

Table 2. Bond lengths (Å) and angles (°)

P(1)—S(1)	2.130 (1)	P(1)—S(2)	2.135 (1)
P(1)—S(3)	1.984 (1)	P(1)—S(4)	1.948 (1)
S(1)—S(2')	2.060 (1)	N—C(1)	1.504 (2)
N—C(3)	1.510 (2)	N—C(5)	1.505 (2)
C(1)—C(2)	1.511 (3)	C(3)—C(4)	1.505 (3)
C(5)—C(6)	1.513 (2)		
S(1)—P(1)—S(2)	103.3 (1)	S(1)—P(1)—S(3)	101.7 (1)
S(2)—P(1)—S(3)	99.6 (1)	S(1)—P(1)—S(4)	112.3 (1)
S(2)—P(1)—S(4)	113.8 (1)	S(3)—P(1)—S(4)	123.6 (1)
P(1)—S(1)—S(2')	102.6 (1)	P(1)—S(2)—S(1')	104.5 (1)
C(1)—N—C(3)	113.1 (1)	C(1)—N—C(5)	114.4 (1)
C(3)—N—C(5)	110.2 (1)	N—C(1)—C(2)	113.3 (1)
N—C(3)—C(4)	112.9 (1)	N—C(5)—C(6)	113.1 (1)

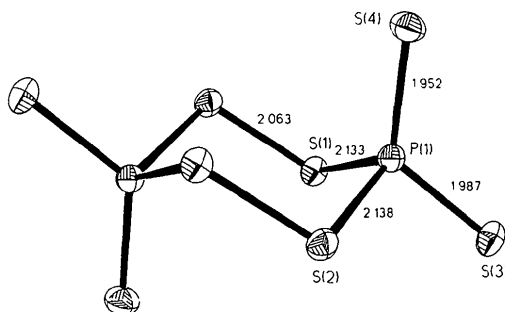
Symmetry operation: (i) $1-x, 1-y, -z$.

Fig. 1. Thermal ellipsoid plot (50% level) of the anion of the title compound, showing the numbering scheme of the independent atoms and the libration-corrected bond lengths.

tion correction. Cell constants were refined from setting angles of 50 reflections in the range 2θ 20–23°.

The structure was solved by routine direct methods and refined anisotropically on F to R 0.026, wR 0.034. H atoms were included using a riding model. The weighting scheme was $w^{-1} = \sigma^2(F) + 0.00015F^2$. 109 parameters; S 1.6; max. Δ/σ 0.001;

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Structure of Cholest-5-en-4-one

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Abstract. $C_{27}H_{44}O$, $M_r = 384.7$, orthorhombic, $P2_12_12_1$, $a = 10.326$ (1), $b = 10.460$ (1), $c = 22.331$ (1) Å, $V = 2412.0$ (3) Å³, $Z = 4$, $D_x =$

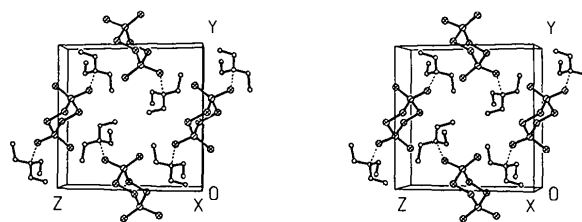


Fig. 2. Stereographic packing diagram of the title compound (H atoms omitted), showing the H bonds as dashed lines.

max., min. $\Delta\rho + 0.39, -0.25 e \text{ \AA}^{-3}$. Final atomic coordinates are presented in Table 1,* with derived bond lengths (uncorrected) and angles in Table 2. A rigid-body libration correction (Schomaker & Trueblood, 1968) was applied ($R_{lib} 0.067$); corrected bond lengths are shown in Fig. 1. A packing diagram is shown in Fig. 2.

Related literature. The pyridinium salt of the same anion was studied by Minshall & Sheldrick (1978) at room temperature. The corrected bond lengths of the anions are almost identical in both studies.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53499 (17 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. Positional ($\times 10^4$) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^3$) for non-H atoms with e.s.d.'s in parentheses

