Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

1,3-Bis(4-methylbenzyl)benzimidazolium chloride monohydrate

Şerife Pınar,[a] Mehmet Akkurt,[a]* Hasan Küçükbay,[b] Nihat Şireci[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

Received 20 April 2006
Accepted 1 May 2006

Key indicators

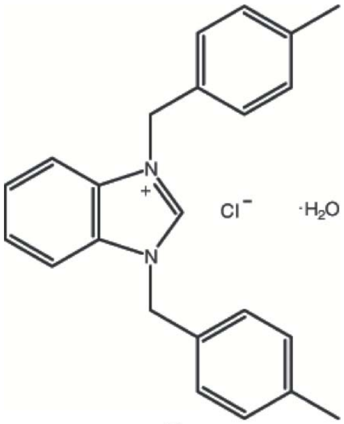
Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.004 Å
R factor = 0.059
wR factor = 0.160
Data-to-parameter ratio = 18.6

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

The cation in the title compound, $C_{23}H_{23}N_2^+ \cdot Cl^- \cdot H_2O$, features a benzimidazole ring connected to two 4-methylbenzyl rings via methylene bridges. The crystal structure is stabilized by C—H···O, C—H···Cl and O—H···Cl hydrogen-bonding interactions, leading to a three-dimensional framework.

Comment

Benzimidazole and its derivatives are interesting heterocyclic compounds with versatile pharmacological activities (Küçükbay *et al.*, 2001, 2003, 2004; Soderlind *et al.*, 1999) and are present in various naturally occurring drugs, such as omeprazole (Carlsson *et al.*, 2002), astemizole and emedastine difumarate (Sakai *et al.*, 1989). Owing to their important pharmacological activities, these compounds have received a great deal of attention in connection with their synthesis and their molecular structures, as determined by X-ray crystallography. The structure of the title compound, (I), was determined in connection with our interest in investigating the crystal structures of benzimidazoles (Akkurt *et al.*, 2003, 2004; Öztürk *et al.*, 2001, 2003; Türktekin *et al.*, 2004).



(I)

As seen in Fig. 1, the asymmetric unit of (I) comprises a 1,3-di(4-methylbenzyl)benzimidazolium cation, one Cl^- anion and one water molecule. The C—C and C—N bond lengths in (I) (Table 1) are similar to those found in similar compounds (Allen *et al.*, 1987). The planes of the C9–C14 and C17–C22 benzyl rings form dihedral angles of 70.35 (16)° with each other and 79.24 (13) and 75.26 (11)°, respectively, with the central benzimidazole ring.

The crystal structure of (I) is stabilized by C—H···O, C—H···Cl and O—H···Cl hydrogen-bonding interactions, as detailed in Table 2, which lead to the formation of a three-dimensional framework (Fig. 2).

