

5,6,7,8-Tetrahydroquinolin-8-one

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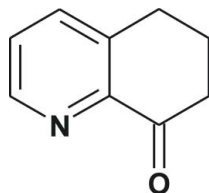
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.050; wR factor = 0.153; data-to-parameter ratio = 7.6.

In the quinoline fused-ring system of the title compound, $\text{C}_9\text{H}_9\text{NO}$, the pyridine ring is planar to within 0.011 (3) Å, while the partially saturated cyclohexene ring adopts a sofa conformation with an asymmetry parameter $\Delta C_s(\text{C}6) = 1.5$ (4)°. There are no classical hydrogen bonds in the crystal structure. Molecules form molecular layers parallel to (100) with a distance between the layers of $a/2 = 3.468$ Å.

Related literature

The title compound is an intermediate for the synthesis of polyheterocycles giving photoluminescence (Kelly & Lebedev, 2002) and a key substrate to synthesis of its 8-amino substituted derivatives with pharmacological activity (e.g. Gudmundsson *et al.*, 2009). For our ongoing study on the synthesis and structure of condensed pyridine and quinoline derivatives, see: Lipińska (2005); Karczmarzyk *et al.* (2010). For the synthesis, see: Kelly & Lebedev (2002). For a related structure, see: OXHYQU (Cygler *et al.*, 1981). For structure interpretation tools, see: Bruno *et al.* (2002); Spek (2009). For a description of the Cambridge Structural Database, see: Allen (2002). For bond-length data, see: Allen *et al.* (1987). For asymmetry parameters, see: Duax & Norton (1975).



Experimental

Crystal data

$\text{C}_9\text{H}_9\text{NO}$	$V = 756.11$ (4) Å ³
$M_r = 147.17$	$Z = 4$
Orthorhombic, $P2_12_12_1$	Cu $K\alpha$ radiation
$a = 6.9393$ (2) Å	$\mu = 0.68$ mm ⁻¹
$b = 8.0885$ (3) Å	$T = 293$ K
$c = 13.4710$ (4) Å	$0.60 \times 0.16 \times 0.15$ mm

Data collection

Bruker SMART APEXII CCD diffractometer	5358 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 2005)	761 independent reflections
$T_{\min} = 0.878$, $T_{\max} = 1.000$	734 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.022$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.050$	100 parameters
$wR(F^2) = 0.153$	H-atom parameters constrained
$S = 1.14$	$\Delta\rho_{\text{max}} = 0.28$ e Å ⁻³
761 reflections	$\Delta\rho_{\text{min}} = -0.18$ e Å ⁻³

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: SHELXL97 and WinGX (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JH2281).

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supporting information

Acta Cryst. (2011). E67, o1365 [doi:10.1107/S1600536811016175]

5,6,7,8-Tetrahydroquinolin-8-one

Teodozja M. Lipińska, Zbigniew Karczmarzyk, Waldemar Wysocki, Ewa Gruba and Andrzej Fruziński

S1. Comment

6,7-Didro-5*H*-quinolin-8-one, (I), is an important intermediate for the synthesis of polyheterocycles giving photoluminescence (Kelly & Lebedev, 2002) and a key substrate to synthesis of its 8-amino substituted derivatives with pharmacological activity (*e.g.* Gudmundsson *et al.*, 2009). As a part of our ongoing study on the synthesis and structure of condensed pyridine and quinoline derivatives (Lipińska, 2005; Karczmarzyk *et al.*, 2010) we report herein the X-ray structure of the title compound. This compound is well-known on organic chemistry but its crystal structure is not current in the Cambridge Structural Database (November 2010 Release; Allen, 2002; Bruno *et al.*, 2002).

The bond lengths and angles for (I) are within expected ranges (Allen *et al.*, 1987) and are comparable to the corresponding values observed in related structure of 8-oxo-2-phenyl-5,6,7,8-tetrahydroquinoline (OXHYQU; CSD, November 2010 Release). In the two-ring fused system the aromatic pyridine ring is planar within 0.011 (3) Å, while the partially saturated cyclohexene ring adopts a sofa conformation with asymmetry parameter $\Delta C_5(C6) = 1.5 (4)^\circ$ (Duax & Norton, 1975).

There are no classical hydrogen bonds in the crystal structure of (I). The nearly planar molecules form molecular layers parallel to (100) crystallographic plane (Fig. 2) imposing in the unit cell the pseudo-mirror plane passing through N, O, C(*sp*²) and C5(*sp*³) atoms (higher pseudosymmetry *Pnma* space group). The distance between neighbouring planes of $a/2 = 3.468$ Å is comparable to a van der Waals distance of about 3.5 Å for the π - π interacting aromatic skeletons of pyridine rings.

