among the conformations reported in all colchicinoid crystal structures. The bond lengths and crystal conformation are shown in the *ORTEP* (Johnson, 1965) drawing, Fig. 1, and it will be observed that the amide group adopts the so-far universal conformation in colchicinoids. The bond angles are given in Table 2 (deposited) and no unusual values are apparent.

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Structure and Stereochemistry of Cacalol Methyl Ether, a Sesquiterpene*

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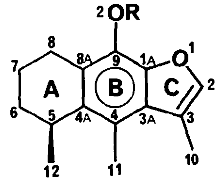
Abstract. 9-Methoxy-3,4,5-trimethyl-5,6,7,8-tetrahydronaphtho[2,3-*b*]furan, $C_{16}H_{20}O_2$, $M_r = 244·3$, monoclinic, $P2_1$, $a = 8·753$ (6), $b = 5·338$ (4), $c = 14·949$ (7) Å, $\beta = 105·93$ (4)°, $V = 672$ (1) Å^3, $Z = 2$, $D_x = 1·21$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0·7107$ Å, $\mu = 0·073$ mm^{-1}, $F(000) = 264$, $T = 293$ K, final $R = 0·068$ for 905 reflections. (5*S*)-Stereochemistry is determined for the title compound and confirms the structure previously assigned on the basis of chemical and spectroscopic evidence. The *A* ring adopts a 1,2-diplanar conformation. The molecules in the crystal are packed at normal van der Waals distances.

Introduction. Cacalol (1) is a sesquiterpene which was isolated from the roots of *Cacalia decomposita* A. Gray, a shrub indigenous to northern Mexico (Romo & Joseph-Nathan, 1964). Following several revisions, structure (1) was proposed essentially simultaneously in three publications (Kakisawa, Inouye & Romo, 1969; Brown & Thomson, 1969; Ruiz, Correa & Maldonado, 1969) and this was confirmed by synthesis (Yuste & Walls, 1976; Huffman & Pandian, 1979).

It was of interest to determine the crystal structure of (2) in order to ascertain its conformation and molecular geometry.



(1) $R = H$

(2) $R = CH_3$

Experimental. Colourless crystal 0·13 × 0·34 × 0·50 mm. Nicolet *R*3 four-circle diffractometer. Lattice parameters from 25 machine-centred reflections with $6·2 < 2\theta < 33·6°$. 1308 reflections with $3 < 2\theta < 50°$, 905 independent with $I > 3·0\sigma(I)$, index range h −10→10, k 0→6, l 0→17, ω-scan mode, variable scan speed, scan width 1·0° (θ), two standard reflections (011, $\bar{1}\bar{1}0$) 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

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using *SHELXTL* (Sheldrick, 1981); least-squares refinement of all non-H atoms anisotropic; H atoms of CH, CH_2 and CH_3 groups riding on bonded C with fixed isotropic temperature factor, $U = 0{\cdot}06$ Å^2, $\sum w(\Delta F)^2$ minimized, $w = \{\sigma^2(F_o) + 0{\cdot}002(F_o)^2\}^{-1}$, where σ is the standard deviation of observed amplitudes based on counting statistics; isotropic extinction parameters $X = 0{\cdot}0014$. In the last cycle $(\Delta/\sigma)_{\max} = 0{\cdot}048$; $\Delta\rho$ from $-0{\cdot}19$ to $0{\cdot}18$ e Å^{-3}; final $R = 0{\cdot}068$, $wR = 0{\cdot}079$; scattering factors from *International Tables for X-ray Crystallography* (1974). All computations performed on a Nova 4S computer and plots drawn on a Tektronix plotter with *SHELXTL* system of programs.

Discussion. Fig. 1 shows the molecular conformation of (2). Final atomic coordinates are in Table 1.* The bond lengths and the valence angles for non-H atoms are listed in Table 2. The molecular structure of (2) determined from X-ray data confirms the structure and establishes the configuration at C(5) as (5*S*). The *A* ring adopts a 1,2-diplanar conformation.

