Monnikendam, Adam & Bohomos, 1962), is a member of the mitomycin family. The structure possesses the quite unique skeleton of an oxazinone ring fused at the 9 and 9a positions (Morton, Van Lear & Fulmer, 1970). The compound was synthesized recently (Kono, Kasai, Shirahata & Hirayama, 1990). The absolute configuration was determined by referring to that of 7-*p*-bromoanilino-7-demethoxymitomycin B (Hirayama & Shirahata, 1987).

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Acta Cryst. (1991). C**47**, 682–684

Structure of Cholest-5-en-3-one

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(*Received* 21 *March* 1990; *accepted* 9 *August* 1990)

Abstract. $C_{27}H_{44}O$, $M_r = 384{\cdot}7$, orthorhombic, $P2_12_12_1$, $a = 11{\cdot}109\ (1)$, $b = 11{\cdot}213\ (2)$, $c = 19{\cdot}104\ (2)$ Å, $V = 2379{\cdot}4\ (5)$ Å^3, $Z = 4$, $D_x = 1{\cdot}07$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 0{\cdot}44$ mm^{-1}, $F(000) = 856$, $R = 0{\cdot}069$, $wR = 0{\cdot}068$ for 2600 unique observed reflections with $F_o > 4\sigma(F_o)$. The *A* and *C* rings have chair conformations and the *B* and *D* rings assume half-chair conformations. The cholesterol side chain is fully extended with a *gauche, trans* conformation of the terminal C26 and C27 methyl groups.

Experimental. Irregularly shaped crystal with dimensions 0·7 × 0·7 × 0·5 mm. Nonius CAD-4 diffractometer, space group determined from Weissenberg photographs, cell dimensions from 25 centered reflections ($25 < 2\theta < 32°$), Cu $K\alpha$ radiation, Ni filtered, scan width $(1{\cdot}2 + 0{\cdot}14\tan\theta)°$, $\theta_{max} = 75°$, $0 < h < 13$, $0 < k < 13$, $0 < l < 23$, 2704 unique reflections measured using θ–2θ scan mode. Three standard reflections ($\bar{1}48$, $0,\bar{4},11$, $6\bar{3}5$) were measured every 1 h and varied in intensity by less than 3% during the data collection.

Positions of all non-H atoms were found using *SHELXS*86 (Sheldrick, 1986) and refined anisotropically by full-matrix least squares on F_o values, using 2600 reflections for which $F_o > 4\sigma(F_o)$ by means of *SHELX*76 (Sheldrick, 1976). All H atoms were placed geometrically and refined as 'riding groups' (Sheldrick, 1976). Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final $R = 0{\cdot}069$, $wR = 0{\cdot}068$, $S = 2{\cdot}988$, $w = 1/\sigma^2$, $(\Delta/\sigma)_{max} = 0{\cdot}1$. Final difference map showed maximum negative and positive peaks of $-0{\cdot}25$ and $0{\cdot}30$ e Å^{-3}.

Atomic fractional coordinates and equivalent thermal parameters for all non-H atoms are given in Table 1. Bond lengths and valency angles are listed in Table 2.* A stereoplot (Johnson, 1970) of the molecule with the atomic numbering and the packing diagram (Motherwell, 1976) are given in Figs. 1 and 2, respectively.

Related literature. Observed bond lengths and bond angles are in good agreement (within three e.s.d.'s) with corresponding values of 'the average' 5-ene

* Lists of structure factors, anisotropic displacement parameters, H-atom parameters and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53478 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors*

