The quinone ring shows normal geometry. The average distances and corresponding angles for the quinone ring are C—C 1·477 (9), C=C 1·333 (9), C=O 1·223 (8) Å; C—C—C 119·5 (6), C=C—C 120·3 (5) and O=C—C 120·2 (5)°, which are in agreement with values determined in other 1,4-benzoquinone compounds (Schmalle, Jarchow, Hausen & Schulz, 1984*a,b,c,d*). The C(9)—C(10) bond distance of 1·509 (8) Å is significantly shorter than the expected value of 1·533 Å for a C—C bond length in *n*-hydrocarbons (Bartell, 1959).

As shown in Fig. 1, the molecular conformation of the side chain at C(3) is the fully extended *trans*-zigzag form and the pertinent torsion angles are given in Table 2.

The crystal structure contains two different types of hydrogen bonds which play an important role in the stabilization. The hydrogen-bonding scheme is shown in Fig. 2. The first type is an intramolecular hydrogen bond between the O(2)—H hydroxyl group and the O(3) carbonyl group. The O(2)···O(3) and H(2)···O(3) distances are 2·669 (6) and 2·34 (7) Å and the O(2)—H(2)···O(3) angle is 107 (5)°. In addition, H(2) participates in a second type of hydrogen bond involving the O(1) carbonyl group of an adjacent molecule (*x*, 1 + *y*, *z*). The O(2)···O(1) and H(2)···O(1) distances are 2·737 (4) and 2·04 (7) Å and the O(2)—H(2)···O(1) angle is 150 (5)°.

An additional intramolecular interaction, C(8)···O(1), 2·826 (7) Å, and the presence of the methyl group C(15) at C(8) appear to define the conformation around the C(3)—C(8) bond which is such that the side chain bonded to C(8) points away from the quinone ring. The dihedral angle between the side chain and the quinone ring is 102·6 (6)°.

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Structure of (1*S*,3*R*,6*R*,7*S*)-2,2,6,10-Tetramethyl-8,11-dioxotricyclo[5.3.1.0³,⁷]undec-9-en-9-yl Acetate (β-Pipitzol Acetate)*

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Abstract. $C_{17}H_{22}O_4$, $M_r = 290·4$, orthorhombic, $P2_12_12_1$, $a = 9·368$ (4), $b = 11·011$ (3), $c = 15·627$ (5) Å, $V = 1612$ (1) Å³, $Z = 4$, $D_x = 1·20$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0·7107$ Å, $\mu = 0·079$ mm⁻¹, $F(000) = 624$, $T = 293$ K. Final $R = 0·060$ for 901 observed reflections. The molecular structure is determined from the X-ray data and confirms the structure previously assigned on the basis of chemical and spectroscopic evidence. Both five-membered rings are in a half-chair conformation. The cyclohexene ring adopts a 1,2-diplanar conformation with C(11) 0·85 (1) Å out

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of the plane of the other five atoms. The absolute configuration is inferred from the known C(6) stereochemistry of (+)-cedrol. The packing in the crystal is entirely due to van der Waals forces.

Introduction. β-Pipitzol (1) is a sesquiterpene which was isolated from the roots of *Perezia cuernavacana*, a member of the Compositae family distributed in the neighbourhood of Cuernavaca (Estado de Morelos, Mexico). Chemical and spectroscopic studies led to the proposal of structure (1) for β-pipitzol (Walls, Padilla, Joseph-Nathan, Giral & Romo, 1965; Walls, Padilla, Joseph-Nathan, Giral, Escobar & Romo, 1966). It was of interest to determine the crystal structure of (2) in order to ascertain its conformation and molecular geometry.

