

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

Examination of the three-dimensional packing of the molecules within the crystalline lattice (shown in Fig. 2) reveals that, excluding H atoms, there are five intermolecular approaches $< 3.4 \text{ \AA}$: C(3a)···O(1), C(3a)···O(3), $(1+x, 1.5-y, 0.5+z)$ 3.13 (1) and 3.26 (1) \AA . No suitable H donors for other H-bond formation are available. The molecules are held in the crystal by van der Waals forces.

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Structure of 5-(3,4-Methylenedioxyphenyl)-1-piperidinopent-2,4-dien-1-one

BY M. NETHAJI AND VASANTHA PATTABHI*

Department of Crystallography and Biophysics,† University of Madras, Guindy Campus, Madras-600 025, India

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Abstract. C₁₇H₁₉NO₃, *M_r* = 285.35, monoclinic, *P*2₁/*n*, *a* = 8.740 (1), *b* = 13.647 (1), *c* = 13.163 (1) \AA , β = 108.77 (1)°, *V* = 1486.6 (3) \AA^3 , *Z* = 4, *D_x* = 1.275 g cm⁻³, Cu *K*α, λ = 1.5418 \AA , μ = 6.7 cm⁻¹, *F*(000) = 608, *T* = 295 K, *R*(*F*) = 0.041, *wR* = 0.044 for 996 unique reflections with $I \geq 2.5\sigma(I)$. The piperidine ring is in a chair conformation, the 3,4-methylenedioxyphenyl group is nearly planar, and the dihedral angle between these is 45.3 (1)°. The crystal structure is stabilized by van der Waals forces only.

Introduction. The title compound, which is a natural product, is extracted from the plant *Vicou Indica* Dc. The present study was undertaken to confirm the structure proposed by chemists (Purushothaman, 1988).

Experimental. Pale-green needles 0.17 × 0.13 × 0.1 mm from methanol, three-dimensional intensity data on Enraf-Nonius CAD-4 automatic diffractometer, $\omega/2\theta$ scan mode with $2 \leq 2\theta \leq 120^\circ$, Cu *K*α radiation, data corrected for direct-beam polarization and Lorentz effects, and for absorption (average transmission factor 0.926, with maximum and minimum 0.861 and 0.999, respectively), $0 \leq h \leq 9$, $0 \leq k \leq 14$, $-13 \leq l \leq 13$, 1753 measured reflections, 1471 unique and 996 observed with $I \geq 2.5\sigma(I)$, cell constants from 16 reflections with $28 \leq 2\theta \leq 59^\circ$, three standard reflections monitored for every 100 reflections showed no significant variation in intensity, scan width using the relation $(A + B \tan \theta)$ where *A* and *B* are 0.8 and 0.14, respectively, aperture width using $(4.0 + 2 \tan \theta)$, maximum time spent on any reflection measurement was 30 s, and the background count was half the scan time. Structure solution by direct methods, H atoms from $\Delta\rho$ synthesis, full-matrix least-squares

* To whom correspondence should be addressed.

† DCB contribution No. 725.

Table 1. Positional parameters, thermal parameters, and their estimated standard deviations

Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameters defined as:

$$B_{\text{eq}} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq} (Å ²)
O1	0.6706 (3)	0.3655 (2)	0.6496 (2)	5.84 (7)
C2	0.6808 (5)	0.4631 (3)	0.6133 (3)	5.7 (1)
O3	0.8167 (3)	0.5099 (2)	0.6909 (2)	6.18 (7)
C4	0.8827 (4)	0.4393 (3)	0.7676 (2)	4.15 (9)
C5	0.7971 (4)	0.3542 (3)	0.7434 (3)	4.3 (1)
C6	0.8378 (4)	0.2734 (3)	0.8060 (3)	4.8 (1)
C7	0.9739 (4)	0.2820 (2)	0.8977 (3)	4.7 (1)
C8	1.0625 (4)	0.3677 (2)	0.9249 (2)	3.96 (9)
C9	1.0145 (4)	0.4502 (2)	0.8568 (3)	4.3 (1)
C10	1.2030 (4)	0.3710 (2)	1.0221 (3)	4.3 (1)
C11	1.3050 (4)	0.4453 (3)	1.0537 (3)	4.5 (1)
C12	1.4385 (4)	0.4501 (3)	1.1533 (3)	4.5 (1)
C13	1.5363 (4)	0.5261 (3)	1.1847 (3)	4.7 (1)
C14	1.6679 (4)	0.5266 (2)	1.2885 (3)	4.5 (1)
O15	1.6908 (3)	0.4548 (2)	1.3481 (2)	6.27 (7)
N16	1.7612 (3)	0.6069 (2)	1.3159 (2)	4.82 (8)
C17	1.8870 (4)	0.6114 (3)	1.4208 (3)	6.3 (1)
C18	1.8398 (5)	0.6813 (3)	1.4928 (3)	6.7 (1)
C19	1.8040 (5)	0.7816 (3)	1.4433 (3)	7.5 (1)
C20	1.6809 (5)	0.7765 (3)	1.3305 (3)	6.4 (1)
C21	1.7322 (5)	0.7027 (3)	1.2625 (3)	5.6 (1)

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

Numbers in parentheses are e.s.d.'s in the least-significant digits.