$U_{eq} = (1/3)\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}(Å^2)
C1	0·8618 (4)	0·9169 (5)	−0·1100 (2)	0·059 (1)
C2	0·8657 (5)	0·9153 (6)	−0·1902 (2)	0·072 (2)
C3	0·9742 (5)	0·9834 (6)	−0·2141 (3)	0·075 (2)
C4	1·0909 (4)	0·9378 (6)	−0·1837 (3)	0·071 (2)
C5	1·0863 (4)	0·9307 (4)	−0·1054 (2)	0·054 (1)
C6	1·1764 (4)	0·9745 (4)	−0·0669 (2)	0·055 (1)
C7	1·1825 (4)	0·9712 (4)	0·0104 (2)	0·053 (1)
C8	1·0906 (3)	0·8848 (4)	0·0437 (2)	0·043 (1)
C9	0·9685 (4)	0·8962 (4)	0·0053 (2)	0·046 (1)
C10	0·9769 (4)	0·8693 (4)	−0·0741 (2)	0·048 (1)
C11	0·8690 (4)	0·8252 (5)	0·0430 (2)	0·059 (1)
C12	0·8609 (4)	0·8481 (4)	0·1224 (2)	0·055 (1)
C13	0·9829 (4)	0·8282 (3)	0·1580 (2)	0·042 (1)
C14	1·0739 (3)	0·9082 (3)	0·1212 (2)	0·041 (1)
C15	1·1851 (4)	0·9058 (4)	0·1681 (2)	0·054 (1)
C16	1·1306 (4)	0·8988 (5)	0·2428 (2)	0·056 (1)
C17	0·9947 (4)	0·8737 (4)	0·2340 (2)	0·047 (1)
C18	1·0208 (4)	0·6945 (3)	0·1537 (2)	0·056 (1)
C19	0·9950 (5)	0·7309 (4)	−0·0873 (2)	0·062 (1)
C20	0·9461 (4)	0·7989 (4)	0·2958 (2)	0·053 (1)
C21	0·8133 (5)	0·7611 (5)	0·2860 (3)	0·068 (1)
C22	0·9603 (5)	0·8710 (4)	0·3643 (2)	0·064 (1)
C23	0·9331 (6)	0·8061 (5)	0·4305 (2)	0·077 (2)
C24	0·9632 (8)	0·8790 (8)	0·4948 (3)	0·127 (3)
C25	0·9358 (9)	0·8133 (9)	0·5639 (3)	0·140 (3)
C26	0·8032 (9)	0·8157 (10)	0·5806 (5)	0·152 (3)
C27	1·0212 (12)	0·8359 (13)	0·6130 (5)	0·236 (6)
O3	0·9672 (4)	1·0707 (5)	−0·2534 (2)	0·116 (2)

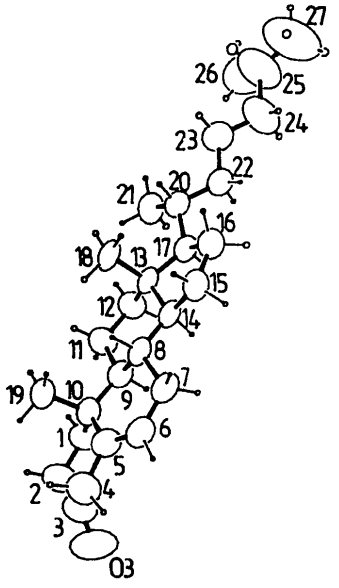

Fig. 1. *ORTEP* stereoview of the molecule with atom numbering with ellipsoids at 65% level of probability (Johnson, 1970).

Table 2. *Bond lengths* (Å), *bond angles* (°) *and the side-chain torsion angles* (°) *with e.s.d.'s in parentheses*

C1—C2	1·533 (5)	C12—C13	1·533 (6)
C1—C10	1·546 (6)	C13—C14	1·523 (5)
C2—C3	1·498 (8)	C13—C17	1·545 (5)
C3—C4	1·510 (8)	C13—C18	1·559 (5)
C3—O3	1·236 (8)	C14—C15	1·526 (6)
C4—C5	1·499 (7)	C15—C16	1·552 (6)
C5—C6	1·336 (6)	C16—C17	1·545 (6)
C5—C10	1·519 (6)	C17—C20	1·546 (6)
C6—C7	1·479 (5)	C20—C21	1·546 (7)
C7—C8	1·545 (6)	C20—C22	1·546 (6)
C8—C9	1·547 (6)	C22—C23	1·490 (6)
C8—C14	1·515 (5)	C23—C24	1·513 (8)
C9—C10	1·549 (5)	C24—C25	1·542 (10)
C9—C11	1·541 (6)	C25—C26	1·507 (14)
C10—C19	1·585 (6)	C25—C27	1·358 (14)
C11—C12	1·541 (5)		

