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1-Benzyl-3-[2-(1-piperidinio)ethyl]benzimidazolium dichloride monohydrate

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.031
wR factor = 0.078
Data-to-parameter ratio = 17.4

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

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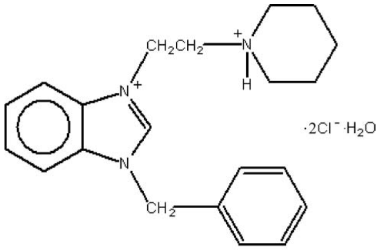
The title compound, $C_{21}H_{27}N_3^{2+}\cdot 2Cl^-\cdot H_2O$, was synthesized from 1-benzylbenzimidazole and 2-chloroethylpiperidine hydrochloride in dimethylformamide. In the cation, the benzimidazole ring is connected to the piperidine ring by an ethylene group. The crystal structure is stabilized by O—H···Cl, N—H···Cl, C—H···O and C—H···Cl hydrogen-bonding interactions.

Comment

Heterocyclic compounds generally exhibit versatile pharmacological activities, such as antitumour, diuretic, fungicidal, bactericidal, antihelmintic, antiallergic, vasodilator, antihistaminic and local analgesic. We have reported the synthesis and antimicrobial activities of many benzimidazole derivatives (Küçükbay *et al.*, 2003, 2004) and elucidated the crystal structures of some benzimidazole derivatives having piperidine or morpholine groups (Türktekin *et al.*, 2004; Akkurt, Türktekin *et al.*, 2004; Akkurt, Öztürk *et al.*, 2004; Akkurt *et al.*, 2005). We now report the synthesis and crystal structure of a biologically interesting piperidine-substituted benzimidazole compound, (I).



(I)

The molecular structure of (I) is illustrated in Fig. 1, and selected bond distances and angles are given in Table 1. All the geometric parameters of (I) agree with the results obtained in our previous studies of related heterocycles (Akkurt *et al.*, 2005; Türktekin *et al.*, 2004). The benzimidazole ring is essentially planar, with maximum deviations of 0.012 (1) Å for atom N1 and −0.012 (1) Å for atom C6. The dihedral angle between the planes of the phenyl and benzimidazole ring systems is 72.83 (6)°. The piperidine ring has a chair conformation [the puckering parameters (Cremer & Pople, 1975) are Q_T = 0.5701 (18) Å, θ = 177.12 (18)° and φ = 195 (4)°].

The crystal structure of (I) is stabilized by O—H···Cl, N—H···Cl, C—H···O and C—H···Cl hydrogen-bonding interactions. Details are given in Table 2 and the hydrogen-bonding involving the Cl1 anion and the water molecule of crystallization, O1, is illustrated in Fig. 2.

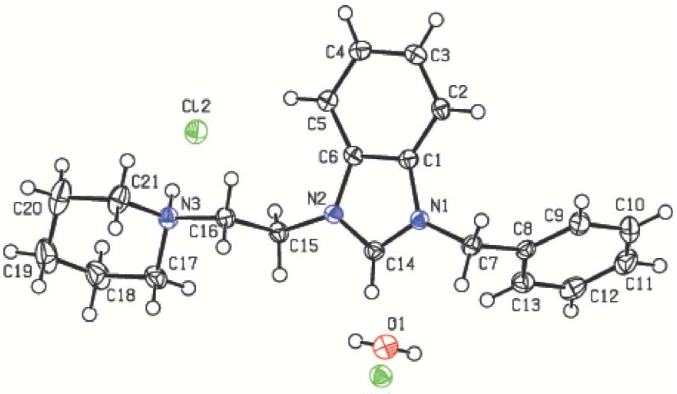


Figure 1
An *ORTEP-3* (Farrugia, 1997) view of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

Experimental

Compound (I) was synthesized by heating, on a water bath for 3 h, a mixture of 1-benzylbenzimidazole (2.00 g, 9.6 mmol) and 2-chloroethylpiperidine hydrochloride (1.8 g, 9.6 mmol) in dimethylformamide (10 ml). The volatiles were then removed under vacuum and the crude solid obtained was crystallized from an EtOH/Et$_2$O (3:1) mixture. Colourless plate-like crystals were obtained (yield 3 g, 79%; m.p. 469–470 K). ^{1}H NMR (D$_2$O): δ 1.4–1.8 (*q*, piperidine, 6H), 3.2 (*q*, piperidine, 4H), 3.6 (*t*, CH$_2$CH$_2$-piperidine, 2H), 4.8 (*t*, CH$_2$CH$_2$-piperidine, 2H), 4.01 (*t*, ring methylene, 4H), 4.13 (*q*, –CH$_2$CH$_3$, 2H), 4.56 (*q*, –CH$_2$CH$_3$, 2H), 5.5 (*s*, CH$_2$—Ph, 2H), 7.3 (*s*, Ar—H, 4H), 9.3 (*s*, benzimidazole-C2—H, 1H). Analysis calculated for $C_{21}H_{29}Cl_2N_3O$: C 61.46, H 7.07, N 10.24%; found: C 63.02, H 6.71, N 10.70%.

