 (4)	4079 (2)	4·0
C(13)	6876 (1)	1087 (3)	3167 (2)	3·2
C(14)	7885 (2)	2215 (3)	3497 (2)	3·1
C(15)	8126 (2)	3360 (4)	2410 (2)	3·9
C(16)	7768 (2)	2107 (4)	1404 (2)	4·6
C(17)	7222 (2)	422 (3)	1960 (2)	3·7
C(18)	5867 (2)	2292 (4)	3049 (2)	4·0
C(19)	6433 (2)	3533 (5)	6997 (2)	4·7
C(20)	8277 (2)	6908 (4)	9859 (2)	4·4
C(21)	5979 (2)	−1872 (4)	1202 (2)	4·5
C(22)	5131 (3)	−2175 (6)	291 (3)	7·4

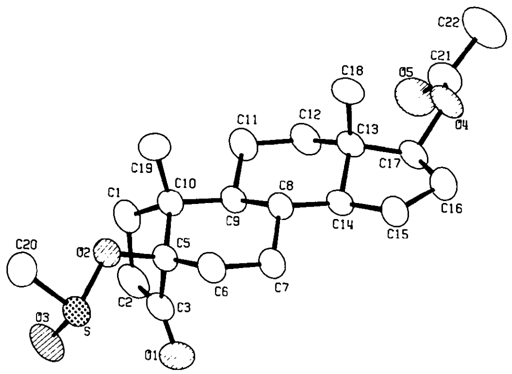


Fig. 1. *ORTEP* (Johnson, 1965) plot showing 50% probability thermal ellipsoids.

Table 2. *Interatomic distances* (Å) *and valence angles* (°)

S–O(2)	1·655 (2)	O(2)–C(5)–C(3)	106·6 (2)
S–O(3)	1·474 (2)	O(2)–C(5)–C(6)	110·8 (2)
S–C(20)	1·763 (3)	O(2)–C(5)–C(10)	104·6 (2)
O(1)–C(3)	1·201 (3)	C(3)–C(5)–C(6)	114·6 (2)
O(2)–C(5)	1·473 (3)	C(3)–C(5)–C(10)	102·5 (2)
O(4)–C(17)	1·458 (3)	C(6)–C(5)–C(10)	116·7 (2)
O(4)–C(21)	1·345 (3)	C(5)–C(6)–C(7)	111·1 (2)
O(5)–C(21)	1·172 (3)	C(6)–C(7)–C(8)	111·7 (2)
C(1)–C(2)	1·519 (4)	C(7)–C(8)–C(9)	110·6 (2)
C(1)–C(10)	1·551 (3)	C(7)–C(8)–C(14)	112·0 (2)
C(2)–C(3)	1·507 (4)	C(9)–C(8)–C(14)	108·2 (2)
C(3)–C(5)	1·541 (3)	C(8)–C(9)–C(10)	112·8 (2)
C(5)–C(6)	1·510 (3)	C(8)–C(9)–C(11)	112·0 (2)
C(5)–C(10)	1·539 (3)	C(10)–C(9)–C(11)	112·4 (2)
C(6)–C(7)	1·528 (3)	C(1)–C(10)–C(5)	101·4 (2)
C(7)–C(8)	1·523 (3)	C(1)–C(10)–C(9)	109·8 (2)
C(8)–C(9)	1·546 (3)	C(1)–C(10)–C(19)	112·5 (2)
C(8)–C(14)	1·526 (3)	C(5)–C(10)–C(9)	108·7 (2)
C(9)–C(10)	1·558 (3)	C(5)–C(10)–C(19)	112·7 (2)
C(9)–C(11)	1·539 (4)	C(9)–C(10)–C(19)	111·4 (2)
C(10)–C(19)	1·525 (4)	C(9)–C(11)–C(12)	114·0 (2)
C(11)–C(12)	1·536 (3)	C(11)–C(12)–C(13)	110·7 (2)
C(12)–C(13)	1·527 (3)	C(12)–C(13)–C(14)	107·8 (2)
C(13)–C(14)	1·541 (3)	C(12)–C(13)–C(17)	115·7 (2)
C(13)–C(17)	1·537 (3)	C(12)–C(13)–C(18)	111·6 (2)
C(13)–C(18)	1·533 (3)	C(14)–C(13)–C(17)	98·5 (2)
C(14)–C(15)	1·532 (3)	C(14)–C(13)–C(18)	113·0 (2)
C(15)–C(16)	1·530 (4)	C(17)–C(13)–C(18)	109·7 (2)
C(16)–C(17)	1·536 (4)	C(8)–C(14)–C(13)	113·0 (2)
C(21)–C(22)	1·495 (4)	C(8)–C(14)–C(15)	119·1 (2)
O(2)–S–O(3)	107·2 (1)	C(13)–C(14)–C(15)	104·6 (2)
O(2)–S–C(20)	91·8 (1)	C(14)–C(15)–C(16)	104·0 (2)
O(3)–S–C(20)	104·9 (1)	C(15)–C(16)–C(17)	106·1 (2)
S–O(2)–C(5)	119·3 (1)	O(4)–C(17)–C(13)	115·1 (2)
C(17)–O(4)–C(21)	119·4 (2)	O(4)–C(17)–C(16)	106·6 (2)
C(2)–C(1)–C(10)	106·0 (2)	C(13)–C(17)–C(16)	105·2 (2)
C(1)–C(2)–C(3)	105·2 (2)	O(4)–C(21)–O(5)	124·1 (2)
O(1)–C(3)–C(2)	127·1 (2)	O(4)–C(21)–C(22)	111·2 (2)
O(1)–C(3)–C(5)	124·6 (2)	O(5)–C(21)–C(22)	124·8 (3)
C(2)–C(3)–C(5)	108·3 (2)		