The *B* ring to which the methoxy and methyl groups are attached shows a slight but significant deviation from planarity (r.m.s. deviation 0·013 Å), which is probably due to the effect of packing [maximum deviation −0·022 (7) Å for C(1a)]. This ring shows normal geometry: 1·398 (8) Å and 120·0 (5)° for the mean aromatic bond distance and the mean internal angle, respectively. This mean bond distance is close to that given by Sutton (1965) [1·395 (5) Å]. It has been pointed out that the intraannular aromatic angle (β) *ortho* to a five-membered fused ring is contracted by 2·2 (2)° (Allen, 1981). The internal angles at C(4) and C(9) [116·3 (5) and 117·5 (5) Å, respectively] are in good agreement with the above observation. The angles between the mean planes of the methoxy group at C(9) and the *B* and *C* rings are 37·4 (8) and 35·1 (8)°, respectively. The *C* ring is planar within the limits of experimental error, with a maximum deviation of 0·007 (7) Å at C(3a). The angle between the best planes through the *B* and *C* rings is 2·4 (8)°.

Examination of the three-dimensional packing of the molecules within the crystalline lattice (shown in Fig. 2) reveals that, excluding H atoms, there is only one intermolecular approach <3·41 Å, C(2)···O(1)(2−*x*,

Table 1. *Atomic coordinates* (×10^4) *and equivalent isotropic temperature factors* (Å^2 ×10^3)

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

	x	*y*	*z*	U_{eq}
O(1)	9644 (5)	7927	1111 (3)	67 (2)
C(2)	8913 (7)	9533 (15)	403 (4)	66 (3)
C(3)	7708 (7)	10768 (14)	557 (4)	57 (2)
C(3a)	7598 (6)	9993 (12)	1455 (4)	47 (2)
C(4)	6614 (6)	10618 (12)	2042 (4)	46 (2)
C(4a)	6940 (6)	9462 (12)	2901 (4)	43 (2)
C(5)	5858 (6)	10040 (13)	3544 (4)	54 (2)
C(6)	6708 (7)	9484 (15)	4563 (4)	66 (3)
C(7)	7413 (7)	6875 (15)	4690 (4)	63 (2)
C(8)	8614 (7)	6460 (14)	4157 (4)	58 (2)
C(8a)	8188 (6)	7724 (14)	3200 (3)	46 (2)
C(9)	9173 (6)	7128 (13)	2629 (4)	49 (2)
C(1a)	8795 (6)	8228 (13)	1766 (4)	49 (2)
C(10)	6785 (7)	12737 (15)	−96 (4)	67 (2)
C(11)	5368 (6)	12641 (15)	1733 (4)	64 (2)
C(12)	4327 (6)	8592 (16)	3237 (4)	70 (3)
O(2)	10444 (5)	5635 (13)	2997 (3)	90 (2)
C(13)	11007 (9)	3968 (17)	2472 (6)	87 (3)

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

O(1)–C(2)	1·376 (7)	O(1)–C(1a)	1·392 (8)
C(2)–C(3)	1·316 (10)	C(3)–C(3a)	1·433 (8)
C(3)–C(10)	1·509 (9)	C(3a)–C(4)	1·427 (9)
C(3a)–C(1a)	1·391 (8)	C(4)–C(4a)	1·382 (8)
C(4)–C(11)	1·514 (9)	C(4a)–C(5)	1·554 (9)
C(4a)–C(8a)	1·409 (8)	C(5)–C(6)	1·530 (8)
C(5)–C(12)	1·505 (8)	C(6)–C(7)	1·514 (11)
C(7)–C(8)	1·500 (10)	C(8)–C(8a)	1·532 (8)
C(8a)–C(9)	1·406 (8)	C(9)–C(1a)	1·371 (8)
C(9)–O(2)	1·357 (8)	O(2)–C(13)	1·365 (11)

C(2)–O(1)–C(1a)	104·1 (4)	O(1)–C(2)–C(3)	113·8 (6)
C(2)–C(3)–C(3a)	106·4 (6)	C(2)–C(3)–C(10)	123·3 (6)
C(3a)–C(3)–C(10)	130·2 (6)	C(3)–C(3a)–C(4)	135·7 (5)
C(3)–C(3a)–C(1a)	105·7 (5)	C(4)–C(3a)–C(1a)	118·6 (5)
C(3a)–C(4)–C(4a)	117·5 (5)	C(3a)–C(4)–C(11)	119·0 (5)
C(4a)–C(4)–C(11)	123·3 (5)	C(4)–C(4a)–C(5)	118·7 (5)
C(4)–C(4a)–C(8a)	122·2 (5)	C(5)–C(4a)–C(8a)	119·0 (5)
C(4a)–C(5)–C(6)	111·4 (5)	C(4a)–C(5)–C(12)	110·7 (5)
C(6)–C(5)–C(12)	110·6 (6)	C(5)–C(6)–C(7)	111·9 (6)
C(6)–C(7)–C(8)	112·9 (6)	C(7)–C(8)–C(8a)	113·9 (5)
C(4a)–C(8a)–C(8)	123·7 (5)	C(4a)–C(8a)–C(9)	120·5 (5)
C(8)–C(8a)–C(9)	115·7 (5)	C(8a)–C(9)–C(1a)	116·3 (5)
C(8a)–C(9)–O(2)	117·2 (5)	C(1a)–C(9)–O(2)	126·3 (6)
O(1)–C(1a)–C(3a)	109·9 (5)	O(1)–C(1a)–C(9)	125·0 (5)
C(3a)–C(1a)–C(9)	124·8 (6)	C(9)–O(2)–C(13)	122·3 (5)

