Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

7-Nitro-1,2,3,4-tetrahydroquinoline

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.002 Å
R factor = 0.035
wR factor = 0.071
Data-to-parameter ratio = 16.0

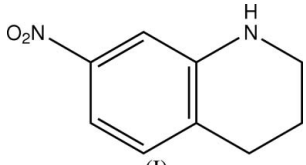
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 23 November 2005
Accepted 1 December 2005
Online 7 December 2005

In the title compound, $C_9H_{10}N_2O_2$, the tetrahydropyridine ring adopts an envelope conformation and the molecule, except for the envelope flap C and the H atoms, is essentially planar.

Comment

7-Nitro-1,2,3,4-tetrahydroquinoline, (I), is a versatile intermediate in the preparation of 7-hydroxy-1,2,3,4-tetrahydroquinoline, which is widely used in the efficient synthesis of laser dyes of the rhodamine class (Field *et al.*, 1994). Bond lengths and angles in the title compound agree with those reported for related structures (Jasinski & Woudenberg, 1993; Sivaraman *et al.*, 1996; Smith *et al.*, 2002; Sankaranarayanan *et al.*, 2003). The tetrahydropyridine ring adopts an envelope conformation, with atom C8 deviating by 0.659 (2) Å from the mean plane through the other five atoms (Fig. 1). This mean plane is almost coplanar with the benzene ring, forming a dihedral angle of 0.46 (5)°. The NO_2 group is slightly twisted out of the benzene ring plane, the angle between the nitro group and the benzene ring being 16.16 (3)°. The nitro group is, therefore, not conjugated with the benzene ring, resulting in a longer C3—N2 bond length.



(I)

Experimental

The title compound was prepared according to the method reported by Kulka & Manske (1952). To sulfuric acid (75 ml, 96.6%) cooled in a salt-ice bath was added dropwise 1,2,3,4-tetrahydroquinoline

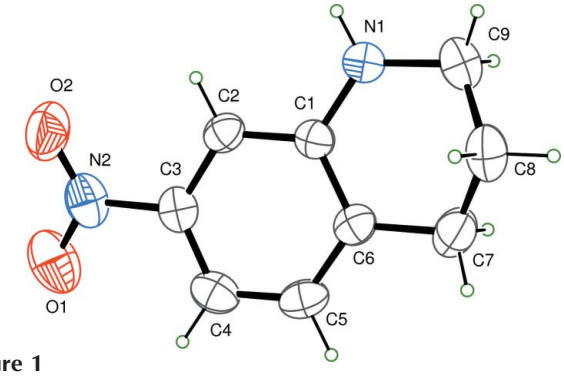


Figure 1
View of the molecular structure of (I) and the atom-numbering scheme. Displacement ellipsoids are drawn at the 40% probability level.

(25 ml, 0.2 mol). After 30 min, addition of nitric acid (9.5 ml, 0.2 mol, 90%) in sulfuric acid (40 ml) was started, keeping the temperature at 278–283 K. The reaction mixture was stirred in the ice bath for 3 h and then poured on to 1.4 kg of ice. The pH was raised to 8 with sodium carbonate (255 g). The precipitate was then collected, washed with water and recrystallized from methanol (200 ml) to give a dark-orange solid (yield 49%, 35 g). It was recrystallized from acetonitrile, giving red crystals of (I) suitable for X-ray diffraction.

Crystal data

$C_9H_{10}N_2O_2$
M_r = 178.19
Orthorhombic, *Pbca*
a = 7.985 (2) Å
b = 10.583 (4) Å
c = 19.790 (5) Å
V = 1672.4 (9) Å^3
Z = 8
D_x = 1.415 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 12005 reflections
θ = 3.1–27.5°
μ = 0.10 mm^{-1}
T = 296 (1) K
Block, red
0.28 × 0.15 × 0.14 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: none
15209 measured reflections
1906 independent reflections
1188 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.036
θ_{max} = 27.5°
$h = -9 \rightarrow 10$
$k = -13 \rightarrow 13$
$l = -25 \rightarrow 25$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.071
S = 1.00
1906 reflections
119 parameters
H-atom parameters constrained
$w = 1/[0.0001F_o^2 + \sigma(F_o^2)]/(4F_o^2)$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: Larson (1970)
Extinction coefficient: 313 (18)

Table 1
Selected bond lengths (Å).

N1—C1	1.3697 (15)	N2—C3	1.4662 (14)
N1—C9	1.4523 (15)		

All H atoms were placed in calculated positions, with C—H = 0.93–0.96 Å and N—H = 0.86 Å, and included in the refinement in the riding-model approximation, with U_{iso}(H) = 1.2U_{eq}(carrier atom).

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

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