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Structure of 3,5-Dihydrobenz[*f*]indolizin-3-one

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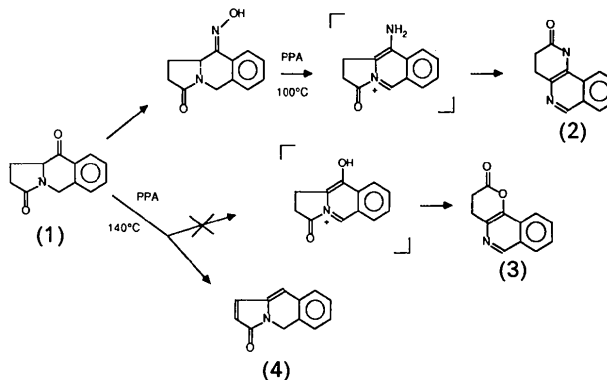
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Abstract. C₁₂H₉NO, *M_r* = 183.2, triclinic, *P* $\bar{1}$, *a* = 8.837 (6), *b* = 7.910 (6), *c* = 6.903 (5) Å, α = 108.2 (1), β = 77.7 (1), γ = 101.4 (1)°, *V* = 443.4 Å³, *Z* = 2, *D_x* = 1.372 g cm⁻³, *Mo K α* , λ = 0.71069 Å, μ = 0.5 cm⁻¹, *F*(000) = 192, *T* = 293 (2) K, *R* = 0.054 for 1626 reflections with *I* ≥ 3σ(*I*). Elemental analysis and the mass spectrum of the compound, and one- and two-dimensionally correlated NMR spectra are in good agreement with the results of the structure analysis, in which the molecule is found to be roughly planar.

Experimental. As described in the literature (Rigo & Kolocouris, 1984), fine powder of (1) (1 g, 4.97 mmol) was quickly added to hot (413 K), stirred polyphosphoric acid (20 g). The mixture was vigorously stirred for 1 h. The hot mixture was decanted over crushed ice (150 ml) and the aqueous solution was extracted with dichloromethane. The organic phase was washed with water, dried over K₂CO₃,

filtered and concentrated to give 70% yield of a red powder, which was then treated overnight in dichloromethane at room temperature, with activated carbon, filtered and concentrated in part; pure yellow crystals of (4), which turned red in air, were obtained by slow evaporation of the solvent at 437 K.



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Table 1. Fractional atomic coordinates ($\times 10^4$) and equivalent isotropic temperature factors ($\text{\AA}^2 \times 10^2$) with *e.s.d.*'s in parentheses

$$U_{\text{eq}} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
C(1)	8350 (3)	1192 (4)	-213 (4)	45 (2)
C(2)	8883 (3)	809 (4)	-2242 (5)	53 (3)
C(3)	7697 (3)	1119 (4)	-3283 (4)	44 (4)
N	6499 (2)	1698 (3)	-1732 (3)	38 (4)
C(5)	5095 (3)	2214 (4)	-2032 (4)	37 (3)
C(5a)	4018 (3)	2846 (4)	-7 (4)	35 (2)
C(6)	2641 (3)	3415 (4)	-36 (4)	50 (3)
C(7)	1629 (4)	4028 (5)	1779 (5)	59 (2)
C(8)	1982 (4)	4058 (5)	3645 (5)	62 (2)
C(9)	3347 (4)	3490 (4)	3703 (4)	52 (2)
C(9a)	4392 (4)	2888 (4)	1886 (4)	35 (3)
C(10)	5846 (3)	2329 (3)	1941 (3)	40 (3)
C(10a)	6833 (3)	1784 (4)	192 (4)	36 (3)
O	7711 (3)	924 (4)	-5117 (3)	66 (3)

Table 2. Selected bond lengths (\AA) and angles ($^\circ$) with *e.s.d.*'s in parentheses