S2. Experimental

The titled compound was obtained by ozonolysis of 8-benzylidene-5,6,7,8-tetrahydroquinoline according to the method described by Kelly & Lebedev (2002). Crystals suitable for X-ray diffraction analysis were grown by slow evaporation of a ethyl acetate/hexane (1:1) solution.

S3. Refinement

The H atoms were positioned geometrically and treated as riding on their C atoms, with C—H distances of 0.93 (aromatic) and 0.97 Å (CH₂), and were refined with $U_{\text{iso}}(\text{H})$ values of $1.5U_{\text{eq}}(\text{C})$. The Flack parameter originally was refined to 0.4 (6), which is essentially indeterminate. For this reason, the Friedel equivalents were merged using MERG4 in *SHELXL97* (Sheldrick, 2008) and the absolute structure was arbitrarily assigned. The *PLATON* symmetry check (Spek, 2009) reveals the presence of pseudosymmetry in the structure suggesting the higher symmetry space group *Pnma* in the unit cell with the cell constants of $a' = b$, $b' = a$ and $c' = c$ and origin shifted to: -0.2500, 0.2577, 0.0000. This pseudosymmetry forces the molecule to locate on the crystallographic mirror plane passing through N, O, all C(*sp*²) and C5(*sp*³) atoms and C6 atom to be disordered over two positions above and below the mirror plane. The attempt to refine

the structure in the $Pnma$ space group resulted in a more disordered model with high R and wR values of 0.199 and 0.527, respectively.

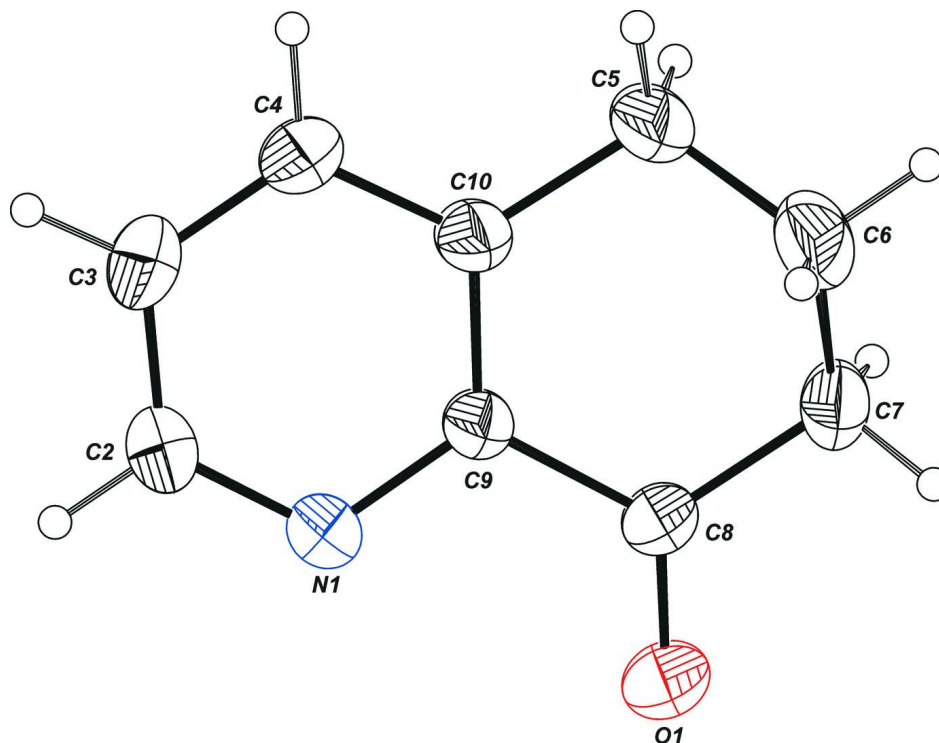


Figure 1

The molecular structure of (I), with atom labels and 30% probability displacement ellipsoids for non-H atoms.

