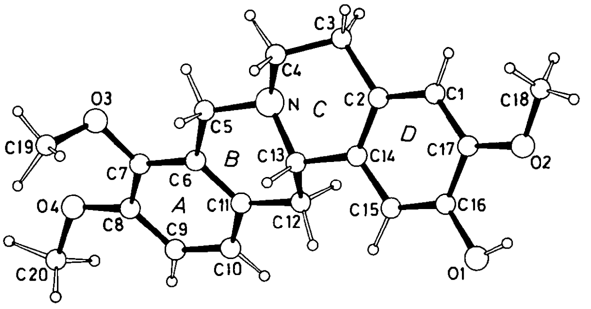

Fig. 1. Perspective view of the molecule.

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 $K\alpha$ 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 *SHELXS*86 (Sheldrick, 1986). Full-matrix least-squares refinement with *SHELX*76 (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 *SHELX*76. 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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Structure of Isocorydine

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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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hybridization of the N atom. Bonds O1—C17 and O4—C19 are in the planes of rings *A* and *D*, respectively, as indicated by the corresponding torsion angles, while O2—C18 is perpendicular to the plane of ring *A*. The C20 methyl group is in an equatorial position, as indicated by the torsion angles. The hydroxyl group forms a strong intramolecular hydrogen bond [O3—H3 0.80 (5), H3···O2 1.78 (5), O3···O2 2.547 (4) Å, O3—H3···O2 160 (5)°]. Molecules are held together by van der Waals contacts.

Experimental. Isocorydine, the aporphine alkaloid, was isolated from aerial parts of *Corydalis solida* collected from the mountain Fruška Gora (Vojvodina), but it was not found in the same plant collected in the locality of Mosor (Dalmacija). Plant material was extracted and alkaloids were separated by the method described by Gašić, Popović & Dragutinović (1985). Data collected on a crystal of dimensions 0.36 × 0.36 × 0.36 mm mounted on a CAD-4 diffractometer (Berne) with graphite-monochromated Mo $K\alpha$ radiation. Cell constants were refined by least-squares fit for 22 reflections with $2.8 < \theta < 8.6°$. Data were collected by $\omega/2\theta$ scan in the range $\theta < 25°$ with $0 \le h \le 9$, $0 \le k \le 12$, $0 \le l \le 28$. Of 2024 measured reflections, 1570 with $I > 3.5\sigma(I)$ were taken as observed. Standard reflection 122 was measured every 200 min and no intensity variations were recorded. Data were corrected for Lorentz and polarization effects but not for absorption. Structure was solved using *SHELXS*86 (Sheldrick, 1986) with all non-H atoms from an *E* map. Full-matrix least-squares refinement with *SHELX*76 (Sheldrick, 1976) minimized $\sum w(\Delta F)^2$ for 239 parameters with unit weight. Final $R = 0.038$, $(\Delta/\sigma)_{\max} = 0.13$. Positions of H atoms, except H3 which was found from the difference Fourier map, were generated from assumed geometries and their positions were taken into account in structure-factor calculations with refined isotropic temperature factors (riding mode). Max. final residual electron density ±0.14 e Å^{-3}. Scattering factors from *SHELX*76. All calculations were performed on a PC/AT computer. The geometrical parameters were computed with *CSU* (Vicković, 1988) and the figure drawn with *PLUTO* (Motherwell & Clegg, 1978). The coordinates of the non-H atoms and equivalent isotropic thermal parameters are listed in Table 1,* and bond distances, bond angles and selected torsion angles in Table 2. A perspective view of the molecule is shown in Fig. 1.

