Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

8-Nitroquinoline

Liang Xu, Bao-Li Xu, Shu-Jun Lu, Bing Wang and Ting-Guo Kang*

Liaoning University of Traditional Chinese Medicine, Dalian 116600, People's Republic of China
Correspondence e-mail: lnzyxuliang@eyou.com

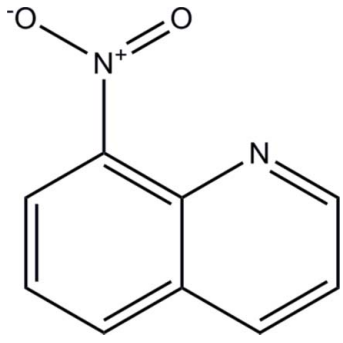
Received 14 March 2011; accepted 17 March 2011

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.147; data-to-parameter ratio = 19.2.

The molecule of the title compound, $C_9H_6N_2O_2$, is almost planar, with a dihedral angle of 3.0 (9)° between the pyridine and benzene rings.

Related literature

For the first synthesis of 8-nitroquinoline, see: Königs (1879). The crystal studied was synthesised according to the method of Yale & Bernstein (1948). For the pharmacological activity of quinoline derivatives, see: Franck *et al.* (2004); Zouhiri *et al.* (2005). For standard bond lengths, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_9H_6N_2O_2$
M_r = 174.16
Monoclinic, $P2_1/c$
a = 7.2421 (11) Å
b = 16.688 (3) Å
c = 7.2089 (11) Å
β = 114.086 (4)°
V = 795.4 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 296 K
0.40 × 0.32 × 0.25 mm

Data collection

Bruker SMART CCD area-detector diffractometer
10084 measured reflections
2287 independent reflections
1827 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.147
S = 1.03
2287 reflections
119 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The authors thank Liaoning University of Traditional Chinese Medicine for supporting this study (No. YXRC0920).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG5010).

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supporting information

Acta Cryst. (2011). E**67**, o957 [doi:10.1107/S1600536811010014]

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S1. Comment

8-Nitroquinoline was first synthesized in 1879 (Königs 1879) and in recent years the quinoline and quinoline derivatives have been found to possess a broad spectrum of pharmacological ativity (Franck *et al.*., 2004, Zouhiri *et al.*, 2005). However, the crytal structure of 8-Nitroquinoline has not been reported so far. Knowledge of the crystal structure of 8-Nitroquinoline gives us not only information about nuclearity of the complex molecule, but is important in understanding the behaviour of this compounds in the vapour phase, and the mechanisms of sublimation and decomposition. Therefore, we have synthesized the title compound, (I), and report its crystal structure here (Fig. 1).

The bond lengths for (I) are within normal ranges (Allen *et al.*, 1987). The molecule is almost flat, with a dihedral angle of 3.0 (9)° between the pyridine and benzene rings.

S2. Experimental

The title compound, (I), was prepared according to the literature procedure of Yale & Bernstein (1948). A mixture of 6.96 g(50 mmol) of *o*-Nitrophenol and 14.2 g(100 mmol) of arsenic acid in 50 ml of 86% phosphoric acid was placed in a 250 ml, 3-necked flask fitted with a thermometer, dropping funnel, reflux condenser and magnetic stirrer. The reaction mixture was warmed to 100°C and 4.75 ml(75 mmol) of acrolein added dropwise with vigorous stirring. After all the acrolein had been added, the reaction mixture was stirred for an additional thirty minutes during which time the temperature was maintained at 100°C by warming with an oil bath. The solution was poured into 200 ml of water, treated with Hyflo Supercel and decolorizing carbon and filtered. The filtrate was made alkaline with aqueous ammonia and the precipitated product filtered. The dried solid was refluxed with 150 ml of ethyl acetate and decolorizing carbon,filtered, and concentrated until crystallization started. The product weighed 5.05 g(58% yield). Crystals suitable for X-ray data collection were obtained by recrystallization from dichloromethane–hexane (1:1 *v*/*v*).

S3. Refinement

H atoms were placed in geometrically idealized positions and allowed to ride on their parent atoms, *C*—H=0.93 for phenyl H atoms, with U_{iso}(H) = 1.2U_{eq}(C) for phenyl H.