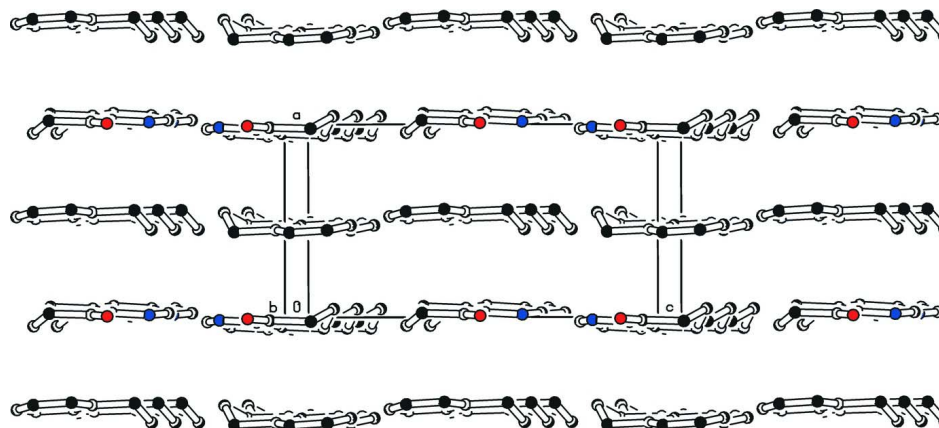


Figure 2

A view of the molecular packing in (I).

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Crystal data

C_9H_9NO

$M_r = 147.17$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 6.9393 (2) \text{ \AA}$

$b = 8.0885 (3) \text{ \AA}$

$c = 13.4710 (4) \text{ \AA}$

$V = 756.11 (4) \text{ \AA}^3$

$Z = 4$
 $F(000) = 312$
 $D_x = 1.293 \text{ Mg m}^{-3}$
 Melting point = 369–371 K
 Cu $K\alpha$ radiation, $\lambda = 1.54178 \text{ \AA}$
 Cell parameters from 40 reflections

$\theta = 7.2\text{--}34.8^\circ$
 $\mu = 0.68 \text{ mm}^{-1}$
 $T = 293 \text{ K}$
 Needle, colourless
 $0.60 \times 0.16 \times 0.15 \text{ mm}$

Data collection

Bruker SMART APEXII CCD diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 2005)
 $T_{\min} = 0.878$, $T_{\max} = 1.000$

5358 measured reflections
 761 independent reflections
 734 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.022$
 $\theta_{\max} = 65.0^\circ$, $\theta_{\min} = 6.4^\circ$
 $h = -8 \rightarrow 8$
 $k = -9 \rightarrow 7$
 $l = -15 \rightarrow 15$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.153$
 $S = 1.14$
 761 reflections
 100 parameters
 0 restraints
 Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
 Hydrogen site location: inferred from neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.1138P)^2 + 0.0433P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.28 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.18 \text{ e \AA}^{-3}$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2 , conventional R-factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F , and R-factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.0077 (5)	0.3562 (2)	0.54598 (13)	0.0827 (8)
N1	0.0144 (4)	0.0574 (3)	0.64019 (14)	0.0617 (6)
C2	0.0232 (4)	-0.0911 (4)	0.68161 (18)	0.0673 (7)
H2	0.0116	-0.0986	0.7502	0.101*
C3	0.0486 (5)	-0.2350 (4)	0.6282 (2)	0.0763 (9)
H3	0.0575	-0.3364	0.6604	0.114*
C4	0.0606 (6)	-0.2257 (3)	0.5270 (2)	0.0784 (10)
H4	0.0776	-0.3212	0.4896	0.118*
C5	0.0569 (7)	-0.0575 (4)	0.36824 (19)	0.0850 (12)
H51	0.1908	-0.0533	0.3478	0.128*
H52	-0.0010	-0.1544	0.3381	0.128*

