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A New Cardenolide: 5β-Hydroxygitoxigenin†

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Abstract. $C_{23}H_{34}O_6$, orthorhombic, $P2_12_12_1$, a = 11·290 (2), b = 24·638 (2), c = 7·122 (4) Å, V = 1981·1 Å^3, Z = 4, D_x = 1·363, D_m = 1·358 Mg m^{-3}. The structure was refined to R = 0·030 for 1847 observed reflexions. Its stereochemistry is similar to that of digitoxigenin, with the two terminal rings *cis* fused to the two central rings. The five-membered ring *D* assumes the α-envelope conformation at C(15). The X-ray analysis confirms the molecular formula and stereochemistry as determined by chemical and spectroscopic methods. There are two inter- and two intramolecular O—H···O bonds.

Introduction. New cardenolides are currently being prepared by chemical and microbiological methods in a search for active but less toxic compounds. A viable alternative method involves bioconversion by the use of plant cell suspension cultures. Recently, 5β-hydroxygitoxigenin has been isolated by I. A. Veliky and A. Jones at the National Research Council of Canada from cultures of *Daucus carota* Ca68 incubated with gitoxigenin. The compound has been identified and its chemical formula assigned on the basis of infrared, NMR and mass spectra. The present X-ray analysis was undertaken to establish the stereochemistry of this novel compound with OH substituents at C(3), C(5), C(14) and C(16). A summary of the biochemical and crystallographic results has been reported by Veliky, Jones, Przybylska & Ahmed (1979).

Colourless prismatic crystals, m.p. 507–509 K (with decomposition), were obtained by evaporation of a solution in ethanol and water and had to be grown to an appreciable length (>2 mm) to get a specimen of satisfactory thickness. A fragment 0·17 × 0·23 × 0·30 mm was mounted along c^* which is the prism axis. The space group was established from precession films, and the data were measured on a Picker four-circle automatic diffractometer with Ni-filtered Cu radiation [$\lambda(K\alpha_1)$ = 1·54050, $\lambda(K\alpha_2)$ = 1·54434 Å]. The cell parameters were derived from the angular settings of eight reflexions (2θ = 66 to 110°) and their equivalents.

Table 1. *Fractional coordinates* (×10^4) *for non-hydrogen atoms and* B_{eq} (Å^2) (= *average of three principal axes of the thermal ellipsoids*)

	x	y	z	B_{eq}
O(1)	7174 (2)	5019 (1)	3139 (3)	5·02
O(2)	9025 (2)	5305 (1)	2940 (3)	5·35
O(3)	6024 (1)	−9 (1)	2079 (3)	4·20
O(4)	8296 (1)	358 (0)	1701 (2)	3·00
O(5)	9256 (1)	2882 (1)	257 (2)	2·79
O(6)	7982 (2)	3703 (1)	−1365 (2)	3·45
C(1)	6836 (2)	949 (1)	4414 (3)	2·74
C(2)	5634 (2)	869 (1)	3482 (4)	3·17
C(3)	5748 (2)	543 (1)	1692 (4)	3·23
C(4)	6651 (2)	802 (1)	382 (3)	2·70
C(5)	7861 (2)	898 (1)	1287 (3)	2·21
C(6)	8707 (2)	1169 (1)	−92 (3)	2·68
C(7)	8427 (2)	1767 (1)	−449 (3)	2·51
C(8)	8313 (2)	2095 (1)	1362 (3)	2·07
C(9)	7402 (2)	1827 (1)	2672 (3)	1·97
C(10)	7763 (2)	1229 (1)	3151 (3)	2·21
C(11)	7166 (2)	2174 (1)	4410 (3)	2·75
C(12)	6777 (2)	2739 (1)	3811 (3)	2·46
C(13)	7723 (2)	3050 (1)	2667 (3)	2·13
C(14)	8114 (2)	2702 (1)	941 (3)	1·95
C(15)	7179 (2)	2839 (1)	−536 (3)	2·46
C(16)	7028 (2)	3452 (1)	−379 (3)	2·68
C(17)	7062 (2)	3554 (1)	1779 (3)	2·28
C(18)	8761 (2)	3189 (1)	3953 (4)	3·11
C(19)	8943 (2)	1213 (1)	4232 (4)	3·00
C(20)	7441 (2)	4117 (1)	2286 (3)	2·30
C(21)	6536 (2)	4519 (1)	2873 (5)	4·39
C(22)	8478 (2)	4371 (1)	2275 (4)	3·06
C(23)	8327 (2)	4932 (1)	2792 (4)	3·49