(1) R = H

(2) R = COCH₃
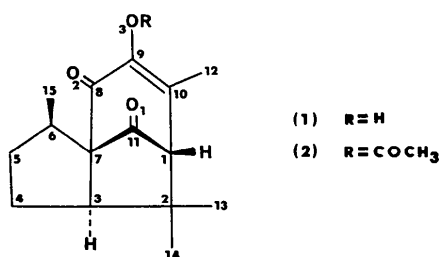
Experimental. Colourless crystal $0.20 \times 0.28 \times 0.28$ mm. Nicolet $R3$ four-circle diffractometer. Lattice parameters from 25 machine-centred reflections with $7.7 < 2\theta < 15.5°$. 1254 reflections with $3 < 2\theta < 45°$, 901 independent with $I > 2.5\sigma(I)$, index range h 0–10, k 0–11, l 0–16, $R_{int} = 0.016$, ω-scan mode, variable scan speed, scan width $1.0°(\theta)$, two standard reflections (200, 002) monitored every 50 measurements, no intensity variation, Lp correction, absorption ignored; structure solved by combination of direct methods and partial structure expansion by an iterative E–Fourier procedure using *SHELXTL* (Sheldrick, 1981); least-squares anisotropic refinement of all non-H atoms; H atoms riding on bonded C with fixed isotropic temperature factor, $U = 0.06$ Å²; function minimized $\sum w(\Delta F)^2$, $w = [\sigma^2(F_o) + 0.001(F_o)^2]^{-1}$, where σ is standard deviation of observed amplitudes, based on counting statistics; isotropic extinction parameter $X = 0.00249$. In the last cycle $(\Delta/\sigma)_{max} = 0.11$; $\Delta\rho$ from -0.20 to $+0.19$ e Å⁻³; final $R = 0.060$, $wR = 0.062$; scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. Atomic coordinates are in Table 1.* A perspective molecular drawing and selected torsion

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and least-squares-planes' calculations have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42581 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

angles are shown in Fig. 1. The bond lengths and angles for non-H atoms are listed in Table 2. The carbon skeleton of the molecule comprises a system of two five-membered rings (A and B) and a six-membered ring (C). The A and B rings are *cis*-fused at C(3)—C(7) and the C ring is fused at C(1)—C(11)—C(7) to the B ring.

The absolute configuration of (2) is inferred from the known C(6) stereochemistry of (+)-cedrol deduced from chemical studies (Stork & Clarke, 1961) and from X-ray studies on cedryl chromate (Amirthalingam, Grant & Senol, 1972). The stereochemistry of (2) is as follows: C(1)—βH *cis* to C(11)—βO(1) and C(3)—αH *trans* to C(6)—βCH₃.

Table 1. *Atomic coordinates* $(\times 10^4)$ *and equivalent isotropic temperature factors* $(\text{Å}^2 \times 10^3)$

