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5-Nitro-1-(2-piperidinoethyl)-1*H*-benzimidazole

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Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.005 Å
R factor = 0.072
wR factor = 0.219
Data-to-parameter ratio = 12.9

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

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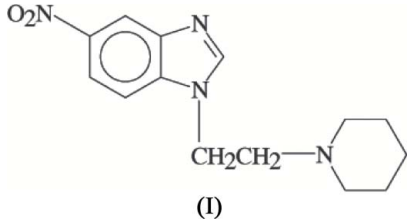
The title compound, $C_{14}H_{18}N_4O_2$, was synthesized from 5-nitrobenzimidazolium nitrate and *N*-(2-chloroethyl)piperidine hydrochloride in KOH/EtOH solution. The piperidine ring has a chair conformation. The structure is stabilized by intra- and intermolecular C—H···O hydrogen bonds.

Comment

Nitrobenzimidazole derivatives have been extensively investigated because of their biological activity. They are often used in drug design and also in non-linear optical materials, food additives and explosives (Rodembusch *et al.*, 2004; Sarlauskas *et al.*, 1997). Piperidine derivatives also exhibit versatile pharmacological activity (Mete *et al.*, 1999; Özmen *et al.*, 1999). Therefore, it seemed to be of interest to prepare compounds that incorporate these two heterocyclic groups and compare the results obtained with those for related nitrobenzimidazole derivatives (Akkurt *et al.*, 2004; Yıldırım *et al.*, 2005). Accordingly, we have synthesized and determined the crystal structure of the title compound, (I).



(I)

The molecular structure of (I) is shown in Fig. 1. All bond lengths and angles are in normal ranges and are comparable with those in related compounds (Akkurt *et al.*, 2004, 2005; Yıldırım *et al.*, 2005). The piperidine ring has the usual chair conformation and the benzimidazole ring system is almost planar. The N1/O1/O2 plane is almost coplanar with the

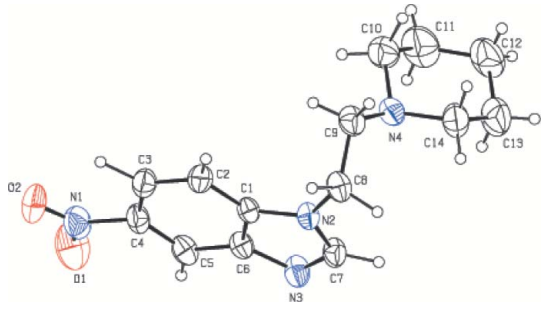


Figure 1
A plot of (I), showing the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level.

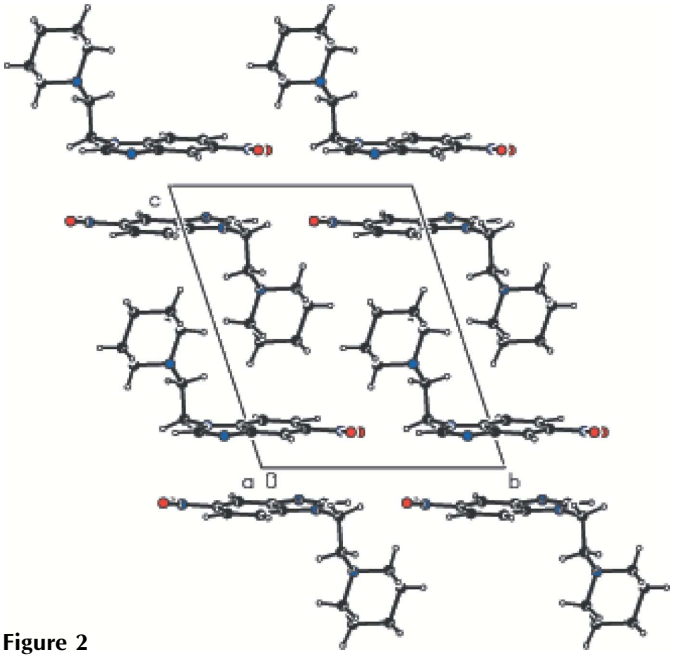


Figure 2
The packing, viewed along the [100] direction.

benzimidazole plane. Atom N1 deviates from this latter plane by 0.018 (2) Å. The torsion angles C1—N2—C8—C9 and C7—N2—C8—C9 are −75.4 (3) and 100.9 (3)°, respectively.

