

can be measured by ΔC_2 (for C5) = 18.2° and ΔC_2 (for C20) = 15.8° (Duax, Weeks & Rohrer, 1976). The acetoxy groups are planar and the carbonyl O atoms are eclipsed to the intracyclic atoms (C15 and C21 respectively).

In the crystal, molecules of raspacionin, related by a screw axis parallel to **b**, are linked by an intermolecular hydrogen bond, which involves the hydroxyl group and the $O38(1-x, \frac{1}{2}+y, \frac{1}{2}-z)$ carbonyl O atom at a distance $2.775(5)$ Å. As shown in Fig. 2, the packing is characterized by channels parallel to **a** and centered on the screw axis, with a sinusoidal shape (Fig. 3) and an approximate cross section of 3.5×6 Å². The identity period *a* and the channel cross section are compatible with the presence of only one bent heptane molecule roughly in a *11g* conformation (Fig. 3). All distances between successively translated molecules are greater than 3.0 Å. This minimal parameter model of the disordered 'guest' structure satisfactorily fitted the residual density. The resulting heptane molecule is roughly planar, with a maximum displacement of 0.32 Å from the mean molecular plane, which is parallel to the *ab* plane. In relation to the 'host' lattice, the heptane molecules are loosely packed and no interactions are below 3.9 Å.

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Structure of Bulbocarpine

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Abstract. $C_{19}H_{19}NO_4$, $M_r = 325.36$, orthorhombic, $P2_12_12_1$, $a = 8.293(5)$, $b = 8.462(5)$, $c = 22.80(1)$ Å, $V = 1600(1)$ Å³, $Z = 4$, $D_x = 1.350$ Mg m⁻³,

$\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $\mu = 0.088$ mm⁻¹, $F(000) = 688$, $T = 293(1)$ K, $R = 0.063$ for 1523 reflections with $I > 3.5\sigma(I)$. The asymmetric C atom C5 has an

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Table 1. Fractional atomic coordinates ($\times 10^4$) and equivalent isotropic temperature factors ($\text{\AA}^2 \times 10^3$)
$$U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33}).$$

	x	y	z	U_{eq}
O1	3512 (4)	-790 (5)	3838 (2)	50 (1)
O2	5339 (5)	-2133 (6)	3259 (2)	66 (1)
O3	-879 (6)	1867 (4)	4765 (2)	53 (1)
O4	719 (5)	749 (4)	3829 (2)	43 (1)
N	-855 (6)	-6512 (5)	3400 (2)	44 (1)
C1	3529 (7)	-4309 (7)	2994 (2)	46 (1)
C2	1996 (7)	-4930 (6)	3085 (2)	38 (1)
C3	1549 (8)	-6470 (7)	2793 (3)	57 (2)
C4	-236 (9)	-6721 (7)	2804 (2)	57 (2)
C5	-759 (6)	-4823 (5)	3570 (2)	32 (1)
C6	-1023 (7)	-4644 (5)	4224 (2)	37 (1)
C7	-970 (6)	-2909 (5)	4398 (2)	32 (1)
C8	-1913 (7)	-2362 (7)	4846 (2)	44 (1)
C9	-1875 (7)	-755 (6)	5002 (2)	47 (1)
C10	-969 (7)	253 (5)	4671 (2)	42 (1)
C11	-70 (6)	-262 (5)	4190 (2)	32 (1)
C12	50 (6)	-1902 (5)	4084 (2)	27 (1)
C13	1255 (6)	-2638 (5)	3685 (2)	30 (1)
C14	2778 (6)	-2102 (6)	3601 (2)	34 (1)
C15	3891 (6)	-2920 (7)	3266 (2)	45 (1)
C16	893 (6)	-4129 (5)	3431 (2)	32 (1)
C17	5154 (8)	-923 (10)	3680 (3)	81 (2)
C18	-1563 (10)	2439 (7)	5291 (2)	68 (2)
C19	-2532 (7)	-7058 (7)	3435 (3)	64 (2)

S configuration. Ring *B* exhibits a half-chair conformation, while the form of the *C* ring is nearest that of a screw-boat. The dihedral angle between the two phenyl rings *A* and *D* is $34.4(2)^\circ$. The molecules are held together by hydrogen bonds forming an infinite chain along the *b* axis.