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Intensities were measured by the θ–2θ scan method at 3·5° take-off angle, while the background was obtained by counting for 20 s at the lower and upper limits of each scan. The 402 and 0,12,0 reflexions were used as standards for scaling. Net intensities were corrected for Lorentz and polarization effects but not for absorption [$\mu(\mathrm{Cu}) = 0{\cdot}750\ \mathrm{mm}^{-1}$]. Of 1965 accessible ($2\theta_{\max} = 130°$) unique reflexions scanned, 1847 (94%) were observed above threshold.

The structure was solved with the tangent formula (Karle & Hauptman, 1956). The starting set of phases consisted of four origin- and enantiomorph-defining reflexions, and four others with trial phase values. The phase set selected had $R_K = 0{\cdot}22$ for the 109 reflexions with $|E| \geq 1{\cdot}8$, and $R_K = 0{\cdot}26$ for the 222 reflexions with $|E| \geq 1{\cdot}5$. The resulting E map, evaluated with 197 E's, revealed all the atoms, but the weakest four peaks were excluded until their positions were confirmed from a Fourier map. All the H atoms were located from a difference map computed at a later stage.

Refinement was by block-diagonal least squares, minimizing $\sum w(|F_o| - |F_c|)^2$, where $w = \{1 + [(|F_o| - 25)/25]^4\}^{-1}$, and excluding the unobserved and four strong reflexions which showed extinction effects. In the final cycle, $R = 0{\cdot}030$ for the observed reflexions and $R_w = 0{\cdot}030$. Mean and maximum shifts/e.s.d. for the parameters of the non-hydrogen atoms were 0·11 and 0·49, respectively. The final difference map showed a residual distribution from −0·27 to 0·21 e Å^{-3}. The atomic parameters are presented in Tables 1 and 2.[*] All calculations were carried out with the NRC programs (Ahmed, Hall, Pippy & Huber, 1973). The scattering factors were those of Hanson, Herman, Lea & Skillman (1964), except for H (Stewart, Davidson & Simpson, 1965).

Table 2. *Fractional coordinates* ($\times 10^3$) *for hydrogen atoms and the isotropic temperature factors* (Å^2)

	x	y	z	B
H(O3)	675 (3)	−2 (1)	233 (5)	6·4 (0·8)
H(O4)	899 (3)	37 (1)	174 (4)	5·8 (0·8)
H(O5)	921 (2)	315 (1)	−31 (4)	4·0 (0·6)
H(O6)	783 (2)	403 (1)	−149 (4)	5·2 (0·7)
H(1,1)	678 (2)	115 (1)	563 (3)	1·8 (0·4)
H(1,2)	712 (2)	61 (1)	478 (3)	2·3 (0·5)
H(2,1)	510 (2)	68 (1)	429 (4)	3·3 (0·6)
H(2,2)	527 (2)	122 (1)	318 (3)	2·9 (0·5)
H(3)	497 (2)	53 (1)	104 (4)	3·5 (0·6)
H(4,1)	677 (2)	58 (1)	−67 (4)	3·7 (0·6)
H(4,2)	632 (2)	117 (1)	−10 (3)	2·3 (0·5)
H(6,1)	956 (2)	114 (1)	44 (4)	3·0 (0·5)
H(6,2)	872 (2)	95 (1)	−125 (3)	2·9 (0·5)
H(7,1)	766 (2)	179 (1)	−121 (3)	3·0 (0·5)
H(7,2)	905 (2)	195 (1)	−127 (3)	2·8 (0·5)
H(8)	909 (2)	207 (1)	199 (3)	1·6 (0·4)
H(9)	667 (2)	180 (1)	197 (3)	1·6 (0·4)
H(11,1)	652 (2)	201 (1)	518 (3)	2·5 (0·5)
H(11,2)	791 (2)	219 (1)	521 (4)	3·6 (0·6)
H(12,1)	651 (2)	295 (1)	492 (3)	2·5 (0·5)
H(12,2)	602 (2)	271 (1)	297 (3)	2·5 (0·5)
H(15,1)	640 (2)	264 (1)	−29 (4)	3·1 (0·5)
H(15,2)	743 (2)	275 (1)	−177 (3)	1·9 (0·4)
H(16)	628 (2)	358 (1)	−87 (4)	3·1 (0·5)
H(17)	625 (2)	352 (1)	224 (3)	2·5 (0·5)
H(18,1)	936 (2)	340 (1)	330 (4)	3·4 (0·5)
H(18,2)	853 (2)	341 (1)	503 (4)	4·0 (0·6)
H(18,3)	914 (2)	285 (1)	444 (4)	3·3 (0·6)
H(19,1)	960 (2)	138 (1)	356 (4)	4·1 (0·6)
H(19,2)	913 (2)	84 (1)	465 (4)	3·6 (0·6)
H(19,3)	884 (2)	143 (1)	544 (4)	4·1 (0·6)
H(21,1)	618 (2)	440 (1)	410 (4)	5·2 (0·7)
H(21,2)	591 (2)	456 (1)	183 (4)	5·3 (0·7)
H(22)	926 (2)	425 (1)	193 (4)	4·0 (0·6)

