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1-(4-Chlorobenzyl)-3-[2-(pyrrolidinium-1-yl)-ethyl]benzimidazolium dichloride monohydrate

Mehmet Akkurt,[a]* Şerife Pınar,[a] Ülkü Yılmaz,[b] Hasan Küçükbay[b] and Orhan Büyükgüngör[c]

[a]Department of Physics, Faculty of Arts and Sciences, Erciyes University, 38039 Kayseri, Turkey, [b]Department of Chemistry, Faculty of Arts and Sciences, İnönü University, 44280 Malatya, Turkey, and [c]Department of Physics, Faculty of Arts and Sciences, Ondokuz Mayıs University, 55139 Samsun, Turkey

Correspondence e-mail: akkurt@erciyes.edu.tr

Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.003 Å
R factor = 0.038
wR factor = 0.105
Data-to-parameter ratio = 18.3

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

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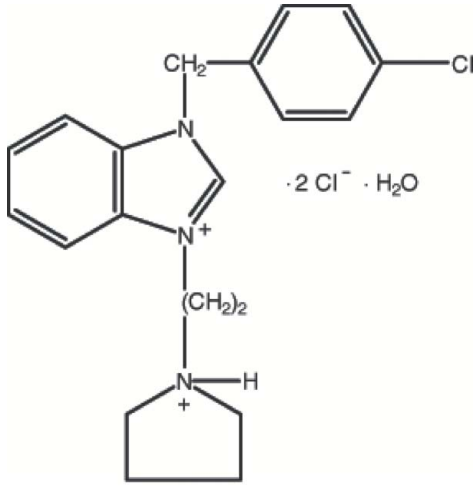
The title compound, $C_{20}H_{26}Cl_3N_3O$, was synthesized from 1-(4-chlorobenzyl)benzimidazole and 1-(2-chloroethyl)pyrrolidine hydrochloride in dimethylformamide. The dihedral angle between the benzene ring and the benzimidazole ring system is 68.33 (8)°. The five-membered pyrrolidine ring has a slightly distorted envelope conformation. The crystal structure is stabilized by O—H···Cl, C—H···Cl, N—H···Cl and C—H···O hydrogen bonds.

Comment

Many clinically useful drugs contain a heterocyclic unit such as benzimidazole, pyridine, piperidine, morpholine, pyrrole or pyrrolidine. These compounds exhibit versatile pharmacological activities such as antitumor, diuretic, fungicidal, bactericidal, anthelmintic, anti-allergic, vasodilator, antihistaminic and local analgesic. We have already reported the syntheses and antimicrobial activities of some heterocyclic compounds (Küçükbay *et al.*, 1995, 2003, 2004) and determined the crystal structures of some (Türktekin *et al.*, 2004; Akkurt, Öztürk *et al.*, 2004; Akkurt, Türktekin *et al.*, 2004, 2005, 2006; Akkurt, Yıldırım *et al.*, 2005, 2006; Karaca *et al.*, 2005). We report here the crystal structure of a new heterocyclic compound (I), containing two heterocyclic units, benzimidazole and pyrrolidine.



(I)

The asymmetric unit of (I) is shown in Fig. 1. All bond distances and angles of (I) are comparable to those obtained in our previous studies of related heterocycles (Pınar *et al.*, 2006; Akkurt, Yıldırım *et al.*, 2006; Akkurt, Türktekin *et al.*, 2006). The plane of the C9–C14 benzene ring makes a dihedral

