C(27)—H^R 8 (4) and H—C(25)—C(27)—H^S −112 (4)° in the crystal (Karplus, 1959).

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Structure of Capnoidine, a Phthalide Isoquinoline Alkaloid

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Abstract. $C_{20}H_{17}NO_6$, M_r = 367·36, orthorhombic, $P2_12_12_1$, a = 7·446 (3), b = 13·239 (5), c = 16·634 (5) Å, V = 1640 (1) Å^3, Z = 4, D_x = 1·488 Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, μ = 0·104 mm^{-1}, F(000) = 768, T = 293 (1) K, R = 0·038 for 1300 unique observed reflections. Ring *B* has almost screw-boat shape. Both C5 and C11 chiral centers possess the *R* configuration. The molecule consists of two approximate planes of atoms with a dihedral angle of 42·3 (2)° and joined by the C5—C11 bond. The epimer of capnoidine is bicuculline.

Introduction. In the course of our studies of natural products we investigated the crystal and molecular structure of capnoidine isolated from *Corydalis cava* collected from Vojvodinas Danube region. Extraction, isolation, separation and identification of capnoidine were performed as described by Slavik & Slavikova (1961), Preininger, Šimanek, Gašić & Šantavy (1973) and Gašić, Popović & Dragutinović (1985). The interest in this work lies in the accurate determination of molecular conformation of the compound which might assist in a better understanding of its biological action, studies of which are in progress.

Experimental. A crystal 0·22 × 0·35 × 0·15 mm was mounted on a CAD-4 diffractometer (University of Berne) using graphite-monochromated Mo $K\alpha$ radiation. Cell constants were refined by a least-squares fit for 22 centered reflections with θ range from 4·2 to 8·5°. Intensity data were collected with ω–2θ scan in the range $2\theta < 50°$; h 0 to 8, k 0 to 15, l 0 to 19. The systematic absences (h00: $h = 2n + 1$, 0k0: $k = 2n + 1$, 00l: $l = 2n + 1$) corresponded to those of orthorhombic space group $P2_12_12_1$. Standard reflection ($\bar{2}01$) was measured every 200 min, but no intensity variations were recorded. 1669 unique reflections, 1300 with $I > 3·5\sigma(I)$ were considered as observed. Data were corrected for Lorentz and polarization effects. The structure was solved using *SHELXS*86 (Sheldrick, 1986) with all non-H atoms obtained from the *E* map (R = 0·173). Full-matrix least-squares (on F) isotropic (R = 0·096) and anisotropic refinement with program *SHELX*76 (Sheldrick, 1976) minimized $\sum w(\Delta F)^2$ for 245 parameters with unit weights. Final R = 0·038, wR = 0·038, $(\Delta/\sigma)_{\max}$ = 0·007. Positions of H atoms were generated from assumed geometries and their positions were taken into account without refinement in structure-factor calculations with common isotropic temperature factor U = 0·072 (4) Å^2. A final

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Table 1. *Fractional atomic coordinates* (× 10^4) *and equivalent isotropic temperature factors* (× 10^3)

$$U_{eq} = \tfrac{1}{3}(U_{11} + U_{22} + U_{33}).$$

	x	y	z	U_{eq}(Å^2)
C1	3046 (6)	−313 (4)	−9879 (3)	51 (1)
C2	2975 (6)	−508 (3)	−9042 (3)	44 (1)
C3	3645 (6)	237 (3)	−8433 (3)	52 (1)
C4	4443 (6)	−326 (4)	−7729 (3)	54 (1)
C5	2050 (6)	−1611 (3)	−7870 (2)	38 (1)
C6	2335 (5)	−1433 (3)	−8765 (2)	36 (1)
C7	1773 (6)	−2193 (3)	−9300 (3)	39 (1)
C8	1862 (6)	−1974 (3)	−10098 (3)	46 (1)
C9	2464 (6)	−1057 (4)	−10378 (3)	50 (1)
C10	1284 (8)	−1901 (4)	−11399 (3)	64 (2)
C11	47 (6)	−1460 (3)	−7706 (2)	37 (1)
C12	−1073 (6)	95 (3)	−7296 (3)	42 (1)
C13	−1232 (5)	−602 (3)	−6614 (2)	34 (1)
C14	−1910 (6)	−487 (3)	−5858 (2)	37 (1)
C15	−1924 (6)	−1305 (3)	−5345 (2)	41 (1)
C16	−1278 (7)	−2237 (3)	−5556 (3)	50 (1)
C17	−581 (6)	−2364 (3)	−6341 (3)	47 (1)
C18	−580 (5)	−1543 (3)	−6841 (2)	37 (1)
C19	−3020 (7)	42 (4)	−4687 (3)	54 (1)
C20	3883 (7)	−1494 (5)	−6663 (3)	69 (1)
N	3095 (5)	−951 (3)	−7343 (2)	48 (1)
O1	1441 (5)	−2593 (2)	−10745 (2)	59 (1)
O2	2414 (5)	−1052 (3)	−11210 (2)	69 (1)
O3	−2673 (5)	333 (2)	−5503 (2)	53 (1)
O4	−2695 (5)	−1019 (2)	−4624 (2)	60 (1)
O5	−424 (4)	−429 (2)	−7926 (1)	41 (1)
O6	−1452 (5)	975 (2)	−7345 (2)	62 (1)

difference Fourier map showed excursions of density from −0·09 to +0·05 e Å^{-3}. Scattering factors were from *SHELX76*. All calculations were performed on a PC/AT computer.