C(1)—C(2)	1.336 (4)	C(5a)—C(6)	1.384 (4)
C(2)—C(10a)	1.450 (4)	C(5a)—C(9a)	1.404 (4)
C(2)—C(3)	1.486 (4)	C(6)—C(7)	1.386 (4)
C(3)—N	1.371 (3)	C(7)—C(8)	1.381 (5)
C(3)—O	1.225 (3)	C(8)—C(9)	1.381 (5)
N—C(5)	1.453 (3)	C(9)—C(9a)	1.400 (4)
N—C(10a)	1.399 (3)	C(9a)—C(10)	1.450 (4)
C(5)—C(5a)	1.516 (4)	C(10)—C(10a)	1.324 (3)
C(2)—C(1)—C(10a)	109.0 (3)	C(5a)—C(6)—C(7)	120.9 (3)
C(1)—C(2)—C(3)	108.6 (3)	C(6)—C(7)—C(8)	119.9 (3)
C(2)—C(3)—N	105.5 (2)	C(7)—C(8)—C(9)	120.0 (3)
C(2)—C(3)—O	130.0 (3)	C(8)—C(9)—C(9a)	120.8 (3)
N—C(3)—O	124.6 (3)	C(5a)—C(9a)—C(9)	118.9 (3)
C(3)—N—C(5)	124.7 (2)	C(5a)—C(9a)—C(10)	120.1 (3)
C(3)—N—C(10a)	110.9 (2)	C(9)—C(9a)—C(10)	121.0 (3)
C(5)—N—C(10a)	124.4 (2)	C(9a)—C(10)—C(10a)	120.0 (2)
N—C(5)—C(5a)	111.9 (2)	C(1)—C(10a)—N	106.1 (2)
C(5)—C(5a)—C(6)	118.8 (3)	C(1)—C(10a)—C(10)	132.0 (3)
C(5)—C(5a)—C(9a)	121.7 (3)	N—C(10a)—C(10)	121.9 (2)
C(6)—C(5a)—C(9a)	119.6 (3)		

Analysis: % exp. (% theor.): C; 78.23 (78.67); H; 5.32 (4.95); N; 7.43 (7.65).

Single yellow crystal (0.3 \times 0.5 \times 0.4 mm), Philips PW1100 four-circle diffractometer, room temperature, Mo $K\alpha$ radiation, lattice parameters from 25 reflections ($20 \leq \theta \leq 27^\circ$), 3165 intensities with $\sin \theta / \lambda \leq 0.7663 \text{ \AA}^{-1}$ in the ω - 2θ scan mode (width of scan fixed at 1.40° and $-14 \leq h \leq 14$, $-11 \leq k \leq 11$, $l \leq 11$), from which 1626 with $I \geq 3\sigma(I)$ used for structure refinement; variation of standard intensities 2%, measured every 2 h; Lp correction. Absorption and extinction ignored; structure solved by direct methods using *SHELXS86* (Sheldrick, 1985) and Fourier methods; anisotropic full-matrix least squares on *F* (*SHELX76*; Sheldrick, 1976), H atoms from $\Delta\rho$ synthesis, isotropic atoms not refined; atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV), for H atoms from Stewart, Davidson & Simpson (1965);

final $R = 0.054$, unit weights; $S = 2.06$; $\Delta\rho$ in final map = $+0.26$, -0.39 e \AA^{-3} ; $(\Delta/\sigma)_{\text{max}} = 0.09$.

Final atomic coordinates of non-H atoms and equivalent isotropic U 's are listed in Table 1; bond lengths and angles are listed in Table 2.*

Related literature. We recently described the synthesis of 1,2,3,5,10,10a-hexahydrobenz[*f*]indolizin-3,10-dione (1) (Rigo & Kolocouris, 1984). Martin, Scott, Agnew & Setescak (1986) found that the treatment of the oxime of this ketone under Beckman conditions (PPA, 373 K) gave 1,4-dihydrobenzo[*c*]-1,5-naphthyridin-2(3*H*)-one (2). In an attempt to obtain the oxygenated analogue (3) of the lactam (2), the ketone (1) was subjected to the same treatment. However, a new dehydration was observed, and the lactam (4) was obtained with 70% yield as the sole isolated product (see Scheme).

The crystal conformation and the atomic numbering are depicted in Fig. 1. The molecule is approximately planar, the maximum deviation from the mean plane being 0.039 (1) \AA for C(2), and there are no intermolecular contacts between non-H atoms shorter than the sums of the corresponding van der Waals radii.