Introduction. In the course of our studies of natural products we investigated the crystal and molecular structure of bulbocapnine isolated from *Corydalis cava*. The aerial parts of the plant were collected at Fruška Gora (Vojvodina) during the flowering phase. The isolation was performed as described by Gašić, Popović & Dragutinović (1985). Bulbocapnine is used clinically for the control of Parkinson's disease and other neurological disorders and contained in preparation for post- and preanaesthetic treatment (Schauenberg & Paris, 1975). It has also been applied as the preparation Biral® for neurovegetative disorders (Odenthal, Molls & Vogel, 1981).

Experimental. A crystal of dimensions $0.2 \times 0.4 \times 0.1$ mm was mounted on a CAD-4 diffractometer (Berne) using graphite-monochromated Mo *K* α radiation. Cell constants were refined by a least-squares fit for 21 reflections with θ range from 3.5 to 8.7° . Intensity data were collected with ω - 2θ scan in the range $2\theta < 50^\circ$, h 0 to 9, k 0 to 10, l 0 to 26. Standard reflection (213) was measured every 200 min, but no intensity variations were recorded. Of 1636 measured reflections, 1523 with $I > 3.5\sigma(I)$ were used in refinement. Data were corrected for

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

O1	C14	1.377 (6)	C3	C4	1.496 (10)		
O1	C17	1.413 (8)	C5	C6	1.515 (7)		
O2	C15	1.373 (7)	C5	C16	1.524 (7)		
O2	C17	1.412 (9)	C6	C7	1.521 (6)		
O3	C10	1.385 (6)	C7	C8	1.367 (7)		
O3	C18	1.412 (7)	C7	C12	1.398 (7)		
O4	C11	1.356 (6)	C8	C9	1.406 (8)		
N	C4	1.463 (7)	C9	C10	1.364 (7)		
N	C5	1.483 (6)	C10	C11	1.396 (7)		
N	C19	1.468 (8)	C11	C12	1.412 (6)		
C1	C2	1.391 (8)	C12	C13	1.488 (7)		
C1	C15	1.362 (8)	C13	C14	1.356 (7)		
C2	C3	1.510 (8)	C13	C16	1.420 (6)		
C2	C16	1.385 (7)	C14	C15	1.384 (7)		
C15	O2	C17	104.4 (5)	O3	C10	C11	113.6 (4)
C10	O3	C18	116.6 (4)	C9	C10	C11	122.2 (5)
C4	N	C5	109.9 (4)	O4	C11	C10	122.5 (4)
C4	N	C19	110.2 (5)	O4	C11	C12	118.8 (4)
C5	N	C19	109.9 (4)	C10	C11	C12	118.6 (4)
C2	C1	C15	117.4 (5)	C7	C12	C11	118.0 (4)
C1	C2	C3	119.0 (5)	C7	C12	C13	117.7 (4)
C1	C2	C16	120.2 (5)	C11	C12	C13	124.3 (4)
C3	C2	C16	120.8 (5)	C12	C13	C14	124.9 (4)
C2	C3	C4	111.0 (5)	C12	C13	C16	118.6 (4)
N	C4	C3	110.2 (5)	C14	C13	C16	115.9 (4)
N	C5	C6	110.2 (4)	O1	C14	C13	128.8 (5)
N	C5	C16	111.4 (4)	O1	C14	C15	109.0 (4)
C6	C5	C16	107.2 (4)	C13	C14	C15	122.2 (5)
C5	C6	C7	110.4 (4)	O2	C15	C1	127.3 (5)
C6	C7	C8	120.3 (4)	O2	C15	C14	110.3 (5)
C6	C7	C12	118.2 (4)	C1	C15	C14	122.4 (5)
C8	C7	C12	121.5 (4)	C2	C16	C5	121.6 (4)
C7	C8	C9	120.2 (5)	C2	C16	C13	121.8 (4)
C8	C9	C10	118.5 (5)	C5	C16	C13	116.6 (4)
O3	C10	C9	124.1 (5)	O1	C17	O2	109.6 (5)

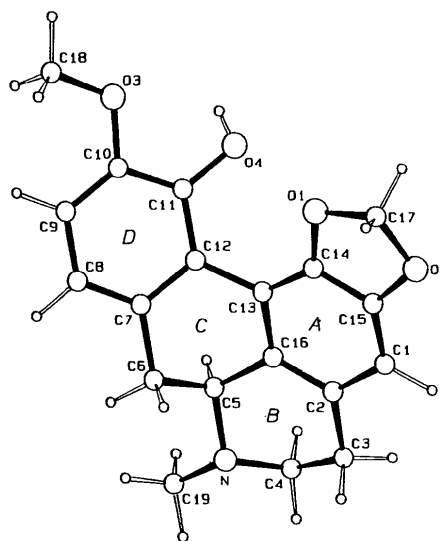


Fig. 1. A view of the molecule showing atomic numbering.

