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Key indicators

Single-crystal X-ray study

$T = 293\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$

R factor = 0.031

w R factor = 0.075

Data-to-parameter ratio = 11.1

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

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

In the title compound, $\text{C}_{13}\text{H}_{17}\text{N}_3\text{O}$, the benzimidazole ring is connected by an ethylene group to the morpholine ring, which has a chair conformation.

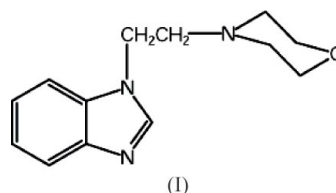
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Comment

Benzimidazole and its derivatives are important heterocyclic compounds with versatile pharmacological activities (Küçükbay *et al.*, 2001, 2003, 2004; Tavman, 2003). The benzimidazole- and pyridine-containing compound omeprazole is the best-selling antiulcer drug currently available (Carlsson *et al.*, 2002). Due to their important pharmacological activities, these compounds have received a great deal of attention in connection with their syntheses and in the elucidation of their crystal structures. The results concerning the title compound, (I), a new benzimidazole derivative, are here compared with those obtained in our previous studies of related benzimidazole derivatives (Akkurt *et al.*, 2003, 2004; Öztürk *et al.*, 2001, 2003; Türktekin *et al.*, 2004).



A view of the molecular structure of (I) is given in Fig. 1, and selected geometric parameters are listed in Table 1. In the morpholine ring, the average $\text{Csp}^3-\text{Nsp}^3$, $\text{Csp}^3-\text{Csp}^3$ and $\text{Csp}^3-\text{Osp}^3$ bond distances [1.456 (3), 1.499 (3) and 1.411 (3) Å, respectively] are comparable with the literature values (Allen *et al.*, 1987). The lengths of the $-\text{C}(\text{H}_2)-\text{C}(\text{H}_2)-$ and mean $\text{C}-\text{N}$ bonds in the ethylene group, which connects the benzimidazole ring to the morpholine ring [1.508 (3) and 1.459 (2) Å, respectively], are in agreement with

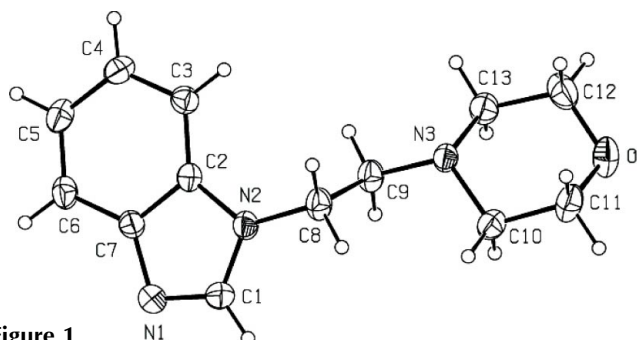


Figure 1

A view of the molecular structure of (I), with the atom-numbering scheme and 30% probability displacement ellipsoids.

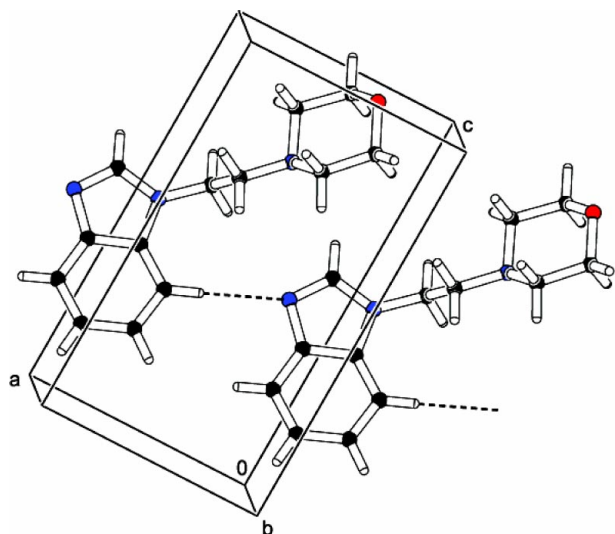


Figure 2
A view, down the *b* axis, of the crystal packing and C—H...N hydrogen-bonding (dashed lines).

the values found for related structures (Akkurt *et al.*, 2003, 2004; Öztürk *et al.*, 2003; Türktekin *et al.*, 2004).

The benzimidazole ring of (I) is planar, with maximum deviations from the least-squares plane (C1—C7/N1/N2) of 0.019 (2), −0.019 (2), 0.014 (2) and −0.018 (2) Å for atoms N1, N2, C4 and C6, respectively (Nardelli, 1995). The morpholine ring has a chair conformation [puckering amplitude $Q_T = 0.566$ (2) Å, $\theta = 178.8$ (2)° and $\varphi = 228$ (8)° (Cremer & Pople, 1975)].