O1	C2	1.428 (5)	C11	C12	1.449 (4)		
O1	C5	1.376 (3)	C12	C13	1.322 (5)		
C2	O3	1.442 (4)	C13	C14	1.476 (4)		
O3	C4	1.381 (4)	C14	O15	1.231 (4)		
C4	C5	1.363 (5)	C14	N16	1.345 (4)		
C4	C9	1.363 (4)	N16	C17	1.464 (4)		
C5	C6	1.355 (5)	N16	C21	1.467 (5)		
C6	C7	1.400 (4)	C17	C18	1.493 (6)		
C7	C8	1.384 (5)	C18	C19	1.505 (6)		
C8	C9	1.416 (4)	C19	C20	1.530 (5)		
C8	C10	1.462 (4)	C20	C21	1.508 (6)		
C10	C11	1.326 (5)					
C2	O1	C5	106.6 (2)	C8	C10	C11	126.1 (3)
O1	C2	O3	107.6 (2)	C10	C11	C12	125.6 (3)
C2	O3	C4	105.3 (3)	C11	C12	C13	124.7 (3)
O3	C4	C5	110.8 (2)	C12	C13	C14	121.7 (3)
O3	C4	C9	126.3 (3)	C13	C14	O15	120.3 (3)
C5	C4	C9	122.9 (3)	C13	C14	N16	118.5 (3)
O1	C5	C4	109.7 (3)	O15	C14	N16	121.2 (3)
O1	C5	C6	128.0 (3)	C14	N16	C17	119.6 (3)
C4	C5	C6	122.3 (3)	C14	N16	C21	127.0 (3)
C5	C6	C7	116.1 (3)	C17	N16	C21	112.1 (3)
C6	C7	C8	122.9 (3)	N16	C17	C18	110.4 (3)
C7	C8	C9	118.8 (3)	C17	C18	C19	111.6 (4)
C7	C8	C10	119.6 (3)	C18	C19	C20	111.2 (3)
C9	C8	C10	121.6 (3)	C19	C20	C21	110.9 (3)
C4	C9	C8	117.0 (3)	N16	C21	C20	110.5 (3)

refinement on F_o , non-hydrogen atoms anisotropic and H atoms isotropic to a final $R(F) = 0.041$ and $wR = 0.044$ with individual weighting scheme based on counting statistics where $w = 4|F_o|^2/[\sigma^2(|F_o|^2)]$, $\sigma^2(F_o) = [\sigma^2(I) + p^2I^2]^{1/2}/Lp$ and p is the ignorance factor ($p = 0.02$), $(\Delta/\sigma)_{\text{max}} = 0.26$, $(\Delta/\sigma)_{\text{min}} = 0.09$, $S = 1.867$ for 266 parameters, data/parameter ratio 3.74; final map had no peaks $> 0.12 \text{ e \AA}^{-3}$. All

calculations on VAX 11/730 computing system using SDP Package (Frenz, 1978).

Discussion. Atomic positions with equivalent isotropic temperature factors for the non-H atoms are given in Table 1.* Fig. 1 shows a stereoview of the molecule and Fig. 2 the packing in the unit cell. The bond lengths and angles involving non-H atoms are given in Table 2. The average e.s.d.'s in bond lengths and angles are 0.004 Å and 0.03°. The bond lengths and angles around the

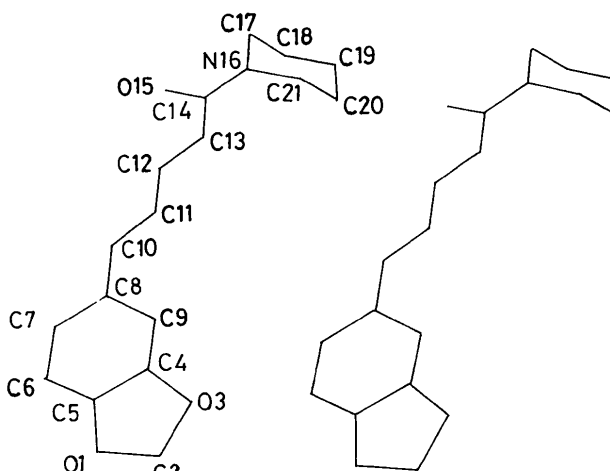
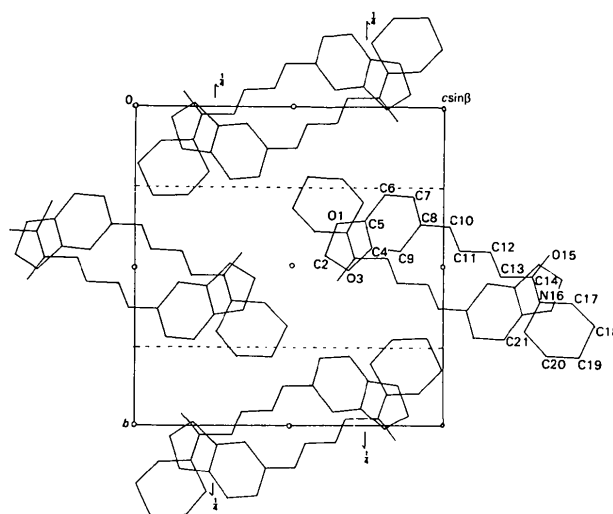