C6	-0.0431 (6)	0.0917 (4)	0.3321 (2)	0.0899 (11)
H61	-0.1807	0.0782	0.3419	0.135*
H62	-0.0203	0.1031	0.2613	0.135*
C7	0.0218 (5)	0.2459 (3)	0.38350 (19)	0.0685 (8)
H71	-0.0596	0.3370	0.3626	0.103*
H72	0.1526	0.2708	0.3630	0.103*
C8	0.0157 (4)	0.2338 (3)	0.49507 (19)	0.0547 (6)
C9	0.0250 (3)	0.0646 (3)	0.54022 (17)	0.0498 (6)
C10	0.0472 (4)	-0.0740 (3)	0.48064 (18)	0.0593 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.1330 (18)	0.0423 (10)	0.0727 (12)	-0.0004 (12)	0.0010 (14)	-0.0078 (7)
N1	0.0823 (14)	0.0571 (12)	0.0455 (10)	0.0067 (12)	0.0018 (10)	-0.0021 (8)
C2	0.0834 (17)	0.0693 (15)	0.0491 (12)	0.0051 (16)	0.0046 (12)	0.0103 (11)
C3	0.099 (2)	0.0553 (16)	0.0748 (16)	0.0030 (15)	0.0054 (16)	0.0198 (12)
C4	0.121 (3)	0.0434 (15)	0.0710 (17)	0.0049 (16)	0.0102 (17)	-0.0019 (11)
C5	0.146 (3)	0.0603 (17)	0.0493 (14)	-0.0020 (19)	0.0052 (16)	-0.0100 (11)
C6	0.141 (3)	0.080 (2)	0.0489 (13)	-0.011 (2)	-0.0132 (16)	0.0019 (13)
C7	0.0942 (18)	0.0564 (14)	0.0549 (13)	-0.0025 (14)	-0.0030 (14)	0.0134 (11)
C8	0.0668 (13)	0.0410 (12)	0.0563 (12)	-0.0016 (11)	0.0007 (12)	-0.0020 (9)
C9	0.0616 (12)	0.0445 (12)	0.0434 (10)	-0.0013 (11)	0.0012 (10)	-0.0015 (8)
C10	0.0805 (16)	0.0462 (13)	0.0512 (13)	-0.0051 (13)	0.0066 (11)	-0.0042 (10)

Geometric parameters (Å, °)

O1—C8	1.205 (3)	C5—H51	0.9700
N1—C2	1.326 (3)	C5—H52	0.9700
N1—C9	1.350 (3)	C6—C7	1.497 (4)
C2—C3	1.380 (4)	C6—H61	0.9700
C2—H2	0.9300	C6—H62	0.9700
C3—C4	1.368 (4)	C7—C8	1.507 (3)
C3—H3	0.9300	C7—H71	0.9700
C4—C10	1.380 (4)	C7—H72	0.9700
C4—H4	0.9300	C8—C9	1.499 (3)
C5—C6	1.475 (5)	C9—C10	1.387 (3)
C5—C10	1.521 (3)		
C2—N1—C9	117.2 (2)	C5—C6—H62	109.0
N1—C2—C3	123.4 (2)	C7—C6—H62	109.0
N1—C2—H2	118.3	H61—C6—H62	107.8
C3—C2—H2	118.3	C6—C7—C8	113.5 (2)
C4—C3—C2	118.7 (2)	C6—C7—H71	108.9
C4—C3—H3	120.6	C8—C7—H71	108.9
C2—C3—H3	120.6	C6—C7—H72	108.9
C3—C4—C10	119.7 (2)	C8—C7—H72	108.9
C3—C4—H4	120.1	H71—C7—H72	107.7

C10—C4—H4	120.1	O1—C8—C9	121.4 (2)
C6—C5—C10	112.3 (3)	O1—C8—C7	121.0 (2)
C6—C5—H51	109.1	C9—C8—C7	117.57 (19)
C10—C5—H51	109.1	N1—C9—C10	123.3 (2)
C6—C5—H52	109.1	N1—C9—C8	116.21 (19)
C10—C5—H52	109.1	C10—C9—C8	120.5 (2)
H51—C5—H52	107.9	C4—C10—C9	117.7 (2)
C5—C6—C7	112.8 (3)	C4—C10—C5	121.7 (2)
C5—C6—H61	109.0	C9—C10—C5	120.7 (2)
C7—C6—H61	109.0		
C9—N1—C2—C3	2.2 (4)	O1—C8—C9—C10	-175.6 (3)
N1—C2—C3—C4	-1.8 (5)	C7—C8—C9—C10	2.9 (4)
C2—C3—C4—C10	0.1 (6)	C3—C4—C10—C9	1.0 (5)
C10—C5—C6—C7	52.3 (4)	C3—C4—C10—C5	-179.1 (4)
C5—C6—C7—C8	-51.6 (4)	N1—C9—C10—C4	-0.5 (4)
C6—C7—C8—O1	-158.0 (3)	C8—C9—C10—C4	178.1 (3)
C6—C7—C8—C9	23.5 (4)	N1—C9—C10—C5	179.6 (3)
C2—N1—C9—C10	-1.0 (4)	C8—C9—C10—C5	-1.8 (4)
C2—N1—C9—C8	-179.7 (2)	C6—C5—C10—C4	154.3 (3)
O1—C8—C9—N1	3.1 (4)	C6—C5—C10—C9	-25.9 (5)
C7—C8—C9—N1	-178.4 (3)		