Crystal data

$C_{21}H_{27}N_3^{2+}\cdot 2Cl^-\cdot H_2O$
$M_r = 410.37$
Monoclinic, $P2_1/n$
$a = 17.1422$ (12) Å
$b = 6.9643$ (3) Å
$c = 18.2627$ (13) Å
$\beta = 106.961$ (5)°
$V = 2085.4$ (2) Å^3
$Z = 4$
$D_x = 1.307$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 17638 reflections
$\theta = 1.9$–27.2°
$\mu = 0.33$ mm^{-1}
$T = 100$ K
Plate, colourless
0.78 × 0.49 × 0.18 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
$T_{\min} = 0.784$, $T_{\max} = 0.943$
17638 measured reflections
4535 independent reflections
3875 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.032$
$\theta_{\max} = 27.0°$
$h = -21 \rightarrow 21$
$k = -8 \rightarrow 8$
$l = -23 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.078$
$S = 1.04$
4535 reflections
260 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0383P)^2 + 0.7441P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.43$ e Å^{-3}
$\Delta\rho_{\min} = -0.18$ e Å^{-3}

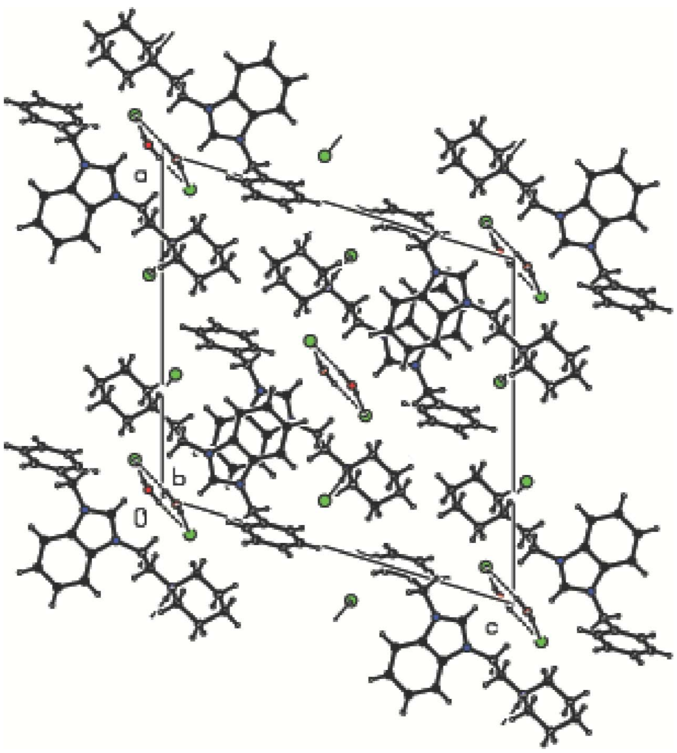


Figure 2
The crystal packing of (I), viewed along the *b* axis. Dashed lines indicate O—H···Cl hydrogen-bonding contacts (details are give in Table 2).

Table 1
Selected geometric parameters (Å, °).

N1—C1	1.3947 (16)	N2—C15	1.4637 (16)
N1—C14	1.3279 (16)	N3—C17	1.5004 (18)
N1—C7	1.4747 (18)	N3—C16	1.4834 (17)
N2—C14	1.3342 (18)	N3—C21	1.4974 (19)
N2—C6	1.3973 (16)		
C1—N1—C7	125.86 (11)	N1—C1—C6	106.61 (10)
C1—N1—C14	108.36 (11)	N2—C6—C1	106.42 (11)
C7—N1—C14	125.76 (11)	N2—C6—C5	131.60 (11)
C6—N2—C14	108.17 (10)	N1—C7—C8	112.37 (11)
C6—N2—C15	126.09 (11)	N1—C14—N2	110.43 (11)
C14—N2—C15	125.74 (11)	N2—C15—C16	109.17 (10)
C16—N3—C21	109.32 (11)	N3—C16—C15	112.05 (11)
C17—N3—C21	111.10 (11)	N3—C17—C18	110.35 (13)
C16—N3—C17	112.11 (11)	N3—C21—C20	110.29 (13)
N1—C1—C2	131.10 (12)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···Cl2	0.92 (2)	2.10 (2)	3.0066 (13)	171 (2)
O1—H22···Cl1$^{\rm i}$	0.84 (2)	2.35 (2)	3.1919 (12)	176 (2)
O1—H23···Cl1$^{\rm ii}$	0.85 (2)	2.33 (2)	3.1722 (12)	175 (2)
C2—H2···Cl2$^{\rm iii}$	0.93	2.81	3.6549 (14)	152
C7—H7A···Cl1	0.97	2.80	3.6930 (15)	153
C9—H9···Cl2$^{\rm iii}$	0.93	2.75	3.6243 (15)	158
C15—H15A···O1$^{\rm ii}$	0.97	2.57	3.2734 (18)	129
C15—H15B···Cl2	0.97	2.79	3.5749 (15)	139

Symmetry codes: (i) $x, y-1, z$; (ii) $-x+1, -y+1, -z+1$; (iii) $-x+\frac{3}{2}, +y+\frac{1}{2}, -z+\frac{3}{2}$.

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All H atoms were found in difference Fourier maps. The water molecule H atoms and the N-bound H atom were refined isotropically. The other H atoms were refined with a riding model, with C—H = 0.93–0.97 Å, and with $U_{\rm iso}$ constrained to be $1.2U_{\rm eq}$ of the carrier 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 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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