The crystal packing in (I) is influenced by C—H···O hydrogen bonds (Table 1). The packing of (I) is shown in Fig. 2.

Experimental

2-Chloroethylpiperidine hydrochloride (4.07 g, 22.1 mmol) was added to a solution of 5-nitrobenzimidazolium nitrate (5.00 g, 22.1 mmol) and KOH (3.75 g, 67.0 mmol) in EtOH (30 ml) and the mixture heated under reflux for 15 h. The mixture was then cooled, the precipitated potassium chloride filtered off and the solvent removed from the filtrate *in vacuo*. The residue was treated with chloroform (30 ml), and the chloroform extract was washed with NaOH solution, then water. The volatiles were then driven off *in vacuo* to give an oily residue. The residue was crystallized from EtOH (20 ml) (yield: 4.13 g, 68%; m.p.: 435–436 K). ^{1}H NMR ($CDCl_3$): δ 1.4 (*m*, ring methylene, 2H), 1.5 (*m*, ring methylene, 4H), 2.3 (*m*, ring methylene, 4H), 2.7 (*t*, CH_2CH_2 piperidine, 2H), 4.3 (*t*, CH_2CH_2 piperidine, 2H), 7.4 (*d*, Ar—H, 1H), 8.2 (*d* + *s*, Ar—H, 2H), 8.6 (*s*, 2-CH, 1H). ^{13}C NMR($CDCl_3$): δ 24.04, 25.91, 43.31, 54.74, 58.07, 109.65, 117.80, 118.57, 143.75, 147.07. Analysis calculated for $C_{14}H_{18}N_4O_2$: C 61.31, H 6.59, N 20.43%; found: C 60.72, H 6.26, N 19.95%.

Crystal data

$C_{14}H_{18}N_4O_2$
M_r = 274.32
Triclinic, $P\overline{1}$
a = 6.3202 (2) Å
b = 10.1321 (4) Å
c = 12.4362 (5) Å
α = 104.109 (3)°
β = 103.953 (2)°
γ = 102.546 (2)°
V = 716.61 (5) Å^3
Z = 2
D_x = 1.271 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2162 reflections
θ = 3.2–24.9°
μ = 0.09 mm^{-1}
T = 273 (2) K
Needle, colorless
0.76 × 0.23 × 0.19 mm

Data collection

Siemens SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
9940 measured reflections
3272 independent reflections
2158 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 27.5°
$h = -7 \rightarrow 8$
$k = -13 \rightarrow 13$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.072
$wR(F^2)$ = 0.219
S = 1.06
3272 reflections
253 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.1044P)^2 + 0.199P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—N1	1.227 (5)	N3—C6	1.388 (3)
O2—N1	1.227 (5)	N3—C7	1.303 (4)
N1—C4	1.461 (4)	N4—C9	1.454 (4)
N2—C1	1.369 (3)	N4—C10	1.452 (4)
N2—C7	1.364 (3)	N4—C14	1.455 (4)
N2—C8	1.463 (3)		
O1—N1—O2	123.6 (3)	N2—C1—C6	105.68 (18)
O1—N1—C4	118.1 (3)	N1—C4—C3	117.0 (3)
O2—N1—C4	118.3 (3)	N1—C4—C5	118.7 (3)
C1—N2—C7	106.1 (2)	N3—C6—C1	109.59 (19)
C1—N2—C8	127.3 (2)	N3—C6—C5	130.7 (2)
C7—N2—C8	126.5 (2)	N2—C7—N3	114.1 (2)
C6—N3—C7	104.5 (2)	N2—C8—C9	111.7 (2)
C9—N4—C10	112.2 (3)	N4—C9—C8	113.0 (3)
C9—N4—C14	112.2 (3)	N4—C10—C11	110.9 (4)
C10—N4—C14	109.8 (3)	N4—C14—C13	111.6 (4)
N2—C1—C2	131.9 (2)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O2	1.10 (2)	2.19 (2)	2.692 (4)	104.8 (16)
C7—H7···O2^{i}	1.00 (3)	2.43 (3)	3.425 (4)	174 (2)

Symmetry code: (i) $x, y-1, z$.

The H atoms were found in a difference Fourier map and were refined isotropically, with C—H = 0.87 (5)–1.10 (2) Å.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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