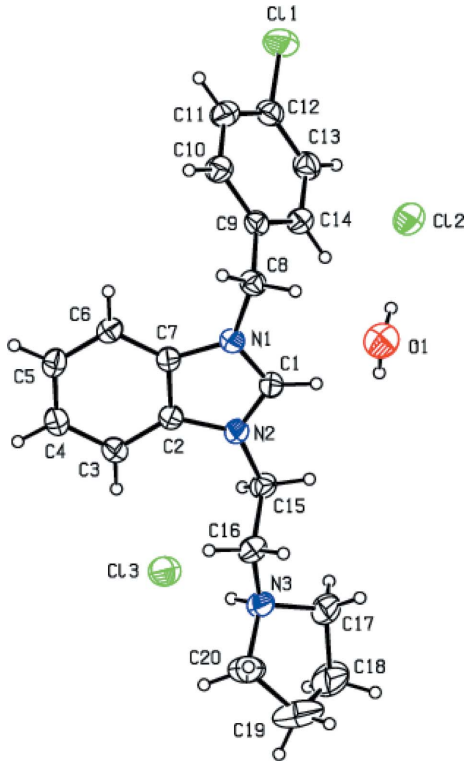


Figure 1
The asymmetric unit of (I), with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level.

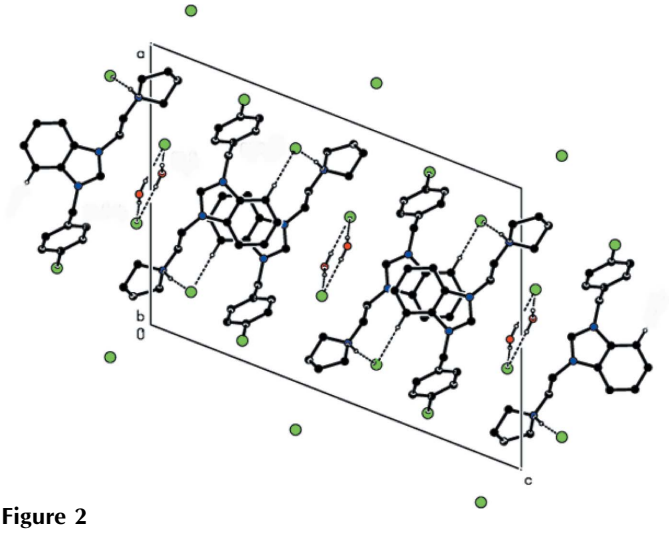


Figure 2
Part of the crystal structure of (I), showing hydrogen bonds as dashed lines. For the sake of clarity, H atoms not involved in hydrogen bonding have been omitted.

angle of 68.33 (8)°, with the mean plane of the benzimidazole ring system (N1/N2/C1–C7). The five-membered pyrrolidine ring (N3/C17–C20) has a slightly distorted envelope conformation, with puckering parameters of $Q(2)$ = 0.359 (3) Å and $\varphi(2)$ = 235.5 (5)° (Cremer & Pople, 1975), and with C18 at the flap.

The crystal structure of (I) is stabilized by O—H···Cl, C—H···Cl, N—H···Cl and C—H···O hydrogen bonds (Table 1 and Fig. 2).

Experimental

A mixture of 1-(4-chlorobenzyl)benzimidazole (1.00 g, 4.12 mmol) and 2-chloroethylpyrrolidine hydrochloride (0.7 g, 4.12 mmol) was heated at reflux in DMF (5 ml) for 2 h. All volatiles were then removed *in vacuo*. The resulting crude title compound (I) was crystallized from an EtOH–Et_2O (3:1) mixture (yield 1.40 g, 79%; m.p. 431–433 K). ^{1}H-NMR (D_2O): δ 1.5–2.1 (*m*, ring methylene, 4H), 3.0 (*m*, ring methylene, 2H), 3.4 (*t*, ring methylene, 2H), 4.0 (*t*, pyrrolidine-CH_2CH_2–, 2H), 4.9 (*t*, pyrrolidine-CH_2CH_2–, 2H), 5.8 (*s*, $PhCH_2$–, 2H), 6.8–7.8 (*m*, Ar—H, 8H), 9.7 (*s*, pyrrolidine-1H). Analysis calculated for $C_{20}H_{26}N_3OCl_3$: C 55.75, H 6.04, N 9.76%; found: C 55.63, H 5.58, N 9.80%.