Discussion. A parallel projection of the molecule produced by Johnson's (1965) *ORTEP* program, showing 50% probability thermal ellipsoids, is given in Fig. 1. It represents the absolute configuration assigned on the basis of that of the parent compound gitoxigenin and the comparison of the optical activity of closely related cardenolides (Veliky *et al.*, 1979).

The bond lengths and angles, not corrected for thermal vibration, are shown in Fig. 2. The C(sp^3)–C(sp^3) values vary from 1·513 to 1·581 Å with mean 1·538 Å. The longest bonds [C(9)–C(10), C(13)–C(17), C(5)–C(10) and C(13)–C(14)] are associated with highly substituted C atoms. C(17)–C(20) and C(20)–C(21) are shortened to 1·496 and 1·482 Å respectively, as expected for the C(sp^3)–C(sp^2) type. The shortest C–C bond is between C(22) and C(23), and is in perfect agreement with the C–C value in C=C–C=O given by Sutton (1965). The C=C, C–O and C=O bonds do not deviate significantly from the corresponding values listed in *Molecular Structures and Dimensions* (1972) with the exception of O(1)–C(23) (1·343 Å), which is shortened through the influence of the adjacent C=O group. The C–O bonds fall into two categories. C(3)–O(3) and C(16)–O(6) are 1·424 and 1·427 Å respectively and are considered to be of normal length. However, C(5)–O(4) and C(14)–O(5) are significantly elongated (1·449 and 1·448 Å), probably as a result of the strain of attach-

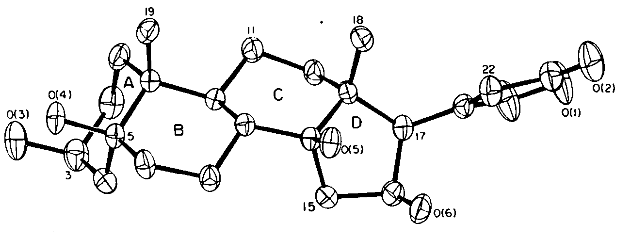

Fig. 1. Drawing of the molecule showing vibration ellipsoids.

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[*] Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34550 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

