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1-(2-Piperidinoethyl)-1*H*-benzimidazole

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.041
wR factor = 0.105
Data-to-parameter ratio = 16.7

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

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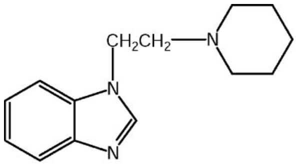
The title compound, $C_{14}H_{19}N_3$, was synthesized from benzimidazole and *N*-(2-chloroethyl)piperidine hydrochloride in KOH/EtOH solution. The benzimidazole ring is connected to the piperidine ring by an ethylene group. The piperidine ring displays a chair conformation.

Comment

Benzimidazole and many other heterocyclic compounds generally exhibit versatile pharmacological activities, such as antibacterial, antifungal, antineoplastic, antihelmintic, antiallergic, local analgesic, antihistaminic, vasodilator, hypotensive, spasmolytic activities and antiulcer activities (Küçükbay & Durmaz, 1997; Soderlind *et al.*, 1999; Küçükbay *et al.*, 2001; Carlsson *et al.*, 2002; Küçükbay *et al.*, 2003, 2004). The benzimidazole- and pyridine-containing compound omeprazole is currently the best selling anti-ulcer drug (Carlsson *et al.*, 2002). Therefore, it is of interest to prepare compounds having two important heterocyclic groups. The objective of this study therefore was to synthesize and elucidate the crystal structure of a new benzimidazole compound and compare the results obtained with those of our previous studies on related benzimidazole derivatives (Akkurt *et al.*, 2003, 2004; Öztürk *et al.*, 2001, 2003).



(I)

A view of the molecule of (I) is shown in Fig. 1. The geometric parameters are given in Table 1. In the piperidine ring, the average Csp^3—Csp^3 and Csp^3—Nsp^3 bond distances [1.496 (3) and 1.442 (4) Å, respectively] are in agreement with

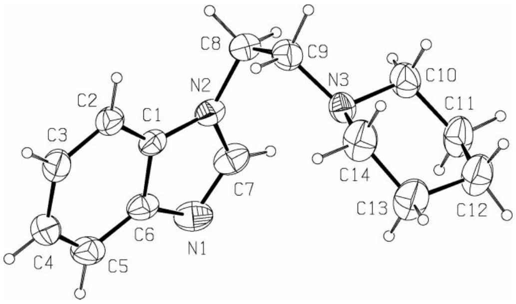


Figure 1
A view of the title compound, with the atom-numbering scheme and 20% probability displacement ellipsoids.

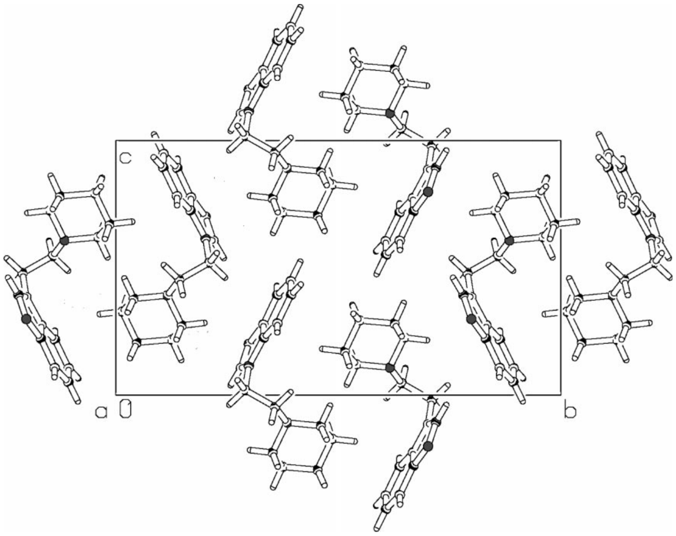


Figure 2
A view of the packing in the unit cell.

the literature data (Allen *et al.*, 1987). The values of the C—C and C—N (mean) bond lengths in the bridge between the benzimidazole and piperazine rings [1.506 (3) and 1.455 (3) Å, respectively] are comparable with the values found for related structures (Akkurt *et al.*, 2003, 2004; Öztürk *et al.*, 2003).