C2—C1—C10	114·6 (4)	C11—C12—C13	111·2 (3)
C1—C2—C3	108·7 (4)	C12—C13—C14	107·2 (3)
C2—C3—C4	113·6 (5)	C12—C13—C17	116·4 (3)
C2—C3—O3	122·6 (5)	C12—C13—C18	110·8 (3)
C4—C3—O3	123·8 (5)	C14—C13—C17	100·5 (3)
C3—C4—C5	111·9 (5)	C14—C13—C18	111·3 (3)
C4—C5—C6	120·3 (4)	C17—C13—C18	110·1 (3)
C4—C5—C10	116·4 (4)	C8—C14—C13	115·5 (3)
C6—C5—C10	123·3 (4)	C8—C14—C15	118·1 (3)
C5—C6—C7	125·1 (4)	C13—C14—C15	104·8 (3)
C6—C7—C8	113·4 (3)	C14—C15—C16	103·0 (3)
C7—C8—C9	109·4 (3)	C15—C16—C17	106·9 (4)
C7—C8—C14	112·0 (3)	C13—C17—C16	104·2 (3)
C9—C8—C14	110·0 (3)	C13—C17—C20	120·6 (3)
C8—C9—C10	113·3 (3)	C16—C17—C20	110·9 (4)
C8—C9—C11	111·4 (3)	C17—C20—C21	112·9 (4)
C10—C9—C11	113·6 (4)	C17—C20—C22	109·1 (3)
C1—C10—C5	109·3 (4)	C21—C20—C22	110·1 (4)
C1—C10—C9	108·5 (3)	C20—C22—C23	116·3 (4)
C1—C10—C19	111·9 (4)	C22—C23—C24	112·4 (5)
C5—C10—C9	110·2 (3)	C23—C24—C25	113·2 (6)
C5—C10—C19	106·2 (3)	C24—C25—C26	111·4 (7)
C9—C10—C19	110·7 (3)	C24—C25—C27	111·4 (8)
C9—C11—C12	114·6 (4)	C26—C25—C27	122·2 (9)

C13—C17—C20—C22	−175·1 (4)	C22—C23—C24—C25	−179·9 (6)
C17—C20—C22—C23	−172·1 (4)	C23—C24—C25—C26	−78·0 (9)
C20—C22—C23—C24	173·4 (5)	C23—C24—C25—C27	141·8 (8)

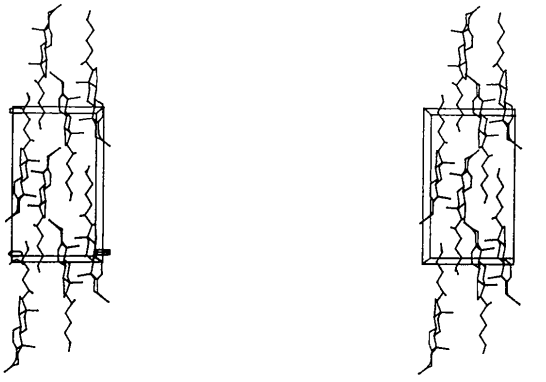

Fig. 2. Stereoview of the molecular packing in the unit cell viewed down the *a* axis.

steroid molecule (Griffin, Duax & Weeks, 1984). The *A* and *C* rings have chair conformations. The *B* and *D* rings have half-chair conformations with asymmetry parameters $\Delta C_2^{5,6} = 2{\cdot}3$, $\Delta C_s^6 = 18{\cdot}8$ and $\Delta C_s^{13} = 13{\cdot}8$, $\Delta C_2^{16} = 5{\cdot}3$ (Duax & Norton, 1975). Search of the Cambridge Structural Database (Allen, Kennard & Taylor, 1983) revealed three hits of 5-en-3-one steroids (ANDEDP10: Carrell, Glusker, Covey, Batzold & Robinson, 1978; HMAMDR: Cox, Mkandawire & Mallison, 1981; MANDIA10:

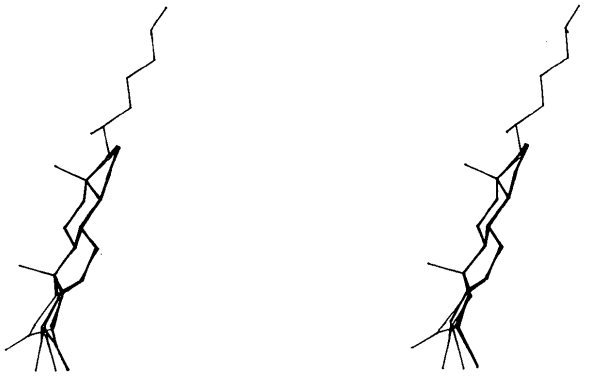

Fig. 3. The superposition of the present molecule (darker line) and the ANDEDP10, HMANDR and MANDIA10 (lighter line) molecules. Atoms C5 through C17 were used to superimpose the molecules.

Ferguson, Marsh, Midgley & Whalley, 1978). The superposition of these three molecules and the title molecule is shown in Fig. 3. Atoms C5 through C17 were used in the program *FITMOL* (Rohrer & Smith, 1980) to superimpose the four molecules. The *B*, *C* and *D* rings of the molecules fit one another very closely but the *A* rings adopt different conformations, ranging from chair to boat.

The side chain at C17 belongs to the most populated conformer *A* (Duax, Griffin, Rohrer & Weeks, 1980).

The authors express their appreciation to Dr Andrzej Szyczewski from the Adam Mickiewicz University for providing crystals. This work was supported by project RP.II.10 from the Polish Ministry of National Education.

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Acta Cryst. (1991). C**47**, 684–686

Structure of an Ascochlorin Derivative (AS-6)

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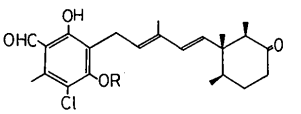
(*Received* 9 *July* 1990; *accepted* 29 *August* 1990)

Abstract. 2-Chloro-4-formyl-5-hydroxy-3-methyl-6-{3-methyl-5-[(1*R*,2*S*,6*R*)-1,2,6-trimethyl-3-oxocyclohexyl]-(2*E*,4*E*)-2,4-pentadien-1-yl}phenoxyacetic acid, AS-6 (1), $C_{25}H_{31}ClO_6$, $M_r = 462{\cdot}98$, triclinic, $P\bar{1}$, $a = 11{\cdot}8423\ (7)$, $b = 12{\cdot}9348\ (10)$, $c = 8{\cdot}3598\ (9)$ Å, $\alpha = 103{\cdot}49\ (1)$, $\beta = 101{\cdot}11\ (1)$, $\gamma = 87{\cdot}02\ (1)°$, $V = 1221{\cdot}8$ Å^3, $Z = 2$, $D_x = 1{\cdot}258$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 16{\cdot}878$ cm^{-1}, $F(000) = 492$, $T = 298$ K, final $R = 0{\cdot}059$ for 3978 unique reflections [$F_o^2 > 2\sigma(F_o^2)$]. The asymmetric unit contains two AS-6 molecules, of which conformations are pseudo-mirror symmetric to each other. The molecules are held together by hydrogen bonds between the carboxy groups to form a dimer.

Experimental. Colorless plates of title compound were grown from benzene/cyclohexane (56:44 *v*/*v*) solution. Crystal size 0·50 × 0·45 × 0·13 mm, Enraf–Nonius CAD-4 κ-cradle diffractometer, Cu $K\alpha$



(1) R= CH_2COOH
(2) R= H

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