Table 3. *Endocyclic torsion angles* (°)

Ring *A*	
C(10)–C(1)–C(2)–C(3)	21·0 (3)
C(1)–C(2)–C(3)–C(5)	4·3 (3)
C(2)–C(3)–C(5)–C(10)	−27·8 (2)
C(3)–C(5)–C(10)–C(1)	39·2 (2)
C(5)–C(10)–C(1)–C(2)	−37·9 (2)
Ring *B*	
C(10)–C(5)–C(6)–C(7)	−51·7 (3)
C(5)–C(6)–C(7)–C(8)	53·9 (3)
C(6)–C(7)–C(8)–C(9)	−57·1 (3)
C(7)–C(8)–C(9)–C(10)	56·7 (3)
C(8)–C(9)–C(10)–C(5)	−51·0 (3)
C(9)–C(10)–C(5)–C(6)	49·6 (3)
Ring *C*	
C(14)–C(8)–C(9)–C(11)	−52·2 (3)
C(8)–C(9)–C(11)–C(12)	50·8 (3)
C(9)–C(11)–C(12)–C(13)	−53·2 (3)
C(11)–C(12)–C(13)–C(14)	56·7 (2)
C(12)–C(13)–C(14)–C(8)	−62·8 (2)
C(13)–C(14)–C(8)–C(9)	60·0 (2)
Ring *D*	
C(17)–C(13)–C(14)–C(15)	45·7 (2)
C(13)–C(14)–C(15)–C(16)	−33·1 (2)
C(14)–C(15)–C(16)–C(17)	6·5 (3)
C(15)–C(16)–C(17)–C(13)	22·3 (2)
C(16)–C(17)–C(13)–C(14)	−41·2 (2)

Discussion. The refined atomic parameters are listed in Table 1.* A parallel projection of the molecule, drawn in the absolute configuration assigned on the basis of that of the parent compound, is presented in Fig. 1. The bond lengths and angles, not corrected for thermal vibration, are shown in Table 2.

The longest C(sp^3)—C(sp^3) bonds of 1·551 (3) and 1·558 (3) Å are to the fully saturated C(10) atom; the shortest [C(5)—C(6) 1·510 (3) Å] is influenced by the adjacent O(2) atom. The bond lengths and angles of the acetate group are very similar to those found in other androstane derivatives (Jones & Karle, 1974; Duax, Eger, Pokrywiecki & Osawa, 1971). The angle O(4)—C(17)—C(13) is appreciably larger than O(4)—C(17)—C(16) [115·1 (2) *vs* 106·6 (2)°]. This difference may be caused by the repulsion between O(4) and H(18,2) as the distance between these two atoms is only 2·59 (2) Å. The C—H bonds range from 0·89 to 1·09 (σ = 0·02–0·04) Å.

