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Structure of Isocorypalmine

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Abstract. $C_{20}H_{23}NO_4$, $M_r = 341.41$, trigonal, $P3_2$, $a = 10.131\ (1)$, $c = 14.913\ (3)$ Å, $\gamma = 120°$, $V = 1325.5\ (6)$ Å^3, $Z = 3$, $D_x = 1.283$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.7107 Å, $\mu = 0.083$ mm^{-1}, $F(000) = 546$, $T = 293\ (1)$ K, $R = 0.031$ for 1977 reflections with $I > 3\sigma(I)$. According to the puckering parameters [Cremer & Pople (1975) *J. Am. Chem. Soc.* **97**, 1354–1358] of the *trans* fused ring *B* [$Q = 0.564\ (4)$ Å, $\varphi = 331.7\ (6)$, $\theta = 52.2\ (4)°$] and ring *C* [$Q = 0.515\ (4)$ Å, $\varphi = 136.0\ (6)$, $\theta = 50.5\ (5)°$] the former assumes an almost perfect half-chair conformation, while the latter has a transitional form between sofa and half-chair. The dihedral angle between the two phenyl rings *A* and *D* is 33.8 (1)°. The mean of the three C—N—C angles is 110.2 (2)° indicating sp^3 hybridization of the N atom. Molecules are held together by hydrogen bonds. O1 participates in an intra- and an intermolecular hydrogen bond in which H(O1) is bifurcated. These bonds have the following parameters: O1—H(O1) 0.75 (6), H(O1)···O2 2.33 (6), O1···O2 2.721 (4) Å, O1—H(O1)···O2 113 (2)° and H(O1)···N$^{\mathrm{i}}$ 2.15 (5), O1···N$^{\mathrm{i}}$ 2.849 (4) Å, O1—H(O1)···N$^{\mathrm{i}}$ 155 (2)° [(i) = $1 - x + y$, $2 - x$, $\frac{1}{3} + z$], respectively.

Table 1. *Fractional atomic coordinates* (× 10^4) *of non*-H *atoms and* U_{eq} *values* (Å^2 × 10^3)

$$U_{\mathrm{eq}} = \tfrac{1}{3}[(U_{11} + U_{22} - U_{12})(aa^*)^2 + U_{33}(cc^*)^2].$$

	x	y	z	U_{eq}
C1	6680 (3)	7947 (3)	2941 (2)	45 (1)
C2	7445 (3)	7133 (3)	2815 (2)	44 (1)
C3	7563 (4)	6605 (5)	1882 (2)	57 (1)
C4	7769 (4)	5236 (4)	1940 (2)	54 (1)
C5	9438 (3)	4354 (3)	2452 (2)	47 (1)
C6	10751 (3)	4638 (3)	3052 (2)	41 (1)
C7	11466 (3)	3786 (3)	2922 (2)	46 (1)
C8	12710 (3)	4045 (3)	3446 (2)	51 (1)
C9	13212 (3)	5131 (3)	4108 (2)	51 (1)
C10	12471 (3)	5951 (3)	4259 (2)	46 (1)
C11	11236 (3)	5722 (3)	3740 (2)	41 (1)
C12	10398 (3)	6580 (3)	3919 (2)	43 (1)
C13	8841 (3)	5839 (3)	3457 (2)	41 (1)
C14	8058 (3)	6776 (3)	3547 (2)	42 (1)
C15	7885 (3)	7249 (3)	4403 (2)	43 (1)
C16	7134 (3)	8050 (3)	4528 (2)	43 (1)
C17	6524 (3)	8413 (3)	3782 (2)	44 (1)
C18	4947 (5)	9382 (5)	3250 (3)	65 (1)
C19	10078 (6)	1168 (4)	2521 (4)	86 (1)
C20	14366 (6)	3116 (7)	3886 (4)	93 (2)
N	9100 (2)	5600 (2)	2504 (2)	42 (1)
O1	6955 (3)	8426 (3)	5379 (2)	57 (1)
O2	5769 (2)	9195 (2)	3970 (1)	55 (1)
O3	10944 (3)	2725 (2)	2232 (2)	59 (1)
O4	13361 (3)	3168 (3)	3234 (2)	73 (1)

Experimental. Isocorypalmine, the berberine alkaloid, was isolated from the aerial parts of

Table 2. *Interatomic distances* (Å) *and angles* (°)

C1—C2	1.398 (5)	C9—C10	1.389 (5)
C1—C17	1.377 (4)	C10—C11	1.389 (4)
C2—C3	1.517 (5)	C11—C12	1.512 (5)
C2—C14	1.390 (5)	C12—C13	1.531 (4)
C3—C4	1.505 (7)	C13—C14	1.517 (5)
C4—N	1.471 (4)	C13—N	1.487 (4)
C5—C6	1.507 (4)	C14—C15	1.405 (4)
C5—N	1.466 (4)	C15—C16	1.375 (5)
C6—C7	1.390 (5)	C16—C17	1.408 (5)
C6—C11	1.400 (4)	C16—O1	1.363 (4)
C7—C8	1.392 (4)	C17—O2	1.377 (4)
C7—O3	1.388 (4)	C18—O2	1.428 (5)
C8—C9	1.373 (4)	C19—O3	1.435 (4)
C8—O4	1.382 (5)	C20—O4	1.428 (7)