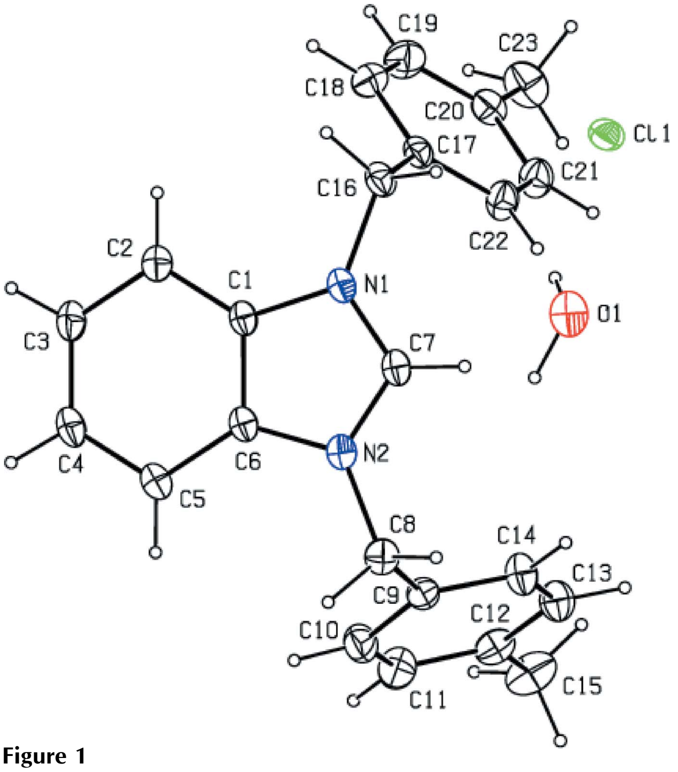


Figure 1
The asymmetric unit of (I), with the atom-numbering scheme and 10% probability displacement ellipsoids.

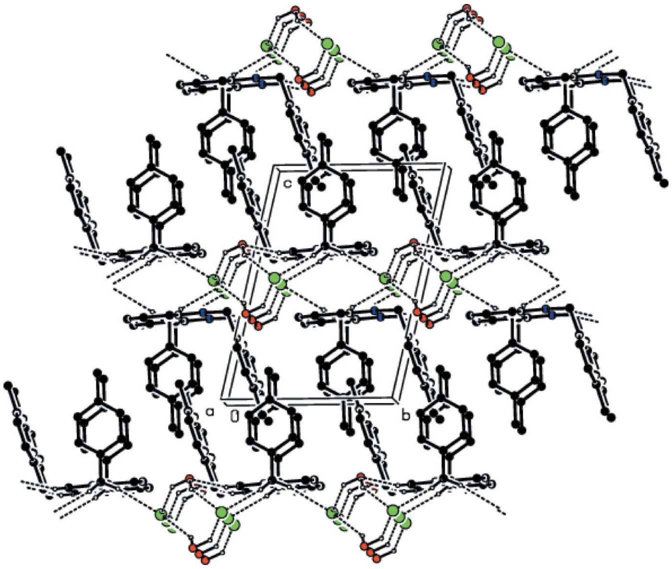


Figure 2
A view of the packing for (I). ydrogen bonds are shown as dashed lines. The H atoms not involved in hydrogen bonding have been omitted for clarity.

Experimental

4-Methylbenzyl chloride (12.15 g, 84.6 mmol) was added to a solution of benzimidazole (5.00 g, 42.3 mmol) and KOH (2.40 g, 42.7 mmol) in EtOH (60 ml). The mixture was heated under reflux for 6 h. The mixture was then cooled, after which KCl was filtered off and the volatiles were removed from the filtrate *in vacuo*. The residue obtained was then crystallized from EtOH–dimethylformamide (3:1) (yield 11.84 g, 73%; m.p. 470–471 K). In the ^{13}C NMR spectrum (details in the CIF), only nine signals are observed, owing to the magnetic equivalence of the C2 and C3 nuclei. Analysis, calculated for $C_{23}H_{25}ClN_2O$: C 72.53, H 6.57, N 7.36%; found: C 72.56, H 6.62, N 7.49%.

Crystal data

$C_{23}H_{23}N_2^+\cdot Cl^-\cdot H_2O$
M_r = 380.90
Triclinic, $P\overline{1}$
a = 9.3636 (8) Å
b = 9.4834 (8) Å
c = 12.6509 (12) Å
α = 75.558 (7)°
β = 82.798 (7)°
γ = 73.581 (7)°

V = 1041.70 (17) Å^3
Z = 2
D_x = 1.214 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.20 mm^{-1}
T = 296 K
Prism, colourless
0.58 × 0.55 × 0.52 mm

Data collection

Stoe IPDS2 diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
$T_{\min}$ = 0.894, $T_{\max}$ = 0.904

