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1-Isopropyl-3-(2-morpholinioethyl)benzimidazolium diiodide

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.004 Å
R factor = 0.020
wR factor = 0.045
Data-to-parameter ratio = 15.5

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

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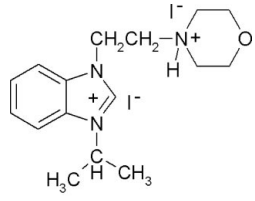
The title compound, $C_{16}H_{25}N_3O^{2+}\cdot 2I^-$, was synthesized from 1-(2-morpholinoethyl)benzimidazole and isopropyl iodide in tetrahydrofuran. In the molecule, the benzimidazole ring is connected to the morpholine ring by an ethylene group. The crystal structure has been determined at 150 K and exhibits intermolecular C—H···I interactions.

Comment

For some years we have synthesized and investigated crystal structures of many benzimidazole derivatives, which constitute an important class of heterocyclic compounds (Akkurt *et al.*, 2003, 2004, 2004*a*,*b*; Öztürk *et al.*, 2001, 2003; Türktekin *et al.*, 2004). They also show versatile pharmacological activities, such as antibacterial, antifungal, antihelmintic, antiallergic, antineoplastic, local analgesic, antihistaminic, vasodilative, hypotensive and spasmolytic activities (Easmon *et al.*, 2001; Güneş & Coşar, 1992). We have also observed that many benzimidazole derivatives and related heterocyclic compounds have shown considerable antimicrobial activities against standard strains: *Enterococcus faecalis* (ATCC 29212), *Staphylococcus aureus* (ATCC 29213), *Escherichia coli* (ATCC 25922), *Pseudomonas aeruginosa* (ATCC 27853) and yeasts *Candida albicans* and *Candida tropicalis* (Küçükbay *et al.*, 2001, 2003, 2004). The aim of this study was to synthesize and elucidate the crystal structure of the new benzimidazole title compound, (I).



(I)

Fig. 1 shows the molecular structure of (I) and the atomic numbering scheme. Selected geometric parameters are listed in Table 1. All bond distances and angles lie within the ranges of normally accepted values.

In (I), the benzimidazole ring (N2/C7–C12/N3/C13) is essentially planar, with maximum deviations of 0.009 (2) Å for N2 and −0.013 (2) Å for C13. The morpholine ring (O1/C2/C1/N1/C4/C3) has a chair conformation (Boeyens, 1978), and puckering parameters Q_T = 0.575 (2) Å, θ = 0.0 (2)° and φ = 34 (5)° (Cremer & Pople, 1975).

The crystal structure is stabilized by van der Waals interactions; close intermolecular contacts are listed in Table 2. The molecular packing and hydrogen-bonding interactions are illustrated in Fig. 2.

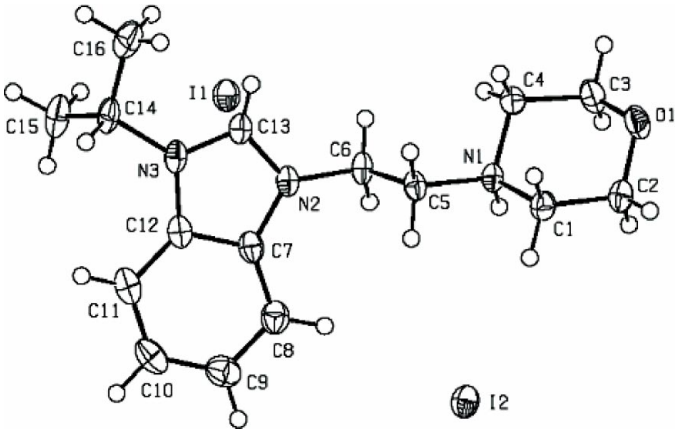


Figure 1
An *ORTEP*-3 plot (Farrugia, 1997) of the title compound, showing the atom-numbering scheme and 50% probability displacement ellipsoids.

Experimental

1-(2-Morpholinoethyl)benzimidazole was synthesized from benzimidazole and *N*-(2-chloroethyl)morpholine hydrochloride according to the literature method of Akkurt *et al.* (2004). A mixture of 1-(2-morpholinoethyl)benzimidazole (13.04 g, 56.45 mmol) and isopropyl iodide (11.28 ml, 112.90 mmol) was heated on a water bath for 3 h. The mixture was cooled to room temperature and Et_2O (20 ml) was added to precipitate the crude product. The crude product was then crystallized from EtOH/Et_2O (3:1) mixture (yield: 17.2 g, 58%; m.p.: 531–532 K). ^{1}H NMR (D_2O): δ 1.6 [*d*, $CH(CH_3)_2$, 6H], 3.4 (*t*, CH_2CH_2-morpholine, 2H), 3.8 (*t*, ring methylene, 4H), 3.9 (*t*, CH_2CH_2-morpholine, 2H), 4.8–5.1 (*m*, $CHMe_2$, 1H), 4.9 (*t*, ring methylene, 4H), 7.6–8.2 (*m*, Ar-H, 4H), 9.5 (*s*, 2-CH, 1H). Analysis calculated for $C_{16}H_{25}I_2N_3O$: C 36.29, H 4.72, N 7.94%; found: C 37.23, H 4.66, N 7.27%.