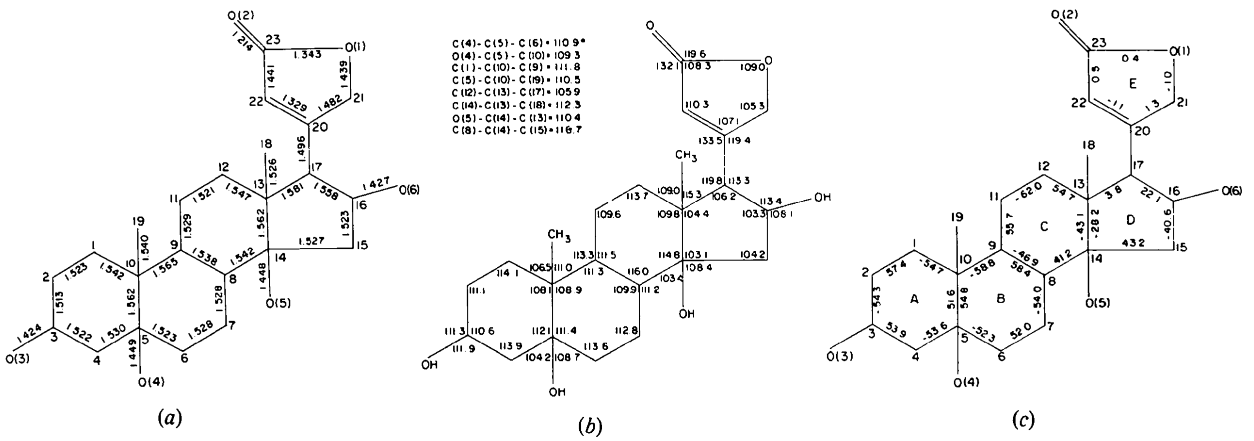

Fig. 2. (*a*) Bond lengths (Å). The e.s.d.'s vary from 0·002 to 0·004 Å. (*b*) Valency angles (°); the e.s.d. is <0·3°. (*c*) Endocyclic torsion angles (°); the e.s.d. is <0·6°.

ment to fully substituted C atoms. The C—H bonds vary from 0·94 to 1·04 Å with a mean of 0·99 Å, and the C—C—H and H—C—H angles range from 103·4 to 131·1°.

The ring junctions are *A*/*B cis*, *B*/*C trans* and *C*/*D cis*. The torsion angles within the rings are presented in Fig. 2(*c*), where the signs are according to the convention of the IUPAC–IUB Commission on Biochemical Nomenclature (1970). The torsion angle C(13)–C(17)–C(20)–C(22) is 52·3°. Some mean planes and the displacements of atoms from them are listed in Table 3.

Rings *A*, *B* and *C* are in the chair conformation, *C* being slightly more distorted than the other two due to the strain of the junction with the five-membered ring and the substitution at C(13) and C(14).

The stereochemistry of 5β-hydroxygitoxigenin is very similar to that of digitoxigenin (Karle & Karle, 1969). The fact that in both structures the unsaturated C(20)=C(22) bond lies as near as possible to the OH substituents may be due in part to the formation of a hydrogen bond between one of the hydroxyl groups and the double bond. The existence of such bonds in unsaturated alcohols is well documented (Schleyer, Trifan & Bacskai, 1958; Paddon-Row, Patney & Warrener, 1978). In digitoxigenin the distance between the mid-point of the C(20)=C(22) bond and O, which is attached to C(14), is 3·22 Å. The H atom attached to that O was not located, but since it is not clear that it is involved in an intermolecular hydrogen bond, it may be directed towards the π MO's of the double bond. In 5β-hydroxygitoxigenin the corresponding distance of the centre of the double bond (*M*) to O(5) is considerably larger (3·94 Å), but to O(6) it is 2·92 Å. H(O6) is at a distance of 2·74 Å from *M* and its orientation is favourable for H···π bonding.

Table 3. *Mean planes and atomic displacements*

(*a*) Parameters for the planes $lX + mY + nZ = p$, where X,Y,Z are orthogonal coordinates in Å

	l	m	n	p	χ^2
Ring *A*	−0·3446	0·7827	−0·5183	−1·8075	31·3
Ring *B*	−0·9659	0·1707	−0·1944	−8·3792	27·8
Ring *C*	−0·6698	0·0801	−0·7382	−6·6155	593·0
Ring *D*	0·8559	0·5110	−0·0798	11·1739	355·3
Ring *E*	−0·1459	0·2360	−0·9607	−0·4125	1·4
C(20)···O(2)	−0·1358	0·2363	−0·9621	−0·3120	7·7

(*b*) Deviations from the mean planes (Å), e.s.d. = 0·003 Å

Ring *A*		Ring *B*		Ring *C*	
C(2)	0·006	C(10)	−0·005	C(8)	0·025
C(3)	−0·006	C(5)	0·005	C(9)	−0·027
C(5)	0·006	C(8)	0·005	C(12)	0·026
C(10)	−0·006	C(7)	−0·005	C(13)	−0·025
C(1)*	−0·651	C(6)*	−0·611	C(11)*	−0·693
C(4)*	0·625	C(9)*	0·704	C(14)*	0·518

Ring *D*		Ring *E*		C(20)···O(2)	
C(13)	−0·022	C(21)	−0·002	C(20)	0·002
C(14)	0·014	C(22)	0·001	C(22)	−0·001
C(16)	−0·015	C(23)	−0·001	C(23)	−0·006
C(17)	0·022	O(1)	0·001	O(2)	0·003
C(15)*	−0·631	O(2)	0·000	C(17)*	0·079
		C(17)*	0·098		
		C(20)*	0·017		

* Atoms not included in the calculation of the plane.