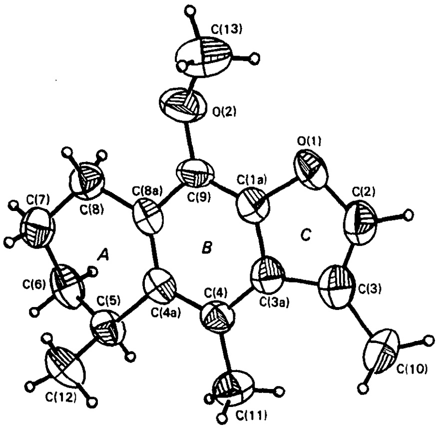

Fig. 1. The molecular conformation of (1), showing the atom numbering. The thermal ellipsoids are drawn at the 50% probability level.

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* List of structure amplitudes, anisotropic thermal parameters, H-atom coordinates and the results of mean-plane calculations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44017 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

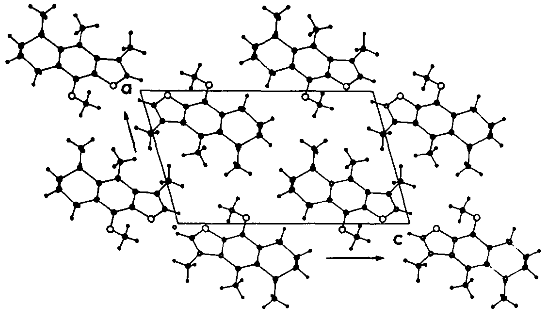

Fig. 2. Packing arrangement as viewed along **b**.

$0{\cdot}5+y, -z)$ 3·40 (1) Å. The molecules are held in the crystal by van der Waals forces.

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Structure du Dibenzylglycolate de Pipéridino-2 Ethyle (PDG) Chlorure d'Hydrogène

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Abstract. $C_{23}H_{30}NO_3^+.Cl^-$, $M_r = 402{\cdot}9$, monoclinic, $P2_1/c$, $a = 15{\cdot}696\,(10)$, $b = 12{\cdot}635\,(6)$, $c = 11{\cdot}168\,(10)$ Å, $\beta = 101{\cdot}7\,(1)^\circ$, $V = 2168{\cdot}7\,(5)$ Å^3, $Z = 4$, $D_m = 1{\cdot}30$, $D_x = 1{\cdot}22$ g cm^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}5418$ Å, $\mu = 16{\cdot}38$ cm^{-1}, $F(000) = 864$, $T = 293$ K, final $R = 0{\cdot}043$ for 1895 observed reflections. On the basis of crystal data and conformational comparisons with the molecular geometry of morphine, it is suggested that the analgesic drug PDG could have the structural requirements necessary to act on specific opiate receptors. The ethyl C—C bond is short (1·31 Å), probably due to disorder. The Cl atom forms hydrogen bonds with H(O3) and H(N).

Introduction. L'étude par des tests classiques de l'activité pharmacologique d'un nouveau dérivé (PDG) de l'acide dibenzylglycolique a révélé pour ce composé l'existence de propriétés analgésiques. Une étude plus fine en électrophysiologie a permis de montrer que le PDG se liait à des sites opiacés communs à ceux de la morphine mais également à d'autres sites plus spécifiques (Hénichart, Carette, Bernier & Houssin, 1985). La structure de PDG, établie par radiocristallographie, est l'objet du présent mémoire.

Partie expérimentale. Le composé synthétisé a été cristallisé dans l'acétone. Cristaux de dimensions 0,12 × 0,20 × 0,60 mm. Densité mesurée par la méthode de flottaison. Les paramètres de la maille déterminés et affinés à partir de 25 réflexions. Diffractomètre à quatre cercles Philips PW1100. Balayage θ–2θ. Largeur de balayage fixée à 1,40°. Cu $K\bar{\alpha}$ filtrée