Lorentz and polarization effects but not for absorption. The structure was solved by direct methods (*SHELXS86*; Sheldrick, 1986), thus obtaining all non-H atoms. Full-matrix least-squares refinement with *SHELX76* (Sheldrick, 1976) minimized $\sum w(\Delta F)^2$ for 218 parameters with unit weight. Final $R =$

Table 3. Comparison of some molecular parameters

	Leucoxinium ion	Isoboldinium ion	Bulbocapninium ion	This work
Angles (°)				
C1...C13—C12	174.4 (1)	178.2 (4)	177.2 (2)	174.4 (5)
C9...C12—C13	177.1 (2)	177.5 (4)	173.4 (2)	169.4 (5)
Torsion angles (°)				
C16—C13—C12—C7	13.6 (4)	21.2 (8)	28.4 (4)	28.5 (6)
C14—C13—C12—C11	15.0 (5)	20.5 (10)	31.8 (5)	34.1 (7)
Angles (°) between plane normals of rings <i>A</i> and <i>D</i>				
	15.5	21.1	30.2 (1)	34.4 (2)

0.063, $(\Delta/\sigma)_{\max} = 0.14$, max. peak height in final $\Delta\rho$ map is $0.28 \text{ e } \text{Å}^{-3}$. H atoms bound to C atoms were generated from assumed geometries while that linked to the O atom was located in a difference Fourier map; their positions were not refined. A common isotropic temperature factor was refined to $U = 0.071 (5) \text{ Å}^2$. Scattering factors were taken from *SHELX76*. Calculations were performed on a PC/AT computer.

Discussion. The final atomic coordinates of non-H atoms, given in Table 1,* were chosen in accordance with the absolute configuration of bulbocapnine methiodide which was established by Wei, Basu, Einstein & Hingerty (1984). According to the CIP code (Cahn, Ingold & Prelog, 1956) the asymmetric C atom C5 of bulbocapnine has an *S* configuration. The bond lengths and angles for non-H atoms are listed in Table 2. The molecular scheme is shown in Fig. 1.

The mean value of three C—N—C angles in the five-membered ring is $110.0 (4)^\circ$ indicating sp^3 hybridization of the N atom. From the puckering parameters (Cremer & Pople, 1975) of rings *B*, $Q = 0.428 (7) \text{ Å}$, $\varphi = 147.4 (9)^\circ$, $\theta = 53.7 (8)^\circ$, and *C*, $Q = 0.548 (6) \text{ Å}$, $\varphi = 19.8 (6)^\circ$, $\theta = 71.5 (6)^\circ$, it can be seen that the former assumes a half-chair conformation, whereas the latter is close to a screw-boat shape.

An aspect of structure which is of considerable interest in the aporphine alkaloid series is the degree of twist about the chiral axis C12—C13. The two torsion angles C16—C13—C12—C7 and C14—C13—C12—C11 (Klyne & Prelog, 1960) are $28.5 (6)^\circ$ and $34.1 (7)^\circ$ respectively. The dihedral angle between the least-squares planes for rings *A* and *D* is $34.4 (2)^\circ$. A comparison of these molecular features with those for the leucoxine and isoboldine hydro-

bromide (Brown & Hall, 1977) and bulbocapnine methiodide (Wei, Basu, Einstein & Hingerty, 1984) is given in Table 3. It can be seen that the twist of rings *A* and *D* around the C12—C13 bond is considerably greater in the title structure. O1 lies $0.105 (4) \text{ Å}$ below the plane of ring *A* while O4 is $0.192 (4) \text{ Å}$ above the plane of ring *D*. All these results indicate that the biphenyl system in this molecule is appreciably strained in order to minimize the interaction between O1 and O4.

H4 participates in a bifurcated hydrogen-bond interaction. The intramolecular component of the bifurcated hydrogen bond with the parameters $O4 \cdots O3 = 2.684 (6)$, $H4 \cdots O3 = 2.31 (7) \text{ Å}$, $O4-H4 \cdots O3 = 111 (6)^\circ$ is the cause of the small C11—C10—O3 bond angle of $113.6 (4)^\circ$. The molecules are bound together by an infinite chain of $OH \cdots N$ hydrogen bonds along the *b* axis with the following parameters: $O4 \cdots N(x, 1+y, z) = 2.834 (6)$, $H4 \cdots N = 2.15 (6) \text{ Å}$, $OH \cdots N = 148 (6)^\circ$.

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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 54315 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.