The H(O6)···*M* distance indicates a decrease of only 0·16 Å from the sum of van der Waals radii of H (1·20 Å) and the double-bonded C atom (in ethylene: 1·70 Å; Bondi, 1964). The extension of the O(6)–H bond in the direction of the covalent bond from 0·83 Å to the neutron diffraction value of 0·97 Å does not bring about any reduction in the H···M distance due to the fact that ∠O—H···M is 94°. The hydrogen bond to the double bond is therefore weak and since H(O6) is also involved in forming an intermolecular hydrogen bond to O(1), we have here an asymmetric bifurcated hydrogen-bond system. Weak bifurcated O—H···O hydrogen bonds with very small differences in the H···O distances from the sum of the two van der Waals radii and O—H···O angles as small as 90° have been found in a number of carbohydrates as described in the precise X-ray and neutron diffraction studies by Takagi & Jeffrey (1977), Jeffrey, McMullan & Takagi (1977), and Newton, Jeffrey & Takagi (1979).

Spectroscopic work is in progress to characterize more quantitatively the intramolecular H···π bonds in cardenolides (Przybylska, Veliky & Gregory, 1979).

It is the additional OH substitution at C(16) in 5β-hydroxygitoxigenin that is probably responsible for the two differences observed in the stereochemistries of these two compounds. The first lies in the different conformation of ring *D* which in digitoxigenin assumes a β-envelope conformation at C(14). In 5β-hydroxygitoxigenin ring *D* has the α-envelope conformation at C(15), which is situated 0·63 Å below the plane formed by the other four atoms. The second difference lies in the orientation of the lactone ring, which in digitoxigenin is nearly parallel to C(13)—C(14), whereas in 5β-hydroxygitoxigenin it is more symmetrically placed with respect to C(13)—C(18) and C(16)—O(6) (Fig. 1).

The five-membered ring *D* can be characterized by the maximum angle of puckering $\varphi_m = 49{\cdot}7°$ and the phase angle of pseudo-rotation $\Delta = -99{\cdot}3°$ (Altona, Geise & Romers, 1968). The phase angle for digitoxigenin is also negative (−30·9°; *Terpenoids and Steroids*, 1974).

The lactone ring is nearly planar, with C(20) only 0·02 Å from the plane calculated for the other atoms (Table 3). The χ^2 value of 21·1 was obtained for a plane through the five atoms of ring *E*. The conjugated bonds C(20)=C(22)—C(23)=O(2) are planar as shown in Table 3, and C(17) is 0·08 Å from this plane.

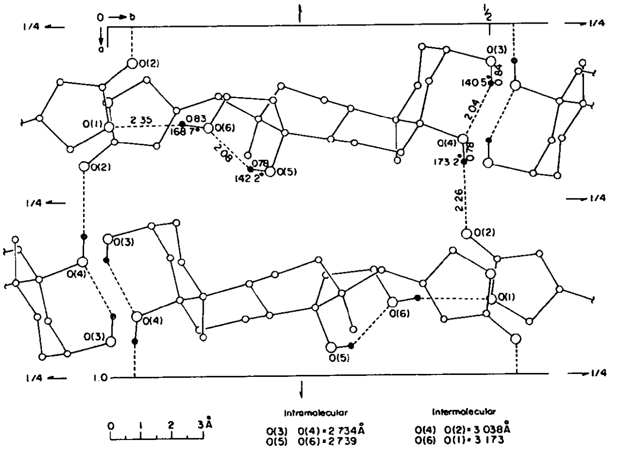

Fig. 3. Projection along *c* showing the hydrogen bonds. The e.s.d.'s are within 0·003 Å for O···O, 0·03 Å for O—H and H···O, and 3·0° for O—H···O.

