

Discussion. The atomic numbering and the bond lengths are shown in Fig. 1. Bond angles are given in Table 2. The structure consists of independent molecules which are almost planar. Except for the C(11)—N(3) bond all other bond lengths have values expected for such structures. The C(11)—N(3) bond has partial double-bond character suggesting some form of delocalization in the structure. This effect has been observed in some azo pigments (Goliński, Reck & Kutschabsky, 1982; Whitaker, 1977*a,b*, 1983*a,b*, 1984). Further, the interatomic distances N(1)—O(1) = 2.557 (7) Å and N(3)—O(2) = 2.692 (8) Å suggest that both these contacts involve hydrogen bonds. These values compare well with 2.591 and 2.666 Å, the hydrogen-bond lengths found by Whitaker (1983*b*). This hydrogen bonding inhibits any rotation and will tend to cause the molecule to be planar. This is confirmed by the values of the torsion angles, the maximum deviation from 0 or 180° being 7.5 (9)°, indicating only a very minor deviation from planarity. The intermolecular interactions are all van der Waals. This structure confirms the results of the physico-chemical studies.

An ORTEP (Johnson, 1965) diagram of the molecule is shown in Fig. 2.

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Structure of Leuconolam, an Alkaloid Containing an Unusual Nine-Membered Ring

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Abstract. C₁₉H₂₂N₂O₃·C₂H₆O, $M_r = 372.51$, $P2_12_12_1$, $a = 8.073$ (5), $b = 11.377$ (5), $c = 20.461$ (9) Å, $V = 1879$ (2) Å³, $Z = 4$, $D_x = 1.32$ g cm⁻³, $\lambda(\text{Mo } K\alpha_{1,2}) = 0.70926$, 0.713554 Å, $\mu = 0.98$ cm⁻¹, $F(000) = 800$, $T = 297$ K, $R = 3.7\%$, 2046 unique reflections. The oxygen atoms are involved in hydrogen bonding, with the ethanolic oxygen atom acting both as a donor and as an acceptor intermolecularly. In the conformation adopted by the molecule the two conjugated groups make angles of 54.5 (2) and 60.4 (3)° with the plane of the benzene ring.

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Introduction. X-ray analysis was undertaken to determine the structure and stereochemistry of leuconolam, an uncommon alkaloid isolated in fairly good quantities from *Leuconotis griffithii* (Apocynaceae). Spectral (NMR, MS, IR and UV) data obtained were too ambiguous for structural assignment (Goh, Chen Wei & A. Razak Mohd. Ali, 1984). The MS and NMR spectral data indicated considerable unsaturation in the molecule but this was not reflected in the UV spectrum, which appears to exclude 'expected' indole or acylated *o*-substituted aniline derivatives. The data suggest a new or unusual structural formula for this alkaloid and, since spectral comparisons with structurally related

compounds are excluded, the structure and stereochemistry were determined by X-ray crystallography.

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters (\AA^2)

	x	y	z	B_{eq}^*
O(2)	1251 (4)	872 (3)	7020 (1)	3.8
O(5)	6233 (4)	-1047 (3)	7742 (2)	3.9
O(21)	3822 (4)	-746 (3)	9637 (1)	3.1
O(e)†	7644 (5)	4839 (3)	4163 (2)	4.5
N(1)	1777 (5)	2152 (3)	7831 (2)	2.6
N(4)	4391 (5)	-1321 (3)	8592 (2)	2.4
C(2)	1107 (5)	1151 (4)	7603 (2)	2.5
C(3)	4505 (6)	-2599 (4)	8654 (3)	3.7
C(5)	5207 (5)	-690 (4)	8138 (2)	2.6
C(6)	4630 (6)	529 (4)	8228 (2)	2.6
C(7)	3522 (5)	606 (4)	8713 (2)	2.0
C(8)	2739 (5)	1704 (3)	8947 (2)	2.1
C(9)	2848 (6)	2038 (4)	9597 (2)	2.8
C(10)	2220 (7)	3120 (4)	9799 (2)	3.3
C(11)	1463 (6)	3871 (4)	9363 (2)	3.3
C(12)	1341 (6)	3535 (4)	8710 (2)	3.1
C(13)	1953 (5)	2454 (4)	8506 (2)	2.4
C(14)	2785 (7)	-3109 (4)	8538 (3)	3.7
C(15)	1523 (6)	-2483 (4)	8970 (2)	3.6
C(16)	97 (5)	399 (4)	8058 (2)	2.5
C(17)	783 (5)	-843 (4)	8192 (2)	2.4
C(18)	-1559 (6)	-1012 (5)	9337 (2)	4.1
C(19)	257 (6)	-652 (4)	9425 (2)	2.7
C(20)	1443 (5)	-1127 (4)	8892 (2)	2.2
C(21)	3236 (5)	-636 (4)	8985 (2)	2.5
C(1e)†	8889 (7)	5578 (5)	3917 (3)	4.8
C(2e)†	8279 (9)	6602 (5)	3535 (3)	5.9

$$* B_{\text{eq}} = (B_{11} + B_{22} + B_{33})/3.$$

† O(e), C(1e) and C(2e) refer to the ethanol molecule.