The benzimidazole ring of (I) is planar, with a maximum deviation from the weighted least-squares plane (C1–C7/N1/N2) of 0.013 (2) Å for atom C3 (Nardelli, 1995). The piperidine ring has a chair conformation [puckering amplitude Q_T = 0.555 (3) Å, θ = 177.3 (2)° and φ = 163 (6)° (Cremer & Pople, 1975)] for the N3/C10–C14 sequence.

Experimental

To a solution of benzimidazole (2.00 g; 16.9 mmol) and KOH (2.38 g, 42.5 mmol) in EtOH (20 ml) was added 2-chloroethylpiperidine hydrochloride (3.12 g, 16.9 mmol) and the mixture was heated under reflux for 8 h. The mixture was then cooled, after which potassium chloride was filtered off and washed with a little EtOH. The solvent was then removed from the filtrate *in vacuo*. The residue was treated with chloroform (10 ml), and the chloroform extract was washed with NaOH solution, then water. The volatiles were driven off *in vacuo* to give an oily residue. The residue was crystallized from Et_2O (yield: 2.3 g, 59%; m.p.: 337–338 K). ^{1}H NMR ($CDCl_3$): δ, 1.5 (*t*, ring methylene, 6H), 2.2 (*t*, ring methylene, 4H), 2.6 (*t*, CH_2CH_2-piperidine, 2H), 4.1 (*t*, CH_2CH_2-piperidine, 2H), 7.2–7.7 (*m*, Ar–H, 4H), 7.9 (*s*, 2-CH, 1H). $\nu_{(N=C)}$: 1620 cm^{-1}. Analysis calculated for $C_{14}H_{19}N_3$: C 73.36, H 8.30, N 18.3%; found: C 72.10, H 8.34, N 18.61%.

Crystal data

$C_{14}H_{19}N_3$
M_r = 229.32
Monoclinic, $P2_1/n$
a = 6.3741 (8) Å
b = 19.042 (2) Å
c = 11.1476 (14) Å
β = 102.178 (10)°
V = 1322.6 (3) Å^3
Z = 4
D_x = 1.152 Mg m^{-3}
Mo Kα radiation
Cell parameters from 3947 reflections
θ = 2.1–24.6°
μ = 0.07 mm^{-1}
T = 293 K
Block, yellow
0.42 × 0.33 × 0.24 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: by integration (*XRED*32; Stoe & Cie, 2002)
T_{min} = 0.963, T_{max} = 0.981
9349 measured reflections
2596 independent reflections
1132 reflections with $I > 2\sigma(I)$
R_{int} = 0.050
θ_{max} = 26.0°
$h = -7 \rightarrow 7$
$k = -23 \rightarrow 23$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.105
S = 0.80
2596 reflections
155 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0515P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.10 e Å^{-3}
$\Delta\rho_{min}$ = −0.08 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.037 (3)

Table 1
Selected geometric parameters (Å, °).

N1—C6	1.378 (3)	N2—C8	1.458 (2)
N1—C7	1.305 (3)	N3—C9	1.452 (2)
N2—C1	1.373 (3)	N3—C10	1.447 (3)
N2—C7	1.354 (2)	N3—C14	1.438 (3)
C6—N1—C7	103.69 (18)	N2—C1—C6	105.07 (16)
C1—N2—C7	106.04 (16)	N1—C6—C1	110.61 (19)
C1—N2—C8	126.78 (15)	N1—C6—C5	130.2 (2)
C7—N2—C8	126.92 (17)	N1—C7—N2	114.60 (19)
C9—N3—C10	112.68 (16)	N2—C8—C9	112.15 (16)
C9—N3—C14	113.08 (16)	N3—C9—C8	112.08 (16)
C10—N3—C14	110.24 (16)	N3—C10—C11	111.88 (19)
N2—C1—C2	132.51 (18)	N3—C14—C13	111.3 (2)
C1—N2—C8—C9	72.8 (2)	C10—N3—C9—C8	82.5 (2)
C7—N2—C8—C9	−100.4 (2)	N2—C8—C9—N3	60.4 (2)
C14—N3—C9—C8	−151.69 (17)		

The H atoms were positioned geometrically and refined using a riding model, fixing the aromatic C—H distance at 0.93 Å, and the methylene and piperidine C—H distances at 0.97 Å. U_{iso}(H) values were set at $1.2U_{eq}$ of the carrier atom.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED*32 (Stoe & Cie, 2002); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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o818 Sevim Türktekin *et al.* • $C_{14}H_{19}N_3$ *Acta Cryst.* (2004). E**60**, o817–o819

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