$$U_{eq} = (U_{11} \times U_{22} \times U_{33})^{1/3}.$$

|	x	y	z	U_{eq}
O(1)	119 (5)	−272 (4)	−128 (3)	63 (2)
O(2)	2099 (6)	1649 (4)	−2201 (3)	76 (2)
O(3)	2849 (5)	3329 (4)	−1030 (3)	52 (2)
O(4)	5003 (5)	2894 (4)	−1544 (3)	76 (2)
C(1)	2471 (7)	457 (5)	266 (4)	48 (2)
C(2)	3670 (7)	−472 (6)	41 (4)	50 (2)
C(3)	3527 (7)	−570 (6)	−958 (4)	50 (3)
C(4)	3490 (9)	−1861 (6)	−1369 (5)	73 (3)
C(5)	1948 (10)	−2085 (6)	−1625 (4)	75 (3)
C(6)	1410 (8)	−853 (6)	−1892 (4)	60 (3)
C(7)	2020 (7)	−11 (5)	−1206 (4)	44 (2)
C(8)	2221 (7)	1293 (5)	−1478 (4)	50 (2)
C(9)	2683 (6)	2123 (5)	−784 (4)	41 (2)
C(10)	2804 (6)	1771 (5)	36 (4)	44 (2)
C(11)	1290 (8)	21 (6)	−322 (4)	48 (2)
C(12)	3247 (8)	2618 (6)	727 (4)	67 (3)
C(13)	3298 (9)	−1659 (7)	507 (4)	74 (3)
C(14)	5157 (8)	−43 (7)	276 (5)	79 (3)
C(15)	−204 (8)	−729 (7)	−2057 (5)	84 (3)
C(16)	4059 (7)	3607 (6)	−1488 (4)	50 (2)
C(17)	3977 (9)	4825 (6)	−1869 (5)	73 (3)
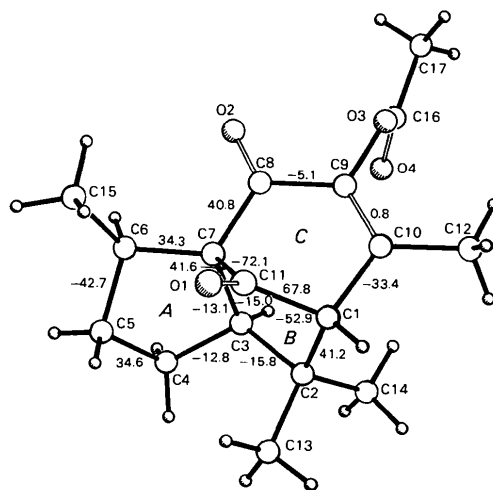
Fig. 1. The molecular structure of the title compound (2) showing the atom labelling and selected torsion angles (°); e.s.d.'s are 0.6–0.9°.

The conformation of both five-membered rings may be described by the parameters Δ and φ_m (Altona, Geise & Romers, 1968). The A and B rings have Δ and φ_m values of $0 \cdot 0$, $-42 \cdot 7$ $(7)°$ and $0 \cdot 3$ (6), $-52 \cdot 9$ $(6)°$, respectively, indicating in both cases a half-chair conformation.

Table 2. *Bond lengths (Å) and angles (°) with e.s.d.'s in parentheses*

O(1)-C(11)	1·183 (9)	O(2)-C(8)	1·201 (7)
O(3)-C(9)	1·391 (7)	O(3)-C(16)	1·375 (8)
O(4)-C(16)	1·186 (8)	C(1)-C(2)	1·560 (9)
C(1)-C(10)	1·524 (8)	C(1)-C(11)	1·516 (9)
C(2)-C(3)	1·571 (9)	C(2)-C(13)	1·536 (9)
C(2)-C(14)	1·516 (10)	C(3)-C(4)	1·561 (9)
C(3)-C(7)	1·588 (9)	C(4)-C(5)	1·520 (12)
C(5)-C(6)	1·505 (10)	C(6)-C(7)	1·528 (9)
C(6)-C(15)	1·540 (11)	C(7)-C(8)	1·509 (8)
C(7)-C(11)	1·542 (9)	C(8)-C(9)	1·483 (9)
C(9)-C(10)	1·343 (9)	C(10)-C(12)	1·486 (9)
C(16)-C(17)	1·470 (9)		