Table 2. Interatomic distances (\AA) and bond angles ($^\circ$)

N(1)	C(2)	1.344 (6)	C(15)	C(20)	1.553 (7)
N(1)	C(13)	1.431 (6)	C(16)	C(17)	1.541 (6)
C(2)	O(2)	1.239 (5)	C(17)	C(20)	1.561 (6)
C(2)	C(16)	1.505 (6)	C(18)	C(19)	1.533 (7)
C(3)	N(4)	1.462 (6)	C(19)	C(20)	1.549 (6)
C(3)	C(14)	1.523 (7)	C(20)	C(21)	1.563 (6)
N(4)	C(5)	1.346 (6)	C(21)	O(21)	1.422 (5)
N(4)	C(21)	1.457 (6)	O(e)	C(1e)	1.404 (7)
C(5)	O(5)	1.228 (6)	C(1e)	C(2e)	1.486 (8)
C(5)	C(6)	1.474 (6)	N(1)	H(1)	0.90 (4)
C(6)	C(7)	1.339 (6)	<C—C>		1.389 (6)*
C(7)	C(8)	1.479 (6)	<C—H>		0.98 (5)
C(7)	C(21)	1.536 (6)	<O—H>		0.84 (5)
C(14)	C(15)	1.525 (7)			

C(2)	N(1)	C(13)	125.4 (4)	C(2)	C(16)	C(17)	115.9 (4)
N(1)	C(2)	O(2)	120.9 (4)	C(16)	C(17)	C(20)	118.4 (4)
N(1)	C(2)	C(16)	119.0 (4)	C(18)	C(19)	C(20)	114.5 (4)
N(4)	C(3)	C(14)	107.9 (4)	C(15)	C(20)	C(19)	107.5 (4)
C(3)	N(4)	C(5)	124.0 (4)	C(15)	C(20)	C(21)	107.7 (4)
C(3)	N(4)	C(21)	121.6 (4)	C(17)	C(20)	C(19)	111.3 (4)
C(5)	N(4)	C(21)	114.1 (4)	C(17)	C(20)	C(21)	110.7 (4)
N(4)	C(5)	O(5)	127.5 (4)	C(19)	C(20)	C(21)	111.2 (4)
N(4)	C(5)	C(6)	105.2 (4)	N(4)	C(21)	C(7)	101.3 (3)
O(5)	C(5)	C(6)	127.3 (4)	N(4)	C(21)	C(20)	109.5 (4)
C(5)	C(6)	C(7)	111.4 (4)	N(4)	C(21)	O(21)	104.9 (3)
C(6)	C(7)	C(8)	125.5 (4)	C(7)	C(21)	C(20)	115.0 (4)
C(6)	C(7)	C(21)	108.0 (4)	C(7)	C(21)	O(21)	111.8 (4)
C(8)	C(7)	C(21)	126.5 (4)	C(20)	C(21)	O(21)	113.0 (4)
C(3)	C(14)	C(15)	110.0 (4)	O(e)	C(1e)	C(2e)	114.9 (5)
C(14)	C(15)	C(20)	115.6 (4)	<C—C—C>			120.1 (4)*

* Average phenyl values.

Experimental. Colorless crystal [0.25 × 0.6 × 0.7 mm, faces (100), ($\bar{1}00$), (010), ($0\bar{1}0$), (001), (00 $\bar{1}$)] of leuconolam (from aqueous ethanol) isolated from alkaloidal fraction of methanol extracts of dried *Leuconotis griffithii* (Apocynaceae). Crystal selected was polyhedron, mounted on Nicolet P3m diffractometer, niobium-filtered Mo $K\alpha$ radiation. 15 independent reflections, chosen from Polaroid photograph, computer centered; angular coordinates autoindexed; θ - 2θ scan technique (Storm *et al.*, 1983; Nicolet, 1980) for $4 < 2\theta < 50^\circ$; 0.8° above and below $K\alpha_1$ and $K\alpha_2$ used; observed systematic absences for $h00$, $0k0$ and $00l$ with $h, k, l = 2n+1$ respectively indicated $P2_12_12_1$; intensities of three standard reflections showed no greater fluctuations during data collection than expected from Poisson statistics; Lorentz-polarization corrections applied; 2046 intensities collected, ranges of h, k, l 0 to 10, 0 to 15 and 0 to 25 respectively; 1214 with $F_o^2 > 3\sigma(F_o^2)$, $\sigma(F_o^2)$ estimated from counting statistics (Corfield, Doedens & Ibers, 1967); nonhydrogen atoms located using *MULTAN74* (Main, Woolfson, Lessinger, Germain & Declercq, 1974; Germain, Main & Woolfson, 1971); full-matrix least-squares refinement on F (Freyberg, Mockler & Sinn, 1976)* for 244

* *REFINE* is used for least-squares refinement, *MAP* for Fourier, Patterson and related functions, *UNICEL* for unit-cell refinement, *FIN* for final Fourier, *F* lists *etc.*, *TABLE* for tabulations of atom parameters, bond lengths and angles.