Discussion. Atomic coordinates of non-H atoms are listed in Table 1.* The bond lengths and angles for non-H atoms are listed in Table 2. The molecular geometry with atomic numbering is shown in Fig. 1.

The capnoidine molecule consists of two approximate planes of atoms defined by two aromatic rings, *A* and *D*, with a dihedral angle of 42·3 (2)° between them. The two planes are joined by the C5—C11 bond. The torsion angle N—C5—C11—O5 is 64·5 (4)°. The epimer of capnoidine is bicuculline (Gorinsky & Moss, 1973). The corresponding dihedral angle of two approximate planes and torsion angle in bicuculline are 14·3 and 164·1° respectively. Based on a comparison of the NMR, ORD and CD spectral data, both C5 and C11 chiral centers of capnoidine possess the *R* configuration, while the corresponding atoms of its epimer bicuculline are of *R* and *S* configuration, respectively (Teitel, O'Brien & Brossi, 1972).

Table 2. *Bond distances* (Å), *bond angles* (°) *and selected torsion angles* (°)

C1	C2	1·417 (7)	C11	C18	1·517 (5)
C1	C9	1·359 (7)	C11	O5	1·456 (5)
C2	C3	1·499 (6)	C12	C13	1·467 (6)
C2	C6	1·392 (6)	C12	O5	1·346 (5)
C3	C4	1·510 (7)	C12	O6	1·201 (5)
C4	N	1·451 (6)	C13	C14	1·364 (5)
C5	C6	1·522 (5)	C13	C18	1·389 (6)
C5	C11	1·529 (6)	C14	C15	1·379 (5)
C5	N	1·462 (5)	C14	O3	1·360 (5)
C6	C7	1·407 (6)	C15	C16	1·370 (6)
C7	C8	1·360 (7)	C15	O4	1·382 (5)
C8	C9	1·375 (7)	C16	C17	1·415 (7)
C8	O1	1·389 (6)	C17	C18	1·369 (6)
C9	O2	1·384 (6)	C19	O3	1·434 (6)
C10	O1	1·427 (6)	C19	O4	1·429 (6)
C10	O2	1·439 (7)	C20	N	1·463 (7)

C2	C1	C9	117·1 (4)	O5	C12	O6	122·1 (4)
C1	C2	C3	122·1 (4)	C12	C13	C14	132·3 (4)
C1	C2	C6	119·9 (4)	C12	C13	C18	109·0 (3)
C3	C2	C6	118·0 (4)	C14	C13	C18	118·7 (3)
C2	C3	C4	109·3 (4)	C13	C14	C15	119·1 (4)
C3	C4	N	110·6 (4)	C13	C14	O3	130·1 (4)
C6	C5	C11	106·9 (3)	C15	C14	O3	110·8 (3)
C6	C5	N	114·8 (3)	C14	C15	C16	123·1 (4)
C11	C5	N	109·5 (3)	C14	C15	O4	109·0 (3)
C2	C6	C5	120·5 (3)	C16	C15	O4	127·9 (4)
C2	C6	C7	121·4 (4)	C15	C16	C17	118·2 (4)
C5	C6	C7	117·8 (3)	C16	C17	C18	117·8 (4)
C6	C7	C8	116·8 (4)	C11	C18	C13	107·5 (3)
C7	C8	C9	122·3 (4)	C11	C18	C17	129·4 (4)
C7	C8	O1	128·3 (4)	C13	C18	C17	123·1 (4)
C9	C8	O1	109·4 (4)	O3	C19	O4	107·6 (4)
C1	C9	C8	122·5 (5)	C4	N	C5	116·3 (4)
C1	C9	O2	128·0 (5)	C4	N	C20	110·2 (4)
C8	C9	O2	109·5 (4)	C5	N	C20	112·5 (4)
O1	C10	O2	106·7 (4)	C8	O1	C10	103·3 (4)
C5	C11	C18	117·4 (3)	C9	O2	C10	103·3 (4)
C5	C11	O5	108·2 (3)	C14	O3	C19	105·8 (3)
C18	C11	O5	103·4 (3)	C15	O4	C19	106·0 (3)
C13	C12	O5	107·9 (3)	C11	O5	C12	112·0 (3)
C13	C12	O6	130·0 (4)				