C(9)-O(3)-C(16)	116·6 (5)	C(2)-C(1)-C(10)	115·0 (5)
C(2)-C(1)-C(11)	100·5 (5)	C(10)-C(1)-C(11)	107·9 (5)
C(1)-C(2)-C(3)	101·9 (5)	C(1)-C(2)-C(13)	106·7 (5)
C(3)-C(2)-C(13)	113·1 (5)	C(1)-C(2)-C(14)	113·8 (5)
C(3)-C(2)-C(14)	109·9 (6)	C(13)-C(2)-C(14)	111·0 (6)
C(2)-C(3)-C(4)	118·3 (5)	C(2)-C(3)-C(7)	107·0 (5)
C(4)-C(3)-C(7)	103·5 (5)	C(3)-C(4)-C(5)	106·1 (6)
C(4)-C(5)-C(6)	104·2 (6)	C(5)-C(6)-C(7)	103·2 (5)
C(5)-C(6)-C(15)	117·0 (6)	C(7)-C(6)-C(15)	115·5 (6)
C(3)-C(7)-C(6)	105·6 (5)	C(3)-C(7)-C(8)	109·0 (5)
C(6)-C(7)-C(8)	115·3 (5)	C(3)-C(7)-C(11)	100·6 (5)
C(6)-C(7)-C(11)	118·5 (5)	C(8)-C(7)-C(11)	106·6 (5)
O(2)-C(8)-C(7)	124·3 (6)	O(2)-C(8)-C(9)	121·0 (5)
C(7)-C(8)-C(9)	114·6 (5)	O(3)-C(9)-C(8)	114·8 (5)
O(3)-C(9)-C(10)	121·9 (5)	C(8)-C(9)-C(10)	123·0 (5)
C(1)-C(10)-C(9)	118·8 (5)	C(1)-C(10)-C(12)	118·8 (5)
C(9)-C(10)-C(12)	122·4 (6)	O(1)-C(11)-C(1)	127·4 (6)
O(1)-C(11)-C(7)	129·4 (6)	C(1)-C(11)-C(7)	103·1 (6)
O(3)-C(16)-O(4)	120·4 (6)	O(3)-C(16)-C(17)	111·8 (6)
O(4)-C(16)-C(17)	127·8 (7)		

The C ring adopts a 1,2-diplanar conformation with C(11) $0 \cdot 85$ (1) Å out of the best plane formed by the other five ring atoms. The acetoxy group at C(9) is almost perpendicular to the C ring; the pertinent torsion angles are C(16)-O(3)-C(9)-C(8) $= -76 \cdot 4$ (7) and C(16)-O(3)-C(9)-C(10) $= 109 \cdot 6$ $(7)°$. The conformation of ring C is such that ring B is perpendicular [the angle between normals to the best planes through rings C and B is $92 \cdot 1$ $(7)°$].

In all essential details, the molecular geometry of (2) in terms of the bond lengths and angles shows normal values. However, the C(2)-C(3)-C(4) bond angle of $118 \cdot 3$ $(5)°$ has been increased to minimize the steric interactions between the O(1) carbonyl group and the methyl group C(13). The interactions of the O(1) and O(2) carbonyl groups with the methyl group at C(6) have been relieved by an increase in the bond angles C(6)-C(7)-C(11) and C(6)-C(7)-C(8) [$118 \cdot 5$ (5) and $115 \cdot 3$ $(5)°$ respectively]. The C=O bonds agree well with the accepted value of $1 \cdot 215$ (5) Å (Sutton, 1965).

The arrangement of the molecules in the unit cell is shown in Fig. 2. There are four intermolecular approaches $<3 \cdot 4$ Å involving non-H atoms: C(16)$\cdots$O(1), C(17)$\cdots$O(1), C(14)$\cdots$O(3)($0 \cdot 5 + x$, $0 \cdot 5 - y$, $-z$) [$3 \cdot 28$ (1), $3 \cdot 34$ (1), $3 \cdot 36$ (1) Å, respectively]; and C(12)$\cdots$O(4)($-0 \cdot 5 + x$, $0 \cdot 5 - y$, $-z$) [$3 \cdot 34$ (1) Å]. The molecules are held in the crystal by van der Waals forces.
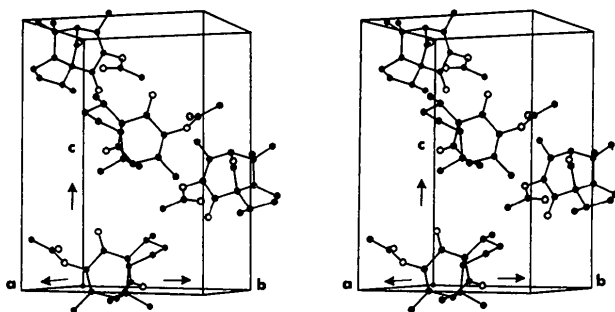
Fig. 2. Stereoscopic view of the molecular conformation and packing.

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