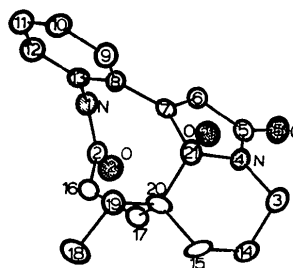


Fig. 1. View of the title compound with atom labels.

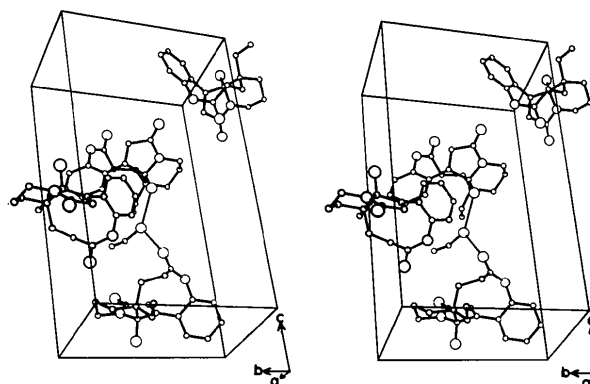


Fig. 2. The packing diagram of the unit cell.

parameters, $\Delta/\sigma < 0.20$ in final cycle; anisotropic temperature factors for nonhydrogen atoms, further Fourier difference functions permitted location of hydrogen atoms, included in refinement; model converged with $R = 3.7$ and $wR = 3.7\%$, $w = 1/\sigma^2(F)$; final difference Fourier map featureless; atomic scattering and anomalous scattering factors for all atoms from Cromer & Waber (1974).*

Discussion. Table 1 lists the atomic coordinates and equivalent isotropic thermal parameters. Table 2 contains bond distances and angles. Fig. 1 shows the single molecule and Fig. 2 is the packing diagram. Hydrogen bonds are formed from O(*e*) to O(2)[$\frac{1}{2}+x$, $\frac{1}{2}-y$, $1-z$], N(1) to O(5)[$1-x$, $\frac{1}{2}+y$, $\frac{3}{2}-z$], O(21) to O(*e*)[$1-x$, $y-\frac{1}{2}$, $\frac{3}{2}-z$] with distances of 2.788 (5), 2.855 (5) and 2.806 (5) Å respectively. O(*e*) thus acts both as a donor and as an acceptor. The structure of leuconolam shows that it is an 'opened-up' indole alkaloid artefact derived from an aspidosperma-type alkaloid. Only one other natural product, rhazinilam, C₁₉H₂₂N₂O (Abraham & Rosenstein, 1972), has previously been found to possess this structural skeleton. As shown in Fig. 1, conjugation of the benzene ring with N(1) as well as the unsaturated five-membered ring is limited by non-planarity [benzene/pyrrolidine and benzene/N(1)CO interplanar

* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42572 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

angles being 125.5 (2) and 60.4 (3)° respectively]. This accounts for the weak UV absorptions and also explains other salient features in the NMR spectra. It may also be noted that the structure can allow for other possible conformational isomers owing to the conformational mobility of the nine- and six-membered rings.

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1,4-Dibromonaphthalene; a Redetermination

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Abstract. C₁₀H₆Br₂, $M_r = 285.99$, monoclinic, $P2_1/c$, $a = 4.063$ (3), $b = 16.476$ (3), $c = 27.274$ (8) Å, $\beta = 91.84$ (3)°, $V = 1824.8$ (18) Å³, $Z = 8$ (two molecules/asymmetric unit), $D_x = 2.081$ g cm⁻³, Mo $K\alpha$, $\lambda\alpha_1 = 0.70930$ Å, $\mu = 87$ cm⁻¹, $F(000) = 1088$, $T = 293$ K, $R = 0.044$ for 1350 observed reflections. Both

molecules are planar, with bond lengths similar to those in naphthalene, but with some bond-angle differences due to the presence of the Br substituents, the endocyclic angles at the substituted 1 and 4 positions having a mean value of 123.8 (6)°.

Introduction. A two-dimensional study of the structure of 1,4-dibromonaphthalene (I) in which only y and z parameters were determined (Trotter, 1961, with a and

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