Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-Ethyl-1-[2-(1-ethylmorpholinium-1-yl)ethyl]-benzimidazolium diiodide monohydrate

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.004 Å
R factor = 0.025
wR factor = 0.056
Data-to-parameter ratio = 18.0

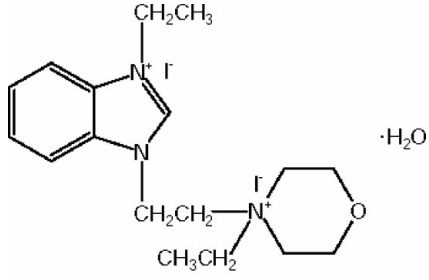
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 10 December 2004
Accepted 13 December 2004
Online 15 January 2005

The title compound, $C_{17}H_{27}N_3O^{2+}\cdot 2I^-\cdot H_2O$, was synthesized from 1-(2-morpholinoethyl)benzimidazole and ethyl iodide in dimethylformamide. In the molecule, the benzimidazole ring is connected to the morpholine ring by an ethylene group. The crystal structure is stabilized by inter- and intramolecular O—H···I, C—H···O and C—H···I interactions.

Comment

Substituted benzimidazoles and morpholines have attracted considerable interest because of their presence in a number of therapeutically and biologically active compounds. They occupy an important place among synthetic medical preparations, as a result of the wide spectrum of their pharmacological properties, such as antitumour, diuretic, fungicidal, bactericidal, anthelmintic, antiallergic, vasodilator, antihistaminic and local analgesic activities. Furthermore, both heterocycles are also used as ligands for Fisher-type carbene complexes, and these complexes act as efficient catalysts in many organic syntheses (Lupi *et al.*, 2004; Stevens & Aristizabal, 1997; Bouron *et al.*, 1999; Szafran, *et al.*, 2004; Küçükbay *et al.*, 2001, 2003, 2004). We have also synthesized and investigated the crystal structures of many benzimidazole derivatives and some morpholine derivatives, which constitute an important class of heterocyclic compounds (Akkurt *et al.*, 2003; Akkurt, Öztürk, Şireci *et al.*, 2004; Akkurt, Öztürk, Küçükbay, Orhan & Büyükgüngör, 2004*a*,*b*; Öztürk *et al.*, 2001, 2003; Türktekin, Akkurt, Orhan *et al.*, 2004; Türktekin, Akkurt, Şireci *et al.*, 2004; Akkurt, Karaca *et al.*, 2005; Akkurt, Türktekin *et al.*, 2005). We now report the synthesis and structure of the title compound, (I), a biologically interesting morpholine-substituted benzimidazole compound.



(I)

Fig. 1 shows the molecular structure of (I) and the atomic numbering scheme. Selected geometric parameters are listed in Table 1. All the geometric parameters of (I) are comparable with those obtained for previously studied related benzimidazole derivatives (Akkurt, Öztürk, Şireci *et al.*, 2004; Akkurt,

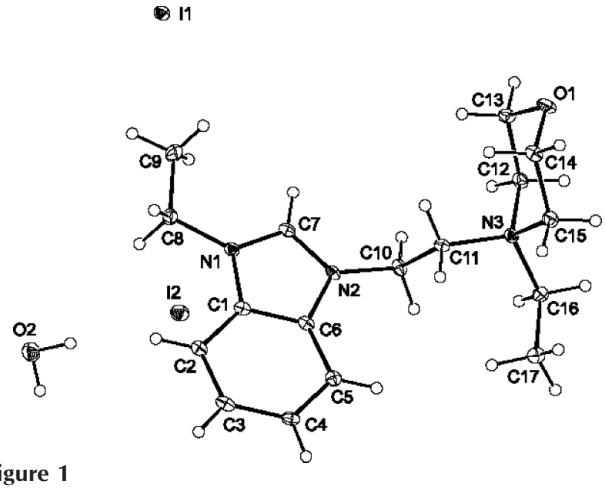


Figure 1
An *ORTEP-3* (Farrugia, 1997) plot of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

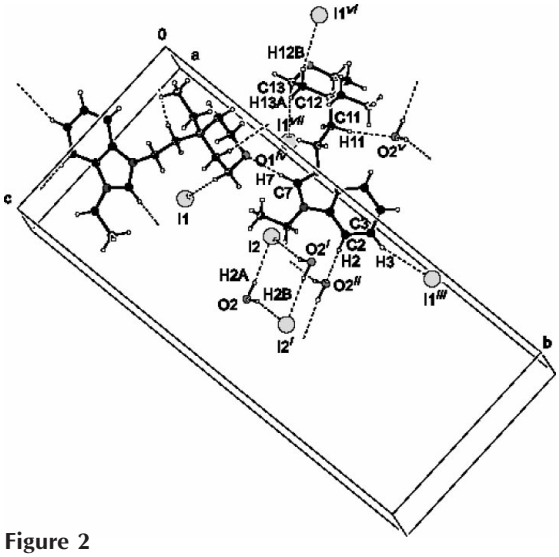


Figure 2
A view of the hydrogen bonds (dashed lines) in the crystal structure of (I). Symmetry operations correspond to those given in Table 2.

Öztürk, Küçükbay, Orhan & Büyükgüngör, 2004*b*; Akkurt, Öztürk, Küçükbay, Yılmaz & Büyükgüngör, 2004; Türktekin, Akkurt, Şireci *et al.*, 2004; Akkurt, Karaca *et al.*, 2005; Akkurt, Türktekin *et al.*, 2005).