Table 1. *Coordinates* (× 10^4) *and equivalent isotropic thermal parameters* (Å^2 × 10^3) *for non*-H *atoms*

	x	*y*	*z*	U_{eq}
O1	3371 (4)	9336 (3)	4164 (1)	58 (1)
O2	2184 (3)	6970 (2)	4432 (1)	38 (1)
O3	1809 (3)	4552 (3)	4089 (1)	45 (1)
O4	159 (4)	2438 (3)	4432 (1)	57 (1)
N	−3731 (4)	8445 (3)	2761 (1)	52 (1)
C1	1198 (5)	9600 (4)	3400 (2)	52 (1)
C2	−296 (5)	9089 (4)	3129 (2)	49 (1)
C3	−1161 (7)	9901 (5)	2660 (2)	75 (1)
C4	−2549 (6)	9089 (5)	2341 (2)	68 (1)
C5	−2760 (5)	7362 (4)	3053 (1)	41 (1)
C6	−3814 (5)	6784 (4)	3550 (1)	40 (1)
C7	−2807 (4)	5634 (4)	3807 (1)	35 (1)
C8	−3650 (5)	4462 (4)	3960 (2)	45 (1)
C9	−2716 (5)	3381 (4)	4176 (2)	50 (1)
C10	−893 (5)	3447 (4)	4227 (2)	42 (1)
C11	5 (4)	4628 (4)	4065 (1)	34 (1)
C12	−942 (4)	5753 (3)	3874 (1)	31 (1)
C13	−157 (4)	7080 (3)	3719 (1)	33 (1)
C14	1313 (4)	7641 (3)	3991 (1)	34 (1)
C15	1974 (5)	8902 (4)	3839 (2)	45 (1)
C16	−1008 (5)	7862 (4)	3291 (1)	39 (1)
C17	4135 (7)	10593 (4)	4023 (2)	88 (2)
C18	1532 (5)	7244 (5)	4992 (2)	57 (1)
C19	−707 (6)	1248 (4)	4634 (2)	63 (1)
C20	−5319 (6)	7932 (5)	2477 (2)	69 (1)

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

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

O1	C15	1.368 (5)	C3	C4	1.517 (7)
O1	C17	1.413 (5)	C5	C6	1.521 (4)
O2	C14	1.392 (4)	C5	C16	1.516 (5)
O2	C18	1.426 (5)	C6	C7	1.497 (5)
O3	C11	1.363 (4)	C7	C8	1.373 (5)
O4	C10	1.364 (5)	C7	C12	1.419 (4)
O4	C19	1.430 (5)	C8	C9	1.379 (6)
N	C4	1.472 (5)	C9	C10	1.381 (5)
N	C5	1.469 (5)	C10	C11	1.406 (5)
N	C20	1.461 (5)	C11	C12	1.399 (5)
C1	C2	1.388 (6)	C12	C13	1.489 (4)
C1	C15	1.370 (6)	C13	C14	1.394 (4)
C2	C3	1.510 (7)	C13	C16	1.420 (4)
C2	C16	1.384 (6)	C14	C15	1.393 (5)

C15	O1	C17	117.5 (3)	O4	C10	C11	115.3 (3)
C14	O2	C18	115.3 (3)	C9	C10	C11	119.7 (4)
C10	O4	C19	117.2 (3)	O3	C11	C10	115.0 (3)
C4	N	C5	109.1 (3)	O3	C11	C12	124.5 (3)
C4	N	C20	110.1 (3)	C10	C11	C12	120.4 (3)
C5	N	C20	111.4 (3)	C7	C12	C11	118.3 (3)
C2	C1	C15	120.3 (4)	C7	C12	C13	116.1 (3)
C1	C2	C3	119.2 (4)	C11	C12	C13	125.6 (3)
C1	C2	C16	120.7 (4)	C12	C13	C14	123.9 (3)
C3	C2	C16	120.1 (4)	C12	C13	C16	118.4 (3)
C2	C3	C4	111.8 (4)	C14	C13	C16	117.6 (3)
N	C4	C3	108.6 (4)	O2	C14	C13	121.5 (2)
N	C5	C6	111.8 (3)	O2	C14	C15	116.8 (3)
N	C5	C16	111.4 (3)	C13	C14	C15	121.8 (3)
C6	C5	C16	107.3 (3)	O1	C15	C1	125.9 (4)
C5	C6	C7	109.3 (3)	O1	C15	C14	114.6 (3)
C6	C7	C8	121.1 (3)	C1	C15	C14	119.5 (4)
C6	C7	C12	118.9 (3)	C2	C16	C5	121.7 (3)
C8	C7	C12	120.0 (3)	C2	C16	C13	119.9 (3)
C7	C8	C9	121.2 (4)	C5	C16	C13	118.2 (3)
C8	C9	C10	120.2 (4)				
O4	C10	C9	125.0 (4)				