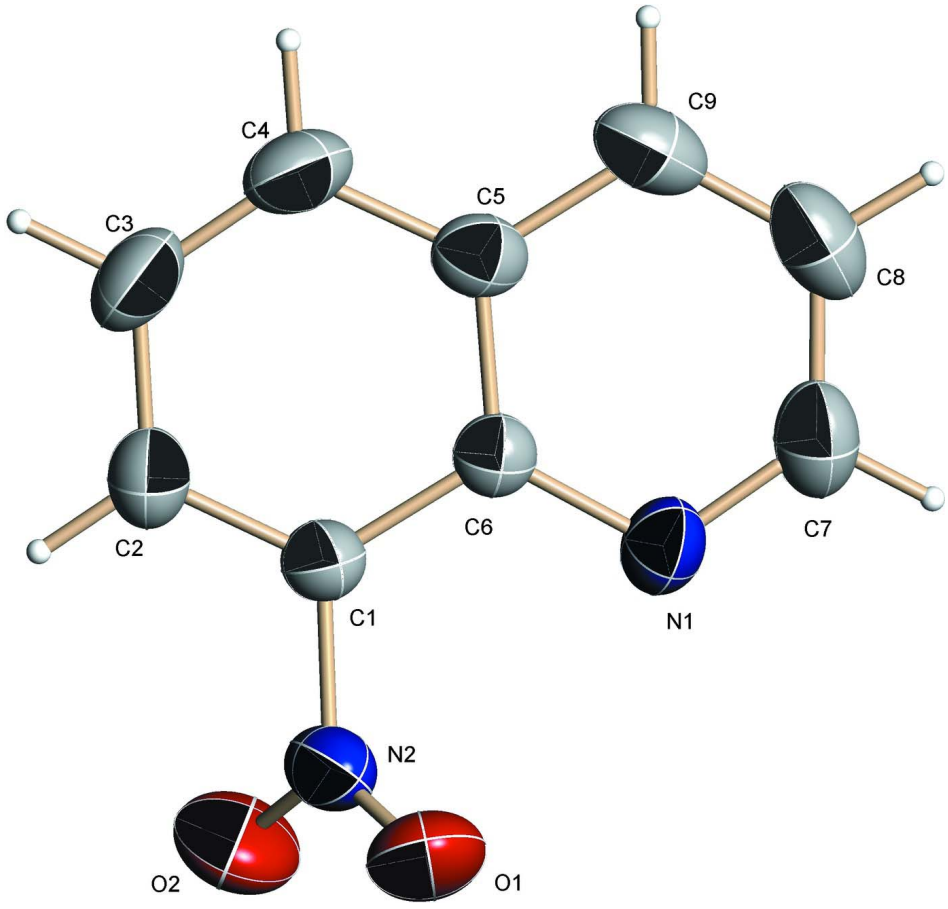

Figure 1

The molecular structure of (I) (thermal ellipsoids are shown at 30% probability levels).

8-Nitroquinoline

Crystal data

$C_9H_6N_2O_2$
$M_r = 174.16$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 7.2421$ (11) Å
$b = 16.688$ (3) Å
$c = 7.2089$ (11) Å
$\beta = 114.086$ (4)°
$V = 795.4$ (2) Å^3
$Z = 4$
$F(000) = 360$
$D_x = 1.454$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3873 reflections
$\theta = 3.1$–$30.0°$
$\mu = 0.11$ mm^{-1}
$T = 296$ K
Block, yellow
0.40 × 0.32 × 0.25 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
10084 measured reflections
2287 independent reflections
1827 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 30.0°$, $\theta_{min} = 3.1°$
$h = -9 \rightarrow 10$
$k = -23 \rightarrow 21$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.147$
$S = 1.03$
2287 reflections
119 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
$w = 1/[\sigma^2(F_o^2) + (0.0809P)^2 + 0.1053P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}
Extinction correction: *SHELXTL* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.133 (14)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 $F^2 > \sigma(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C6	0.25070 (14)	0.69710 (6)	0.26068 (13)	0.0354 (2)
C1	0.25541 (15)	0.61239 (7)	0.26308 (15)	0.0380 (2)
N1	0.41946 (14)	0.73928 (6)	0.28382 (15)	0.0454 (3)
C5	0.06270 (16)	0.73360 (7)	0.22521 (16)	0.0422 (3)
N2	0.44722 (15)	0.57111 (6)	0.30592 (16)	0.0467 (3)
C4	−0.10800 (18)	0.68516 (9)	0.1935 (2)	0.0554 (3)
H4	−0.2304	0.7092	0.1738	0.066*
C2	0.08895 (19)	0.56627 (8)	0.2262 (2)	0.0502 (3)
H2	0.0973	0.5107	0.2241	0.060*
C9	0.0561 (2)	0.81833 (8)	0.21989 (19)	0.0560 (3)
H9	−0.0634	0.8452	0.1985	0.067*
C7	0.4030 (2)	0.81781 (8)	0.27567 (19)	0.0547 (3)
H7	0.5169	0.8474	0.2905	0.066*
C3	−0.09546 (19)	0.60401 (9)	0.1914 (2)	0.0595 (4)
H3	−0.2101	0.5732	0.1668	0.071*
O1	0.59089 (14)	0.58621 (7)	0.46328 (17)	0.0659 (3)
C8	0.2259 (2)	0.86002 (8)	0.2462 (2)	0.0600 (4)
H8	0.2248	0.9157	0.2448	0.072*
O2	0.45132 (17)	0.52226 (7)	0.18178 (19)	0.0742 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C6	0.0357 (5)	0.0374 (5)	0.0308 (4)	−0.0003 (3)	0.0114 (4)	0.0008 (3)