C3	C4	N	C5	50·3 (5)	C6	C5	C11	C18	−176·9 (3)
C3	C4	N	C20	179·9 (4)	C6	C5	C11	O5	−60·5 (4)
C4	C3	C2	C6	31·5 (5)	C11	C5	N	C20	102·6 (4)
C4	N	C5	C6	−8·7 (5)	C18	C11	C5	N	−52·0 (4)
C6	C2	C1	C9	0·3 (7)	N	C5	C11	O5	64·5 (4)

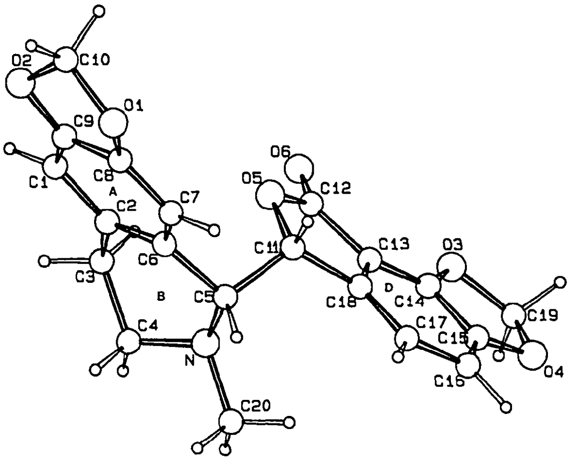

Fig. 1. Perspective view of the molecule.

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* Lists of structure factors, anisotropic thermal parameters, least-squares-planes data and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54195 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Comparison of some molecular features of capnoidine and bicuculline*

	Capnoidine	Bicuculline
Dihedral angle between planes *A* and *D* (°)	42·3 (2)	14·3
Torsion angle N—C5—C11—O5 (°)	64·5 (4)	164·1
Conformation of asymmetric centers C5, C11	*R*, *R*	*R*, *S*
Ring *B*	Almost screw-boat	Half-chair

In the five-membered rings containing methylenedioxy bridges, there are two types of C—O single bonds: the shorter type in the range 1·360 (5)–1·389 (6) Å involve the C atom of an aromatic ring, whereas the longer type in the range 1·427 (6)–1·439 (7) Å involve the C atom of the CH_2 groups. The mean value of the three C—N—C angles is 113·0 (4)° indicating an sp^3 hybridization of the N atom.

In accordance with the puckering parameters of Cremer & Pople (1975) ring *B* [Q = 0·509 (5) Å, φ = 101·9 (6), θ = 68·7 (5)°] has almost a screw-boat shape, while in bicuculline the same ring possesses a half-chair conformation. A comparison of some molecular features of capnoidine and bicuculline is given in Table 3.

The interaction between the molecules, which lie parallel to the *c* axis (Fig. 2), occurs only through van der Waals contacts.

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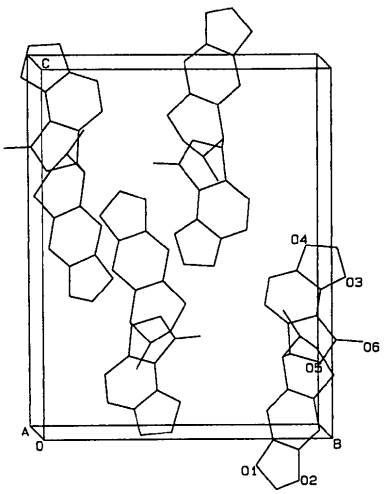

Fig. 2. Crystal packing diagram.

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Structures of Two 1,2,3-Triazine *N*-Oxides

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Abstract. (I) 6-Methyl-4-phenyl-1,2,3-triazine 1-oxide, $C_{10}H_9N_3O$, M_r = 187·2, monoclinic, Pc, a = 10·333 (1), b = 6·153 (1), c = 7·058 (2) Å, β = 92·48 (2)°, V = 448·2 (2) Å^3, Z = 2, D_x = 1·387 Mg m^{-3}, λ(Cu $K\alpha_1$) = 1·5405 Å, μ = 0·782 mm^{-1}, F(000) = 196, T = 295 K, final R = 0·036 for 656 observed reflections. (II) 4,5,6-Tris(*p*-tolyl)-1,2,3-triazine 2-oxide, $C_{24}H_{21}N_3O$, M_r = 367·4, monoclinic, $P1$, a = 9·330 (1), b = 10·073 (1), c = 5·903 (1) Å, α = 94·32 (1), β = 98·62 (1), γ = 113·01 (1)°, V = 499·4 (1) Å^3, Z = 1, D_x = 1·222 Mg m^{-3}, λ(Cu $K\alpha_1$) = 1·5405 Å, μ = 0·612 mm^{-1}, F(000) = 194, T = 295 K, final R = 0·039 for 1453 observed reflections. The bond distances in the *N*-oxides (N^+—O^-) are 1·264 (6) (I) and 1·243 (4) (II) Å. The dihedral angles between the triazine and phenyl rings are 22·7 (4) in (I) and 34·2 (5), 80·1 (5) and 34·5 (6)° in (II).