C16	C13	C12	C7	29.2 (4)	C15	C14	O2	C18	89.6 (4)
C14	C13	C12	C11	34.4 (4)	C3	C4	N	C20	166.3 (4)
C14	C15	O1	C17	178.4 (3)	C16	C5	N	C20	173.6 (3)
C11	C10	O4	C19	176.4 (3)					

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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 54708 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: KA0010]

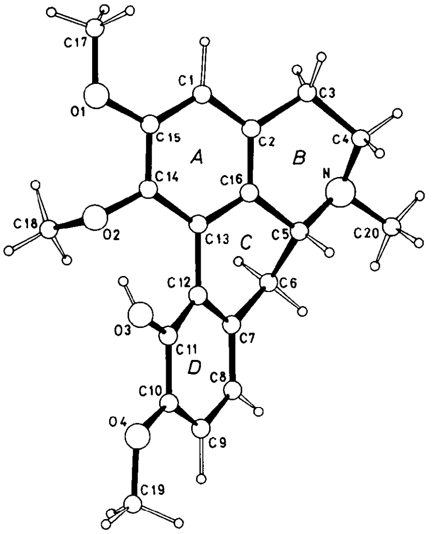

Fig. 1. Perspective view of the molecule.

Related literature. According to Berezhinskaya, Aleshinskaya & Aleshkima (1968) isocorydine exhibits adrenolytic action in anaesthetized cats and rabbits and also anti-adrenergic effects. The dihedral angle between the least-squares planes for phenyl rings *A* and *D* is 32.3 (1)°, whereas this angle is 15.5 and 21.1° in leucoxine and isoboldine hydrobromide (Brown & Hall, 1977), 30.2 (1)° in bulbocapnine methiodide (Wei, Basu, Einstein & Hingerty, 1984), 34.4 (2)° in bulbocapnine (Ribár, Mészáros, Gašić, Kanyó & Engel, 1991) and 24.9° in nanteine (Ribár, Mészáros, Engel, Gašić & Kanyó, 1991).

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Structure of 3,3-Diethyl-1,2-diphenyl-1,4,2-diazaphospholidin-5-one 2-Oxide

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Abstract. $C_{18}H_{21}N_2O_2P$, $M_r = 328.35$, monoclinic, $P2_1/c$, $a = 9.232$ (3), $b = 14.561$ (1), $c = 12.446$ (3) Å, $\beta = 91.07$ (1)°, $V = 1673$ (2) Å^3, $Z = 4$, $D_x = 1.304$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.54184 Å, $\mu = 1.54$ mm^{-1}, $F(000) = 696$, room temperature, $R = 0.050$ for 2123 reflections with $I > 3\sigma(I)$. The five-membered ring is close to planar with deviations from the least-squares plane in the range −0.069 (2) to 0.075 (1) Å. The P—N and P—C distances within the ring are 1.687 (1) and 1.862 (1) Å, respectively. The phenyl group attached to P(1) adopts an axial position while the O atom and phenyl group attached to C(1) and N(1), respectively, adopt equatorial positions. Two molecules (related by a centre of symmetry) are connected by two intermolecular hydrogen bonds of length 2.879 (3) Å between N(2) and O(2), thus forming dimers in the crystal.

Experimental. A crystal with dimensions 0.3 × 0.3 × 0.3 mm was used for X-ray analysis on an Enraf–

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