C1	0.0358 (5)	0.0380 (5)	0.0407 (5)	0.0011 (3)	0.0162 (4)	0.0008 (4)
N1	0.0429 (5)	0.0451 (5)	0.0466 (5)	−0.0068 (4)	0.0167 (4)	0.0050 (4)
C5	0.0399 (5)	0.0452 (6)	0.0362 (5)	0.0056 (4)	0.0101 (4)	−0.0023 (4)
N2	0.0459 (5)	0.0398 (5)	0.0594 (6)	0.0052 (4)	0.0266 (4)	0.0074 (4)
C4	0.0334 (5)	0.0679 (8)	0.0594 (7)	0.0041 (5)	0.0132 (5)	−0.0067 (6)
C2	0.0494 (6)	0.0408 (6)	0.0602 (7)	−0.0091 (4)	0.0222 (5)	−0.0052 (5)
C9	0.0630 (8)	0.0481 (7)	0.0494 (6)	0.0180 (5)	0.0154 (5)	−0.0001 (5)
C7	0.0633 (8)	0.0454 (7)	0.0502 (6)	−0.0134 (5)	0.0180 (6)	0.0035 (5)
C3	0.0374 (6)	0.0663 (8)	0.0721 (8)	−0.0153 (5)	0.0196 (6)	−0.0101 (6)
O1	0.0438 (5)	0.0715 (7)	0.0714 (7)	0.0104 (4)	0.0123 (5)	0.0066 (5)
C8	0.0829 (10)	0.0361 (6)	0.0523 (7)	0.0008 (6)	0.0187 (7)	0.0013 (4)
O2	0.0783 (7)	0.0651 (7)	0.0898 (8)	0.0166 (5)	0.0451 (6)	−0.0117 (5)

Geometric parameters (Å, º)

C6—N1	1.3604 (14)	C4—C3	1.358 (2)
C6—C1	1.4140 (15)	C4—H4	0.9300
C6—C5	1.4163 (14)	C2—C3	1.4035 (19)
C1—C2	1.3619 (16)	C2—H2	0.9300
C1—N2	1.4661 (14)	C9—C8	1.357 (2)
N1—C7	1.3151 (17)	C9—H9	0.9300
C5—C9	1.4148 (18)	C7—C8	1.401 (2)
C5—C4	1.4154 (18)	C7—H7	0.9300
N2—O1	1.2122 (14)	C3—H3	0.9300
N2—O2	1.2198 (15)	C8—H8	0.9300
N1—C6—C1	119.99 (9)	C1—C2—C3	118.90 (12)
N1—C6—C5	123.30 (10)	C1—C2—H2	120.6
C1—C6—C5	116.66 (9)	C3—C2—H2	120.5
C2—C1—C6	123.20 (10)	C8—C9—C5	119.35 (12)
C2—C1—N2	117.57 (10)	C8—C9—H9	120.3
C6—C1—N2	119.23 (9)	C5—C9—H9	120.3
C7—N1—C6	116.71 (10)	N1—C7—C8	124.64 (12)
C9—C5—C4	123.30 (11)	N1—C7—H7	117.7
C9—C5—C6	116.99 (11)	C8—C7—H7	117.7
C4—C5—C6	119.70 (11)	C4—C3—C2	120.57 (11)
O1—N2—O2	123.84 (11)	C4—C3—H3	119.7
O1—N2—C1	118.49 (10)	C2—C3—H3	119.7
O2—N2—C1	117.65 (11)	C9—C8—C7	118.98 (12)
C3—C4—C5	120.90 (11)	C9—C8—H8	120.5
C3—C4—H4	119.6	C7—C8—H8	120.5
C5—C4—H4	119.6		
N1—C6—C1—C2	−175.08 (10)	C6—C1—N2—O2	−124.71 (12)
C5—C6—C1—C2	2.42 (15)	C9—C5—C4—C3	177.10 (13)
N1—C6—C1—N2	4.57 (14)	C6—C5—C4—C3	−1.69 (19)
C5—C6—C1—N2	−177.94 (9)	C6—C1—C2—C3	−2.46 (18)
C1—C6—N1—C7	178.58 (9)	N2—C1—C2—C3	177.89 (11)

C5—C6—N1—C7	1.26 (15)	C4—C5—C9—C8	−178.13 (12)
N1—C6—C5—C9	−1.77 (15)	C6—C5—C9—C8	0.68 (17)
C1—C6—C5—C9	−179.18 (9)	C6—N1—C7—C8	0.35 (18)
N1—C6—C5—C4	177.09 (10)	C5—C4—C3—C2	1.7 (2)
C1—C6—C5—C4	−0.32 (15)	C1—C2—C3—C4	0.3 (2)
C2—C1—N2—O1	−123.73 (13)	C5—C9—C8—C7	0.76 (19)
C6—C1—N2—O1	56.60 (14)	N1—C7—C8—C9	−1.4 (2)
C2—C1—N2—O2	54.95 (15)		