Elemental analysis and the mass spectrum of the lactam (4) are in good agreement with the results of the structure analysis. This is verified by one- and

* 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 53540 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

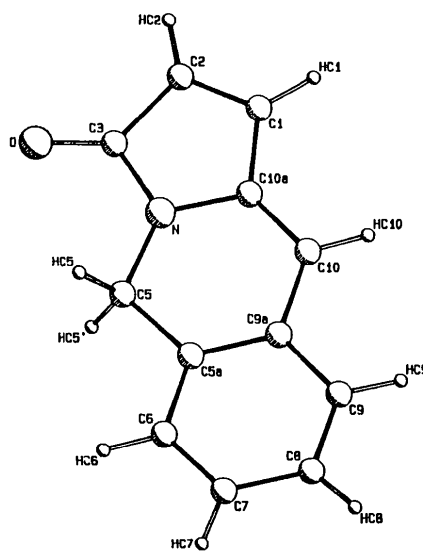


Fig. 1. Perspective view of the molecule (*PLUTO*; Motherwell & Clegg, 1978).

two-dimensionally correlated NMR spectra; in the ^1H spectrum, singlets at 4.89 p.p.m. ($2H$) and 6.18 p.p.m. ($1H$) are assigned respectively to H(C5) and H(C10), whereas the doublets ($J = 5.7$ Hz) at 6.28 p.p.m. ($1H$) and 7.12 p.p.m. ($1H$) are the H(C1) and H(C2) signals; the aromatic protons resonate between 7.15 and 7.25 p.p.m. The only ^{13}C aliphatic signal (47.5 p.p.m.) is assigned to C(5); ^1H - ^{13}C correlated spectra allow the assignment of the C(10) (110.4 p.p.m.), C(2) (124.3 p.p.m.) and C(1) (133.2 p.p.m.) signals. The carbonyl moiety of the amide group resonates at 169.2 p.p.m.

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Structure of (4*RS*)-6,7-Dimethoxy-4-phenyl-1,2,3,4-tetrahydroisoquinoline Hydrochloride

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Abstract. $\text{C}_{17}\text{H}_{20}\text{NO}_2^+ \cdot \text{Cl}^-$, $M_r = 305.80$, triclinic, $P\bar{1}$, $a = 5.959$ (1), $b = 14.823$ (2), $c = 19.307$ (2) Å, $\alpha = 107.53$ (1), $\beta = 95.96$ (2), $\gamma = 81.94$ (1)°, $V = 1607$ (1) Å³, $Z = 4$, $D_x = 1.25$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 20.26$ cm⁻¹, $F(000) = 324$, room temperature, $R = 0.0611$, $wR = 0.0596$ for 2564 observed reflections. The two independent molecules *A* and *B* in the asymmetric unit show conformational differences. The methoxy groups are coplanar with the benzene ring and the flexible aromatic phenyl rings effectively rotated out of conjugation with the isoquinoline system, thus leading to favourable packing arrangements. Small conformational differences are observed in the piperidine rings: molecule *A* is best described as sofa or half boat; molecule *B* as half chair.

Experimental. The synthesis of the title compound has been reported previously (Mondeshka, Ivanov, Angelova, Pavlova, Uzunov, Panova, 1989). Colourless transparent crystals were obtained by

slow evaporation from methanol at room temperature. A selected crystal with dimensions $0.58 \times 0.20 \times 0.20$ mm was mounted on a Siemens AED2 automated diffractometer equipped with a monochromated Cu $K\alpha$ radiation source. Accurate unit-cell parameters were determined by a least-squares fit to setting angles of 35 strong and well centred reflections in the 2θ range 25 – 35° . 4902 unique reflections were measured by the ω - 2θ scan technique up to a maximum of 140° in 2θ , covering the hkl range $0 \leq h \leq 17$, $-17 \leq k \leq 17$ and $-23 \leq l \leq 23$. Three standard reflections monitored periodically every 120 min showed no significant intensity variation. Data reduction included correction for background, Lorentz, polarization and absorption effects. The applied min. and max. transmission factors were 0.943 and 0.967, respectively. 2564 reflections were considered observed with $I \geq 3\sigma(I)$. The structure was solved by direct methods with *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and subjected to blocked full-matrix least-