The ring junction *A*/*B* is *cis*, while *B*/*C* and *C*/*D* are *trans*. Ring *A* has an envelope conformation with the lowest asymmetry parameter ΔC_s[C(10)] = 4·9° (Duax, Weeks & Rohrer, 1976). Ring *B* has an asymmetry parameter of 2·1° for a chair form with atoms C(5) and C(8) situated at −0·581 (2) and 0·677 (2) Å, respectively, from the plane of the other four atoms. The ring is slightly flattened with torsional angles 49·6 (3)–57·1 (3)°. The endocyclic torsion angles are given in Table 3. Ring *C* also has a chair conformation; its asymmetry parameter is 3·4°, with C(11) at −0·614 (3) and C(14) at 0·706 (2) Å from the plane of the remaining atoms. Ring *D* is more distorted than ring *A* and is intermediate between the 13β-envelope form (ΔC_s = 8·3°) and the half-chair form {ΔC_2[C(13)—C(14)] = 12·6°}. C(13) and C(14) are 0·562 (2) and −0·168 (2) Å, respectively, from the plane through C(15), C(16) and C(17).

The S lone pair could not be detected on the last difference map, but approximate tetrahedral geometry is indicated. The S—C(20) bond is almost perpendicular to S—O(2), O(2)—S—C(20) 91·8 (1)°, but the other two angles at S approach tetrahedral values [104·9 (1) and 107·2 (1)°]. Similar stereochemistry was found in (±)-3-bis(phenylsulfinyl)methyl-1,2-dimethylcyclopropene (Beckhaus, Kimura, Watson, Venier & Kojić-Prodić, 1979) and in *cis*-2-ethoxy-1-phenylsulfinylcyclopropane (Kimura, Ward, Watson & Venier, 1979), where the C—S—C angles are appreciably smaller [94·8 (4)–100·2 (2)°] than the C—S=O angles which range from 105·1 (4) to 108·7 (5)°. The S=O bond of 1·474 (2) Å is slightly shorter than the

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* Lists of observed and calculated structure amplitudes, anisotropic thermal parameters and H parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42289 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

corresponding bond lengths found in the above-mentioned structures [1·487 (4), 1·508 (4) and 1·509 (8) Å].

The polarization of the S=O(3) bond enhances the participation of O(3) as an acceptor, and that of C(20), which is attached to S, as a donor in the formation of the intermolecular C—H···O=S bond. The H(20,3)···O(3′) distance is 2·29 (4) Å, C(20)—H(20,3)···O(3′) 137 (3)° and C(20)—H(20,3) 1·02 (4) Å. These values lie in the middle of the range of those compiled for a number of C—H···O bonds (Taylor & Kennard, 1982). The molecules joined by these bonds form chains along the *b* axis. The next shortest intermolecular contact is H(22,2)···H(22,3′) = 2·32 (5) Å and all other H···H contacts are ≥ 2·48 Å.

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Structure of 5-Methyl-2-thiocytosine Hemihydrate

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Abstract. $C_5H_7N_3S.\frac{1}{2}H_2O$, $M_r = 150{\cdot}2$, orthorhombic, $P2_12_12$, $a = 9{\cdot}9233$ (4), $b = 12{\cdot}9693$ (8), $c = 5{\cdot}4094$ (3) Å, $V = 696{\cdot}18$ (6) Å^3, $Z = 4$, $D_m = 1{\cdot}42$, $D_x = 1{\cdot}433$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}7107$ Å, $\mu = 0{\cdot}333$ mm^{-1}, $F(000) = 316$, $T = 297$ K, $R = 0{\cdot}049$ for 1565 observed reflections. The sulfur atom acts as an acceptor of N—H···S and O*W*—H···S hydrogen bonds. The water oxygen sits on a twofold axis along **c** and acts as a donor of two symmetry-related O*W*—H···S hydrogen bonds and at the same time acts as an acceptor of two symmetry-related N—H···O*W* hydrogen bonds. The environment around O*W* is nearly tetrahedral.

Introduction. Methylated cytosine derivatives like 5-methylcytosine and 5-methyl-2-thiocytosine (5M2TC) are of considerable biological importance, since they have long been known to be definite constituents of DNA and RNA (Rink, 1974). Some of these cytosine derivatives seem to regulate nucleic-acid structures by affecting base-pair formation as exemplified in the clover-leaf arrangement of tRNA (Inose, Miyata &

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