The hydrogen bonds, together with the pertinent data, are shown in Fig. 3. There are two intramolecular hydrogen bonds: O(3)—H···O(4) and O(5)—H···O(6). The intermolecular bonds O(4)—H···O(2), which are parallel to *a*, form continuous spirals around the screw axes at $x = 0, \frac{1}{2}$, and $z = \frac{1}{4}$. These spirals of bonded molecules are interconnected by O(6)—H···O(1) bonds parallel to *b*. In addition the H atom attached to O(6) is directed towards O(2) of the neighbouring molecule, but the H···O(2) distance (2·69 Å) is too large for hydrogen bonding, and angle O(6)—H···O(2) is 139·7°.

The shortest O···H intermolecular contacts are between O(3) and H(6,2), H(19,2) and H(21,2), the distances being 2·62, 2·69 and 2·55 Å respectively. They can be considered as normal. All C···H closest approaches are acceptable, being above 2·91 Å. All H···H distances exceed 2·33 Å except H(16,1)···H(6,1) (2·09 Å). There are no other contacts shorter than the sums of the appropriate van der Waals radii.

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3α-Acetylthio-5α-androstan-17-one

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Abstract. $C_{21}H_{32}O_2S$, monoclinic, $C2$, a = 14·725 (8), b = 5·900 (3), c = 22·119 (10) Å, β = 92·28 (3)°, Z = 4, D_c = 1·206 Mg m^{-3}, Cu $K\alpha$ radiation (carbon monochromator), μ = 1·464 mm^{-1}, λ = 1·54051 Å. The structure was solved by an application of the Patterson function and the tangent formula and refined by a large-block least-squares procedure to R = 0·04 for 1452 reflections. The molecular configuration assigned from NMR data, with the S atom adopting the axial (3α) position, was confirmed.

Introduction. Thioacetates of triterpenes are found in unusual concentrations in the urine of young children who also exhibit inexplicably high blood pressure (Edwards & Trafford, 1968). The molecular configurations of these triterpenes have been established by ^{1}H NMR with reference to the thioacetates of the androsterones (Edwards, 1978). The configuration of these thioacetates, however, has not been established by X-ray crystallography and in order to confirm the NMR assignments we have determined the X-ray crystal structure of one of the reference compounds, 3α-acetylthio-5α-androstan-17-one. The crystals were provided by R. W. H. Edwards, Institute of Child Health, London, England.

The crystal system and approximate cell dimensions were determined from Weissenberg photographs. Systematic extinctions $h + k = 2n$ indicated that the space group was either $C2$, Cm or $C2/m$, of which $C2$ was chosen since the material was an optically active natural product. The cell dimensions were refined from the circle angles of 12 general reflections with the Bragg angle 2θ in the range 60–65°, and 1542 unique reflections were measured on a Picker FACS-1 four-circle diffractometer. Of these, 1452 had $I > 3\sigma(I)$. The data were reduced to a standard scale by the routine procedure (Cameron & Cordes, 1979) and were corrected for Lorentz and polarization factors, but not for absorption or extinction.

The positions of the S atom and the two C atoms bonded to it were established from a Patterson function. The positions of the three atoms were used to find approximate phases for the ten reflections with the largest E's. The phases of these ten reflections were used in a tangent refinement (Karle & Hauptman, 1958) to establish the phases of the reflections with the 50 largest E's. The resultant E map clearly showed all the non-hydrogen atoms. After several cycles of full-matrix least-squares refinement [$\sum w(\Delta F)^2$ = minimum] with isotropic temperature factors on all atoms R was 0·13. One cycle of large-block least-squares refinement with anisotropic temperature factors reduced R to 0·09. The H atoms were located on a Fourier difference synthesis calculated at this stage. Refinement continued with isotropic temperature factors for the H atoms and converged with R = 0·042 (wR = 0·047). The weights were calculated (Sheldrick, 1976) from $w = (\sigma^2|F_o| + 0{\cdot}0154F_o^2)^{-1}$, where σ is the standard deviation for each reflection and was derived from the diffractometer counting statistics.

The scattering factors used were taken from *International Tables for X-ray Crystallography* (1974) and

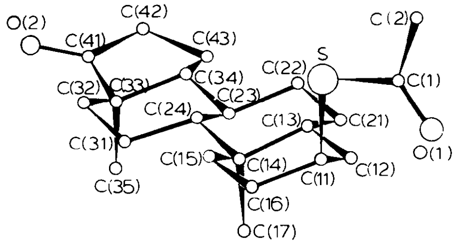

Fig. 1. The unique molecule.

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