23747 measured reflections
4724 independent reflections
3043 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.060
$\theta_{\max}$ = 27.7°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.160
S = 1.02
4724 reflections
254 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0755P)^2 + 0.1339P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max}$ = 0.35 e Å^{-3}
$\Delta\rho_{\min}$ = −0.17 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C1	1.397 (3)	N2—C6	1.390 (3)
N1—C7	1.325 (4)	N2—C7	1.321 (3)
N1—C16	1.479 (3)	N2—C8	1.472 (4)
C1—N1—C7	107.71 (19)	C6—N2—C7	107.9 (2)
C1—N1—C16	126.6 (2)	C7—N2—C8	124.9 (2)
C7—N1—C16	125.6 (2)	N1—C1—C6	106.36 (17)
C6—N2—C8	126.97 (19)		
C1—N1—C16—C17	−84.9 (3)	C6—N2—C8—C9	86.8 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H101···Cl1^{i}	1.02 (5)	2.41 (4)	3.189 (3)	132 (3)
O1—H102···Cl1	0.84 (5)	2.43 (5)	3.155 (3)	146 (4)
C7—H7···O1^{i}	0.93	2.32	3.227 (4)	164
C8—H8a···Cl1^{ii}	0.97	2.74	3.692 (3)	167
C8—H8b···Cl1^{i}	0.97	2.77	3.730 (3)	170
C16—H16a···Cl1	0.97	2.67	3.633 (3)	170

Symmetry codes: (i) $-x+1, -y, -z+1$; (ii) $x, y+1, z$.

H atoms attached to C atoms were placed in geometrically idealized positions, with Csp^3—H = 0.96 Å (for methyl), Csp^3—H = 0.97 Å (for methylene) and Csp^2—H = 0.93 Å, and constrained to

o2224 Pınar *et al.* • $C_{23}H_{23}N_2^+\cdot Cl^-\cdot H_2O$ *Acta Cryst.* (2006). E**62**, o2223–o2225

ride on their parent atoms, with $U_{iso}(H) = 1.5U_{eq}(C)$ (for methyl) and $U_{iso}(H) = 1.2U_{eq}(C)$ (for others). Water H atoms were located in difference Fourier maps and refined freely. The O—H distances are given in Table 2.

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).

The authors acknowledge the Faculty of Arts and Sciences, Ondokuz Mayıs University, Turkey, for the use of the Stoe IPDS2 diffractometer (purchased under grant F.279 of the University Research Fund). HK and NŞ also thank İnönü University (grant No. 2005/36) for financial support for this study.

References

Akkurt, M., Öztürk, S., Küçükbay, H., Okuyucu, N. & Fun, H.-K. (2003). *Acta Cryst.* E**59**, o786–o788.

Akkurt, M., Öztürk, S., Küçükbay, H., Orhan, E. & Büyükgüngör, O. (2004). *Acta Cryst.* E**60**, o219–o221.

Allen, F. H., Kennard, O., Watson, D., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.

Carlsson, E., Lindberg, P. & Unge, S. (2002). *Chem. Br.*, **5**, 42–45.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.

Küçükbay, H., Durmaz, R., Güven, M. & Günal, S. (2001). *Arzneim.-Forsch./Drug Res.* **51**, 420–424.

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 Farmaco*, **58**, 431–437.

Öztürk, S., Akkurt, M., Küçükbay, H. & Fun, H.-K. (2001). *Anal. Sci.* **17**, 1015–1016.

Öztürk, S., Akkurt, M., Küçükbay, H., Okuyucu, N. & Fun, H.-K. (2003). *Acta Cryst.* E**59**, o1014–o1016.

Sakai, T., Hamada, T., Awata, N. & Watanabe, J. (1989). *Pharmacobiol. Dyn.* **12**, 530–536.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Soderlind, K. J., Gorodetsky, B., Singh, A. K., Bachur, N. R., Miller, G. G. & Lown, J. W. (1999). *Anti-Cancer Drug Res.* **14**, 19–36.

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.

Acta Cryst. (2006). E**62**, o2223–o2225 Pınar *et al.* • $C_{23}H_{23}N_2^+ \cdot Cl^- \cdot H_2O$ **o2225**