Crystal data

$C_{16}H_{25}N_3O^{2+}\cdot 2I^-$
M_r = 529.19
Monoclinic, $P2_1/c$
a = 12.078 (5) Å
b = 19.923 (5) Å
c = 8.336 (5) Å
β = 92.537 (5)°
V = 2003.9 (15) Å^3
Z = 4
D_x = 1.754 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4269 reflections
θ = 1.7–27.2°
μ = 3.14 mm^{-1}
T = 150 K
Block, colorless
0.40 × 0.36 × 0.30 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: by integration (*X-RED*32 (Stoe & Cie, 2002)
T_{min} = 0.366, T_{max} = 0.452
14805 measured reflections
4269 independent reflections
3736 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 27.1°
$h = -15 \rightarrow 15$
$k = -25 \rightarrow 25$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.045
S = 0.97
4269 reflections
276 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0287P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.77 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.00223 (16)

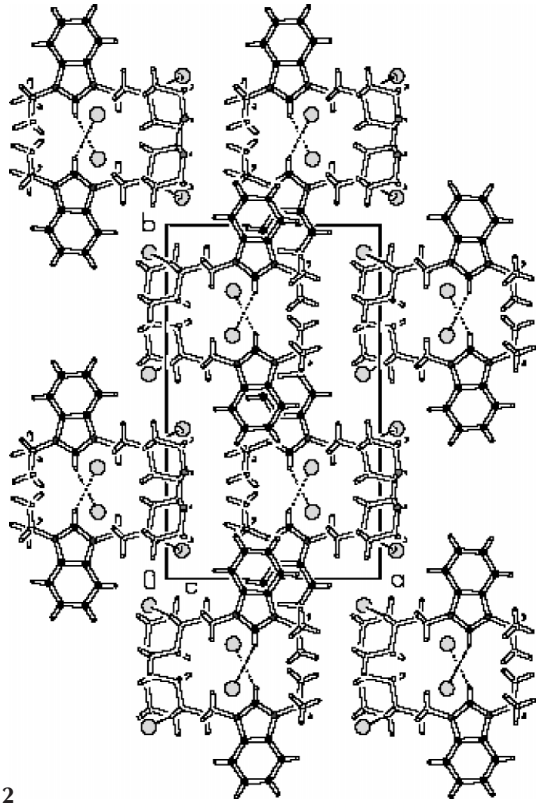


Figure 2
View of the packing and hydrogen bonds (dashed lines) of (I).

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.422 (3)	N2—C7	1.387 (3)
O1—C3	1.416 (3)	N2—C13	1.333 (3)
N1—C1	1.499 (3)	N3—C12	1.395 (3)
N1—C4	1.506 (3)	N3—C13	1.325 (3)
N1—C5	1.501 (3)	N3—C14	1.488 (3)
N2—C6	1.462 (3)		
C2—O1—C3	109.72 (17)	O1—C3—C4	111.88 (19)
C1—N1—C4	109.51 (17)	N1—C4—C3	110.04 (18)
C1—N1—C5	110.33 (16)	N1—C5—C6	111.78 (17)
C4—N1—C5	113.06 (16)	N2—C6—C5	109.71 (17)
C6—N2—C7	127.20 (18)	N2—C7—C12	106.54 (18)
C6—N2—C13	124.12 (19)	N2—C7—C8	131.1 (2)
C7—N2—C13	108.27 (17)	N3—C12—C7	106.69 (18)
C12—N3—C13	108.01 (17)	N3—C12—C11	131.6 (2)
C12—N3—C14	125.33 (17)	N2—C13—N3	110.5 (2)
C13—N3—C14	126.54 (19)	N3—C14—C15	109.30 (18)
N1—C1—C2	109.59 (18)	N3—C14—C16	110.08 (19)
O1—C2—C1	111.34 (18)		
C1—N1—C5—C6	−171.71 (18)	C13—N3—C14—C16	−19.3 (3)
C4—N1—C5—C6	65.3 (2)	C13—N3—C14—C15	105.4 (3)
C7—N2—C6—C5	−82.3 (3)	C12—N3—C14—C16	165.1 (2)
C13—N2—C6—C5	106.0 (2)	N1—C5—C6—N2	176.58 (17)
C12—N3—C14—C15	−70.3 (3)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···I2^{i}	0.95 (3)	2.56 (3)	3.475 (3)	163 (2)
C1—H1B···I2	1.00 (3)	3.00 (3)	3.926 (3)	155.2 (19)
C13—H13···I1^{ii}	0.85 (2)	2.96 (2)	3.740 (3)	153.0 (19)

Symmetry codes: (i) $2-x, -y, 1-z$; (ii) $x, \frac{1}{2}-y, \frac{1}{2}+z$.

The methyl H atoms were positioned geometrically, with C—H distances of 0.96 Å, and refined using a riding model, with U_{iso}(H) = 1.5U_{eq}(C). The other H atoms were located in a difference Fourier map and refined isotropically.

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: *SHELXS*97 (Sheldrick, 1997); 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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