In (I), the nine-membered benzimidazole fused ring system (N1/C1–C6/N2/C7) is essentially planar [the maximum deviations from planarity are 0.020 (2), −0.016 (3) and −0.016 (2) Å, for atoms N1, C2 and N2, respectively]. The conformation of the six-membered morpholine ring (O1/C13/C12/N3/C15/C14) is very similar to a chair form, and the puckering parameters are Q_T = 0.577 (3) Å, θ = 177.1 (3)° and φ = 158 (4)° (Cremer & Pople, 1975).

The hydrogen-bonding interactions are given in Table 2 and the crystal packing is illustrated in Fig. 2.

Experimental

1-(2-Morpholinoethyl)benzimidazole was synthesized according to the method described by Akkurt, Öztürk, Şireci *et al.* (2004). A mixture of 1-(2-morpholinoethyl)benzimidazole (2.00 g, 8.66 mmol) and ethyl iodide (1.4 ml, 17.30 mmol) in dimethyl formamide (3 ml) was heated on a water bath for 3 h. All the volatiles were then removed under vacuum. The resulting crude title compound was crystallized from EtOH/Et_2O (3:1). Yield 3.5 g, 72%. M.p. 470–471 K. ^{1}H NMR (DMSO-d_6): δ 1.36 (*t*, CH_2CH_3, 3H), 1.59 (*t*, CH_2CH_3, 3H), 3.66 (*t*, ring methylene, 4H), 3.82 (*t*, CH_2CH_2-morpholine, 2H), 4.01 (*t*, ring methylene, 4H), 4.13 (*q*, $-CH_2CH_3$, 2H), 4.56 (*q*, $-CH_2CH_3$, 2H), 5.06 (*t*, CH_2CH_2-morpholine, 2H), 7.62–8.16 (*m*, Ar—H, 4H), 9.99 (*s*, benzimidazole-C^2—H, 1H). ^{13}C NMR (DMSO-d_6): δ 7.78, 14.13, 42.96, 54.07, 54.83, 58.10, 59.99, 63.84,114.26, 114.35, 127.18, 127.30, 131.29, 131.39, 133.18. Analysis calculated for $C_{17}H_{29}I_2N_3O_2$: C 36.36, H 5.17, N 7.49%; found: C 36.25, H 5.16, N 7.38%.

Crystal data

$C_{17}H_{27}N_3O^{2+}\cdot 2I^-\cdot H_2O$
M_r = 561.23
Monoclinic, $P2_1/c$
a = 7.527 (5) Å
b = 24.889 (5) Å
c = 11.757 (5) Å
β = 106.007 (5)°
V = 2117.2 (17) Å^3
Z = 4
D_x = 1.761 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 22 695 reflections
θ = 1.6–26.0°
μ = 2.99 mm^{-1}
T = 100 K
Prism, colourless
0.45 × 0.32 × 0.17 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.347, T_{max} = 0.631
23 081 measured reflections
4039 independent reflections
3870 reflections with $I > 2\sigma(I)$
R_{int} = 0.096
θ_{max} = 26.0°
$h = -9 \rightarrow 9$
$k = -30 \rightarrow 30$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.057
S = 1.09
4039 reflections
225 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0084P)^2 + 1.9938P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.82 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C13	1.430 (4)	N2—C7	1.332 (3)
O1—C14	1.431 (3)	N2—C6	1.403 (3)
N1—C7	1.323 (3)	N3—C11	1.519 (3)
N1—C1	1.398 (3)	N3—C16	1.529 (4)
N1—C8	1.464 (4)	N3—C12	1.527 (3)
N2—C10	1.467 (4)	N3—C15	1.519 (4)
C13—O1—C14	110.1 (2)	N1—C1—C6	107.6 (2)
C1—N1—C8	124.29 (19)	N1—C1—C2	129.8 (2)
C7—N1—C8	128.03 (19)	N2—C6—C1	105.8 (2)
C1—N1—C7	107.7 (2)	N2—C6—C5	131.7 (2)
C6—N2—C7	108.3 (2)	N1—C7—N2	110.6 (2)
C6—N2—C10	127.0 (2)	N1—C8—C9	112.2 (2)
C7—N2—C10	124.6 (2)	N2—C10—C11	108.9 (2)
C11—N3—C12	112.90 (19)	N3—C11—C10	113.5 (2)
C11—N3—C15	109.0 (2)	N3—C12—C13	112.4 (2)
C12—N3—C15	106.70 (18)	O1—C13—C12	110.9 (2)
C12—N3—C16	106.1 (2)	O1—C14—C15	109.9 (2)
C15—N3—C16	109.2 (2)	N3—C15—C14	112.3 (2)
C11—N3—C16	112.66 (19)	N3—C16—C17	114.6 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···I2	0.94 (4)	2.58 (4)	3.517 (3)	175 (4)
O2—H2B···I2^{i}	0.83 (6)	2.83 (6)	3.631 (3)	162 (6)
C2—H2···O2^{ii}	0.93	2.40	3.307 (5)	166
C3—H3···I1^{iii}	0.93	3.03	3.899 (3)	157
C7—H7···O1^{iv}	0.93	2.22	3.059 (4)	149
C11—H11A···O2^{v}	0.97	2.41	3.366 (4)	168
C12—H12B···I1^{vi}	0.97	2.98	3.911 (4)	161
C13—H13A···I1^{vii}	0.97	3.01	3.949 (4)	163

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

The H atoms of the water molecule were located in a difference Fourier map and refined isotropically. The other H atoms were placed in calculated positions and treated as riding atoms (C—H = 0.93–0.97 Å), with U_{iso}(H) values of 1.2 or 1.5 times U_{eq}(parent C 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: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors acknowledge the Faculty of Arts and Sciences, Ondokuz Mayıs University, Turkey, for the use of the diffractometer (purchased under grant F.279 of the University Research Fund). HK and ÜY thank İnönü University Scientific Research Unit (BAPB-2002/06 directed project) for financial support of this study.

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