The crystal structure of (I) is stabilized by intermolecular C—H...N hydrogen contacts (Table 2 and Fig. 2).

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 *N*-(2-chloroethyl)morpholine hydrochloride (3.20 g, 16.9 mmol). The mixture was heated under reflux for 8 h, cooled, and potassium chloride filtered off. The filtrate was washed with a little EtOH, and then the solvent was removed from the filtrate under vacuum. The residue was treated with chloroform (30 ml), and the chloroform extract was washed with NaOH solution and water. The volatiles were then driven off *in vacuo* to give an oily residue, which was crystallized from Et₂O (yield 2.4 g, 61%; m.p. 455–456 K). ¹H NMR (CDCl₃, δ , p.p.m.): 2.4 (*s*, ring methylene, 4H), 2.7 (*s*, CH₂CH₂-morpholine, 2H), 3.7 (*s*, ring methylene, 4H), 4.2 (*s*, CH₂CH₂-morpholine, 2H), 7.2–8.1 (*m*, Ar-H, 4H), 8.0 (*s*, 2-CH, 1H); ¹³C NMR (CDCl₃, δ , p.p.m.): 42.38, 53.69, 57.59, 66.85, 109.50, 115.09, 120.01, 122.42, 122.91, 133.76, 141.08. Analysis calculated for C₁₃H₁₇N₃O: C 67.53, H 7.36, N 18.18%; found: C 67.76, H 7.15, N 19.14%.

Crystal data

C ₁₃ H ₁₇ N ₃ O	$D_x = 1.243 \text{ Mg m}^{-3}$
$M_r = 231.30$	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 5862 reflections
$a = 6.3949$ (7) Å	$\theta = 2.3$ – 29.0°
$b = 8.7222$ (7) Å	$\mu = 0.08 \text{ mm}^{-1}$
$c = 11.0868$ (12) Å	$T = 293 \text{ K}$
$\beta = 92.345$ (9)°	Prism, colourless
$V = 617.88$ (11) Å ³	$0.56 \times 0.45 \times 0.29 \text{ mm}$
$Z = 2$	

Data collection

Stoe IPDS II diffractometer	$R_{\text{int}} = 0.032$
φ scans	$\theta_{\text{max}} = 29.0^\circ$
5862 measured reflections	$h = -8 \rightarrow 8$
1721 independent reflections	$k = -11 \rightarrow 11$
1282 reflections with $I > 2\sigma(I)$	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.043P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.031$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.075$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 0.89$	$\Delta\rho_{\text{max}} = 0.14 \text{ e } \text{Å}^{-3}$
1721 reflections	$\Delta\rho_{\text{min}} = -0.11 \text{ e } \text{Å}^{-3}$
155 parameters	Extinction correction: <i>SHELXL97</i>
H-atom parameters constrained	(Sheldrick, 1997)
	Extinction coefficient: 0.090 (8)

Table 1

Selected geometric parameters (Å, °).

O1—C11	1.412 (3)	N2—C2	1.384 (2)
O1—C12	1.409 (3)	N2—C8	1.459 (2)
N1—C1	1.310 (2)	N3—C9	1.459 (2)
N1—C7	1.386 (2)	N3—C10	1.456 (2)
N2—C1	1.350 (2)	N3—C13	1.456 (3)
C11—O1—C12	108.63 (16)	N2—C2—C7	105.12 (13)
C1—N1—C7	103.54 (13)	N1—C7—C2	110.55 (14)
C1—N2—C2	105.90 (13)	N1—C7—C6	130.09 (14)
C1—N2—C8	127.21 (15)	N2—C8—C9	111.14 (15)
C2—N2—C8	126.85 (13)	N3—C9—C8	112.30 (14)
C9—N3—C10	112.27 (13)	N3—C10—C11	110.35 (16)
C9—N3—C13	109.63 (14)	O1—C11—C10	111.67 (18)
C10—N3—C13	108.72 (14)	O1—C12—C13	112.03 (19)
N1—C1—N2	114.88 (16)	N3—C13—C12	110.94 (19)
N2—C2—C3	131.85 (14)		

Table 2

Hydrogen-bonding geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C3—H3...N1 ⁱ	0.93	2.58	3.471 (2)	161

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

Friedel pairs were merged and the $\Delta f''$ term was set to zero. The H atoms were positioned geometrically and refined using a riding model, fixing the aromatic C—H distances at 0.93 Å, and the methylene and morpholine C—H distances at 0.97 Å. $U_{\text{iso}}(\text{H})$ values were set at $1.2U_{\text{eq}}$ of the parent atom.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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