Fig. 1. A stereoview of the molecule.

Fig. 2. The packing of the molecules in the unit cell projected down the *a* axis.

piperidine ring are comparable with those of dyclonine hydrochloride (Sinha, Vasantha Pattabhi, Nethaji & Gabe, 1987). The bond lengths and angles around the 3,4-methylenedioxyphenyl group are in agreement with the values reported in 7-methoxy-3-methyl-2-(3,4-methylenedioxyphenyl)-5-(prop-1-enyl)-2,3-dihydrobenzo[*b*]furan (Ponnuswamy & Parthasarathy, 1981). The double-bond oxygen O(15) is in a *syn* configuration with respect to the olefinic linkage [C(12)=C(13)]. The piperidine ring takes a chair conformation while the 3,4-methylenedioxyphenyl group is planar within experimental error; atom C(2) deviates by 0.02 Å from the latter. The dihedral angle between the two planes is 45.3 (1)°. In the unit cell the molecules are stabilized solely by van der Waals forces.

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Structure of L-Alprenolol D-Tartrate Monohydrate

BY MAREK L. GŁÓWKA* AND PENELOPE W. CODDING†

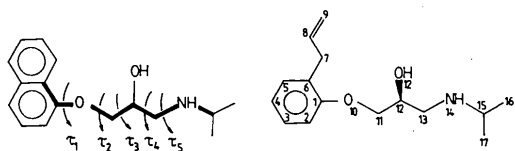
Departments of Chemistry and of Pharmacology and Therapeutics, The University of Calgary, Calgary, Alberta, Canada T2N 1N4

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Abstract. L-1-(2-Allylphenoxy)-3-(isopropylamino)propan-2-ol D-tartrate monohydrate, C₁₅H₂₄NO₂·C₄H₅O₆·H₂O, *M_r* = 417.46, monoclinic, *P*2₁, *Z* = 2, *a* = 7.594 (7), *b* = 8.645 (13), *c* = 16.575 (21) Å, β = 93.37 (2)°, *V* = 1086 (2) Å³, *Z* = 2, *F*(000) = 448, *T* = 198 (5) K, *D_x* = 1.276 g cm⁻³, λ(Cu Kα) = 1.54178 Å, Ni filter, μ = 0.863 cm⁻¹, *R* = 0.054, *wR* = 0.070, for the 2363 reflections included in the refinement; sample from Sigma Chemical Company (A9389). The propoxinolamine side chain is observed in a conformation that is atypical of β-blocking agents. This conformation may result from the three hydrogen bonds that are formed between the side chain and the tartrate anion and water molecule or may be a result of the extensive hydrogen-bonding pattern between tartrate anions.

Introduction. β-Adrenergic blocking agents are useful in the management of cardiovascular disorders, including hypertension, angina pectoris and cardiac arrhythmias (Weiner, 1985); and, selective drugs function by antagonizing the actions of epinephrine or norepinephrine. Propranolol, the first clinically useful β-

adrenergic antagonist, is prototypical of the agents developed to date. Structural similarities, both within each chemical group of β-receptor blocking agents and among the groups, are very close. Proposals for the structural determinants of β-blocking activity (Ammon, Balsamo, Macchia, Macchia, Howe & Keefe, 1975; Macchia *et al.*, 1987) consider the presence of an OH-CH-CH₂-NH-alkyl moiety joined to a planar system to be essential.



Propranolol

Alprenolol (active S isomer shown)

Alprenolol belongs to the arylpropoxinolamine class of β-blockers; Leger, Gadret & Carpy (1977) postulate that the affinity of these ligands for the β-adrenoceptors is related to the relative positions of the aryl moiety, the hydroxyl group [O(12)], and the amine group [N(14)]. Extensive X-ray studies on arylpropoxinolamine β-blockers reveal a uniformity of the conformation of the propoxinolamine chain in the

* Present address: Institute of General Chemistry, Technical University of Łódź, Zwirki 36, 90-924 Łódź, Poland.

† Author to whom correspondence should be addressed.