$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
C1	4578 (4)	5714 (3)	3995 (2)	66 (1)
C2	4317 (4)	6782 (3)	3543 (2)	78 (1)
C3	5436 (4)	6878 (3)	3089 (2)	79 (1)
C4	5720 (4)	5617 (4)	2800 (2)	71 (1)
C5	5674 (3)	4455 (3)	3185 (1)	52 (1)
C6	6523 (3)	3540 (3)	3070 (1)	59 (1)
C7	6654 (3)	2326 (3)	3423 (1)	59 (1)
C8	5533 (3)	2111 (3)	3860 (1)	48 (1)
C9	5158 (3)	3383 (3)	4160 (1)	49 (1)
C10	4695 (3)	4375 (3)	3694 (1)	49 (1)
C11	4188 (4)	3171 (3)	4677 (1)	63 (1)
C12	4600 (4)	2117 (3)	5121 (1)	62 (1)
C13	4853 (3)	857 (3)	4801 (1)	49 (1)
C14	5908 (3)	1135 (3)	4336 (1)	49 (1)
C15	6332 (3)	-187 (3)	4117 (2)	63 (1)
C16	6271 (3)	-1022 (3)	4693 (1)	64 (1)
C17	5540 (3)	-217 (3)	5170 (1)	52 (1)
C18	3603 (3)	330 (3)	4516 (2)	64 (1)
C19	3378 (3)	4007 (3)	3414 (1)	62 (1)
C20	4734 (3)	-1065 (3)	5592 (2)	61 (1)
C21	3962 (4)	-288 (4)	6055 (2)	94 (1)
C22	5596 (3)	-2048 (3)	5912 (2)	71 (1)
C23	4865 (4)	-3115 (4)	6220 (2)	77 (1)
C24	5716 (5)	-4132 (4)	6487 (2)	110 (2)
C25	5002 (5)	-5276 (4)	6746 (3)	108 (2)
C26	4240 (5)	-4919 (5)	7301 (2)	116 (2)
C27	5778 (6)	-6389 (5)	6803 (3)	162 (3)
O4	6046 (3)	5549 (3)	2278 (1)	106 (1)

a distorted chair conformation and the *B* ring has an intermediate sofa-half-chair conformation. The *C* and *D* rings assume chair and envelope conformations, respectively. The cholesterol side chain is fully extended with a *-gauche,trans* conformation of the terminal C26 and C27 methyl groups.

Experimental. An irregularly shaped crystal with dimensions $0.8 \times 0.7 \times 0.6$ mm. Nonius CAD-4 diffractometer, space group determined from Weissenberg photographs, cell dimensions from 25 centered reflections ($25 < 2\theta < 35^\circ$), Cu $K\alpha$ radiation, Ni filtered, no monochromator, scan width $(0.85 + 0.14 \tan \theta)^\circ$, $\theta_{max} = 75^\circ$, $0 < h < 12$, $0 < k < 13$, $0 < l < 28$, 2737 unique reflections measured using θ - 2θ scan mode. Three standard reflections (10 $\bar{6}$, 2 $\bar{1}5$, 30 $\bar{6}$) were measured every hour and varied in intensity by less than 2% during data collection.

Positions of all non-H atoms were found using *SHELXS86* (Sheldrick, 1985) and refined anisotropically by full-matrix least squares on F_o values, using 2588 reflections for which $F_o > 4\sigma(F_o)$ with *SHELX76* (Sheldrick, 1976). All H atoms were placed geometrically and refined as 'riding groups' (Sheldrick, 1976) except H atoms from the terminal C26 and C27 methyl groups which were not refined. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final $R = 0.061$, $wR = 0.060$, $S = 2.871$, $w =$

Table 2. Bond lengths (\AA), bond angles ($^\circ$) and side-chain torsion angles ($^\circ$) with e.s.d.'s in parentheses

C1—C2	1.529 (5)	C12—C13	1.522 (4)
C1—C10	1.558 (5)	C13—C14	1.533 (4)
C2—C3	1.540 (6)	C13—C17	1.563 (4)
C3—C4	1.497 (5)	C13—C18	1.541 (5)
C4—C5	1.490 (5)	C14—C15	1.531 (5)
C4—O4	1.215 (5)	C15—C16	1.556 (5)
C5—C6	1.323 (4)	C16—C17	1.554 (4)
C5—C10	1.523 (4)	C17—C20	1.539 (5)
C6—C7	1.501 (4)	C20—C21	1.538 (6)
C7—C8	1.531 (4)	C20—C22	1.536 (5)
C8—C9	1.539 (4)	C22—C23	1.513 (5)
C8—C14	1.524 (4)	C23—C24	1.503 (6)
C9—C10	1.545 (4)	C24—C25	1.520 (7)
C9—C11	1.544 (4)	C25—C27	1.515 (8)
C10—C19	1.546 (4)	C25—C27	1.419 (7)
C11—C12	1.543 (4)		
C2—C1—C10	112.7 (3)	C11—C12—C13	111.4 (3)
C1—C2—C3	110.5 (3)	C12—C13—C14	106.0 (2)
C2—C3—C4	111.9 (3)	C12—C13—C17	116.9 (2)
C3—C4—C5	117.6 (3)	C12—C13—C18	111.1 (3)
C3—C4—O4	121.3 (4)	C14—C13—C17	99.8 (2)
C5—C4—O4	121.0 (4)	C14—C13—C18	112.5 (2)
C4—C5—C6	117.2 (3)	C17—C13—C18	109.9 (2)
C4—C5—C10	119.8 (3)	C8—C14—C13	114.8 (2)
C6—C5—C10	123.0 (3)	C8—C14—C15	117.1 (3)
C5—C6—C7	124.7 (3)	C13—C14—C15	104.4 (2)
C6—C7—C8	113.0 (2)	C14—C15—C16	103.4 (3)
C7—C8—C9	109.9 (2)	C15—C16—C17	106.4 (2)
C7—C8—C14	110.6 (2)	C13—C17—C16	104.4 (2)
C9—C8—C14	109.8 (2)	C13—C17—C20	119.4 (3)
C8—C9—C10	111.4 (2)	C16—C17—C20	111.7 (2)
C8—C9—C11	111.4 (2)	C17—C20—C21	112.8 (3)
C10—C9—C11	113.5 (2)	C17—C20—C22	110.9 (3)
C1—C10—C5	108.9 (3)	C21—C20—C22	110.0 (3)
C1—C10—C9	109.7 (3)	C20—C22—C23	114.6 (3)
C1—C10—C19	109.3 (2)	C22—C23—C24	114.3 (4)
C5—C10—C9	109.5 (2)	C23—C24—C25	115.1 (4)
C5—C10—C19	107.2 (2)	C24—C25—C26	111.7 (4)
C9—C10—C19	112.2 (2)	C24—C25—C27	114.0 (5)
C9—C11—C12	113.8 (3)	C26—C25—C27	115.0 (5)
C13—C17—C20—C22	-179.7 (3)	C22—C23—C24—C25	-174.4 (4)
C17—C20—C22—C23	-166.3 (3)	C23—C24—C25—C26	-68.4 (5)
C20—C22—C23—C24	175.1 (3)	C23—C24—C25—C27	159.3 (5)