C2—C1—C17	121.1 (3)	C11—C12—C13	112.1 (2)
C1—C2—C3	119.7 (3)	C12—C13—C14	112.7 (2)
C1—C2—C14	120.0 (3)	C12—C13—N	106.7 (2)
C3—C2—C14	120.3 (3)	C14—C13—N	112.1 (2)
C2—C3—C4	110.1 (3)	C2—C14—C13	122.3 (3)
C3—C4—N	109.5 (3)	C2—C14—C15	118.5 (3)
C6—C5—N	111.6 (2)	C13—C14—C15	119.2 (3)
C5—C6—C7	118.9 (3)	C14—C15—C16	121.7 (3)
C5—C6—C11	121.0 (3)	C15—C16—C17	119.5 (3)
C7—C6—C11	120.1 (3)	C15—C16—O1	118.8 (3)
C6—C7—C8	120.6 (3)	C17—C16—O1	121.7 (3)
C6—C7—O3	118.0 (3)	C1—C17—C16	119.3 (3)
C8—C7—O3	121.4 (3)	C1—C17—O2	125.1 (3)
C7—C8—C9	119.5 (3)	C16—C17—O2	115.6 (3)
C7—C8—O4	115.4 (3)	C4—N—C5	109.5 (2)
C9—C8—O4	125.1 (3)	C4—N—C13	111.9 (2)
C8—C9—C10	120.1 (3)	C5—N—C13	109.1 (2)
C9—C10—C11	121.4 (3)	C17—O2—C18	116.8 (3)
C6—C11—C10	118.2 (3)	C7—O3—C19	114.6 (3)
C6—C11—C12	120.2 (3)	C8—O4—C20	116.5 (4)
C10—C11—C12	121.6 (3)		

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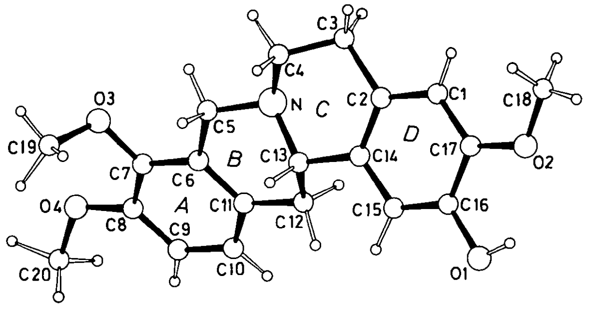

Fig. 1. Perspective view of the molecule.

Corydalis solida collected at Fruška Gora (Vojvodina). The plant material was extracted with ethanol and separated into two portions. Isocorypalmine was isolated from the phenolic part of the first portion by column chromatography on Al_2O_3 and crystallized from the ether fraction. Data collected on crystal (rhombohedron) 0.61 × 0.82 × 0.83 mm mounted on a CAD-4 diffractometer (Ljubljana) using graphite-monochromated Mo *Kα* radiation. Cell constants were refined by least-squares fit of 50 reflections collected in the range $12.0 < \theta < 17.9°$. Reflection condition for $00l$: $l = 3n$. Data were collected by $\omega/2\theta$ scan in range $4.64 < 2\theta < 56.03°$ with h −13 to 13, k −13 to 13, l −19 to 19. Of 11087 measured reflections ($R_{\rm int} = 0.021$), 1977 with $I > 3\sigma(I)$ were taken as observed. Standard reflections ($04\bar{6}$, $32\bar{6}$, 513) were monitored 56 times. Intensity change 0.50%. The phase problem was solved using *SHELXS86* (Sheldrick, 1986). Full-matrix least-squares refinement with *SHELX76* (Sheldrick, 1976) minimized $\sum w(\Delta F)^2$ for 317 parameters with unit weight. Neither absorption nor extinction corrections were applied. Final $R = 0.031$, $(\Delta/\sigma)_{\max} = 0.70$. A final difference Fourier map showed excursion of density from −0.07 to 0.05 e Å^{-3}. H atoms were found in difference Fourier map and refined isotropically. Scattering factors were taken from *SHELX76*. Calculations were performed on an IBM 43/41 computer (Novi Sad). Atomic coordinates of non-H atoms are listed in Table 1.* The bond lengths and angles for non-H atoms are presented in Table 2. A perspective view of the molecule is shown in Fig. 1.

Related literature. According to Corrodi & Hardegger (1956) (+)-isocorypalmine has the same configuration as (+)-tetrahydropalmatine.

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* Lists of structure factors, anisotropic thermal parameters, torsion angles, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54707 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: KA0008]

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Abstract. $C_{20}H_{23}NO_4$, $M_r = 341.41$, orthorhombic, $P2_12_12_1$, $a = 7.536$ (5), $b = 9.926$ (8), $c = 23.397$ (16) Å, $V = 1750$ (2) Å^3, $Z = 4$, $D_x = 1.296$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.7107 Å, $\mu = 0.084$ mm^{-1}, $F(000) = 728$, $T = 293$ (1) K, $R = 0.038$ for 1570 reflections with $I > 3.5\sigma(I)$. Both *B* and *C* rings have a screw-boat conformation and the latter is almost perfect. The C—N—C angles indicate sp^3

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