Crystal data

$C_{20}H_{24}ClN_3^{2+}\cdot 2Cl^-\cdot H_2O$
M_r = 430.79
Monoclinic, $P2_1/c$
a = 15.3427 (8) Å
b = 6.8703 (2) Å
c = 21.7329 (11) Å
β = 111.355 (4)°
V = 2133.56 (18) Å^3
Z = 4
D_x = 1.341 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.45 mm^{-1}
T = 296 K
Prism, colorless
0.50 × 0.34 × 0.17 mm

Data collection

Stoe IPDS-2 diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.808, T_{max} = 0.928
39631 measured reflections
4634 independent reflections
3223 reflections with $I > 2\sigma(I)$
R_{int} = 0.061
θ_{max} = 27.2°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.105
S = 0.98
4634 reflections
253 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0666P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0127 (13)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H*W*1···Cl2^{i}	0.81 (3)	2.34 (3)	3.151 (2)	174 (3)
O1—H*W*2···Cl2	0.87 (3)	2.35 (3)	3.219 (2)	176 (3)
N3—H3*A*···Cl3	0.91	2.09	2.9965 (16)	172
C6—H6···Cl3^{ii}	0.93	2.78	3.6243 (19)	152
C14—H14···O1	0.93	2.56	3.429 (3)	156
C15—H15*B*···Cl3	0.97	2.79	3.5814 (19)	139

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

The H atoms of the water molecule were found in a difference Fourier map and refined isotropically. All other H atoms were positioned geometrically and refined in the riding-model approximation, with N—H = 0.91 and C—H = 0.93–0.97 Å, and with U_{iso}(H) = 1.2U_{eq}(C,N).

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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References

Akkurt, M., Öztürk, S., Şireci, N., Küçükbay, H. & Büyükgüngör, O. (2004). *Acta Cryst.* E**60**, o1185–o1187.
Akkurt, M., Türktekin, S., Küçükbay, H., Yılmaz, Ü. & Büyükgüngör, O. (2004). *Acta Cryst.* E**60**, o2135–o2137.
Akkurt, M., Türktekin, S., Küçükbay, H., Yılmaz, U. & Büyükgüngör, O. (2005). *Acta Cryst.* E**61**, o166–o168.
Akkurt, M., Türktekin, S., Şireci, N., Küçükbay, H. & Büyükgüngör, O. (2006). *Acta Cryst.* E**62**, o185–o187.
Akkurt, M., Yıldırım, S. O., Küçükbay, H., Şireci, N. & Fun, H.-K. (2006). *Acta Cryst.* E**62**, o922–o924.
Akkurt, M., Yıldırım, S. O., Küçükbay, H., Yılmaz, U. & Büyükgüngör, O. (2005). *Acta Cryst.* E**61**, o301–o303.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Karaca, S., Akkurt, M., Yılmaz, U., Küçükbay, H. & Büyükgüngör, O. (2005). *Acta Cryst.* E**61**, o2128–o2130.
Küçükbay, H., Çetinkaya, E. & Durmaz, R. (1995). *Arzneim.-Forsch./Drug. Res.* **45**, 1331–1334.
Küçükbay, H., Durmaz, R., Okuyucu, N., Günal, S. & Kazaz, C. (2004). *Arzneim.-Forsch./Drug. Res.* **54**, 64–68.
Küçükbay, H., Durmaz, R., Orhan, E. & Günal, S. (2003). *Il Farm.*, **58**, 431–437.
Pınar, Ş., Akkurt, M., Küçükbay, H., Şireci, N. & Büyükgüngör, O. (2006). *Acta Cryst.* E**62**, o2223–o2225.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Stoe & Cie (2002). *X-AREA* (Version 1.18) and *X-RED32* (Version 1.04). Stoe & Cie, Darmstadt, Germany.
Türktekin, S., Akkurt, M., Şireci, N., Küçükbay, H. & Büyükgüngör, O. (2004). *Acta Cryst.* E**60**, o817–o819.