$1/\sigma^2$, $(\Delta/\sigma)_{max} = 0.01$. Final difference map showed maximum negative and positive peaks of -0.08 and 0.09 e \AA^{-3} .

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

Related literature. The *A* ring has a distorted chair conformation with asymmetry parameters (Duax & Norton, 1975) $\Delta C_s^3 = 18.7$, $\Delta C_2^{3,4} = 20.6$. The *B* ring

* 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 53465 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

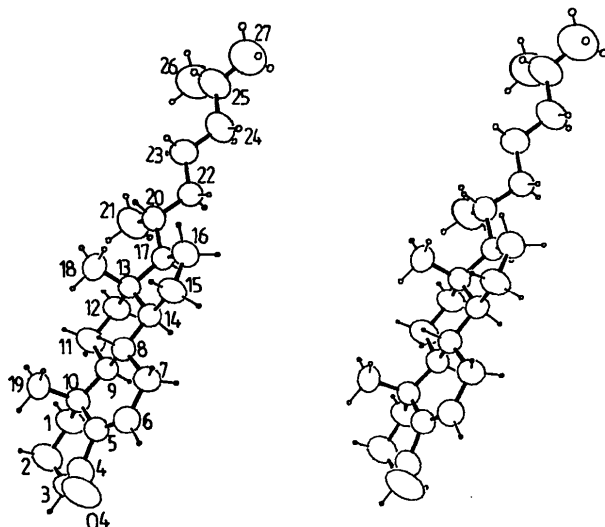


Fig. 1. ORTEP stereoview of the molecule with the atom-numbering scheme; ellipsoids are at the 65% probability level (Johnson, 1970).

has an intermediate sofa-half-chair conformation with asymmetry parameters $\Delta C_2^{5,6} = 9.1$ and $\Delta C_s^6 = 14.8$. The C ring has a chair conformation and the D ring has an envelope conformation with asymmetry parameters $\Delta C_s^{13} = 6.1$ and $\Delta C_2^{16} = 16.0$.

The side chain at C17 is fully extended (see Table 2) with a *-gauche,trans* conformation (Duax, Griffin, Rohrer & Weeks, 1980).

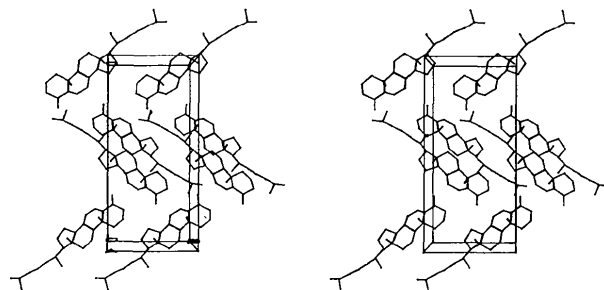


Fig. 2. Stereoview of the molecular packing in the unit cell, viewed down the *a* axis.

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Structure of 6 β ,6' β -Bi(7 α -allyl-3-oxo-4-estren-17 β -yl acetate)

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Abstract. C₄₆H₆₂O₆, *M_r* = 711.0, orthorhombic, *P*2₁2₁2₁, *a* = 20.187 (3), *b* = 22.004 (3), *c* = 9.180 (1) Å, *V* = 4078 (2) Å³, *Z* = 4, *D_x* = 1.16 g cm⁻³, λ (Mo *K* α) = 0.71069 Å, μ = 0.7 cm⁻¹, *F*(000) = 1544, *T* = 295 K, *R* = 0.096 for 3894 unique observed reflections with *F_o* > 2 σ (*F_o*). The title compound is a dimer connected by a single bond between C6 and C6' [bond length 1.560 (7) Å]. The two ster-

oid moieties are oriented β -face to β -face, head to head and lie in almost parallel planes (7.6°), rotated by 45° to one another. The two conformations of the identical portions of the dimer differ chiefly in the orientation of the allyl and acetate groups. C23' (acetate) and O3' form the shortest intermolecular contact less than 3.5 Å; C...O = 3.35 (1) Å.