Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3,3′-Bis(cyclohexylmethyl)-1,1′-propylene-dibenzimidazolium dibromide monohydrate

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.006 Å
R factor = 0.045
wR factor = 0.110
Data-to-parameter ratio = 16.9

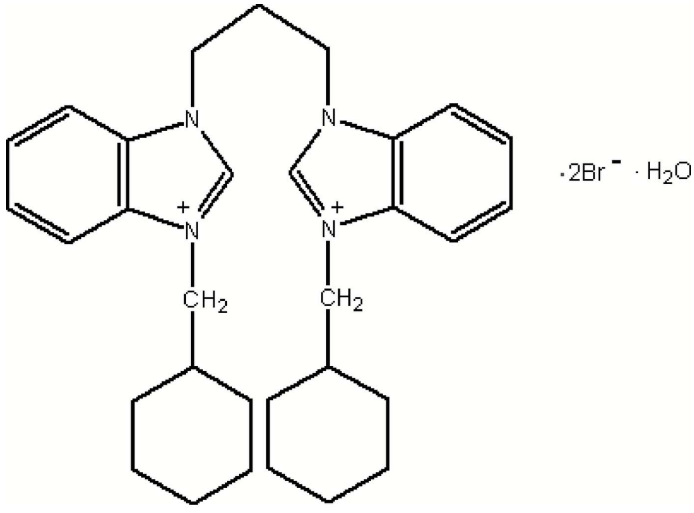
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 14 June 2006
Accepted 19 June 2006

The title compound, $C_{31}H_{42}N_4^{2+}\cdot 2Br^-\cdot H_2O$, was synthesized from 1,1′-propylendibenzimidazole and cyclohexylmethyl bromide in dimethylformamide solution. In the molecule, the benzimidazole ring systems are connected to the cyclohexane rings by methylene groups and to each other by a propylene group. The crystal structure is stabilized by intermolecular O—H···Br hydrogen bonds involving the H atoms of the water molecule.

Comment

In recent years, much attention has been devoted to benzimidazole compounds because of their versatile pharmacological activities, such as antitumour, diuretic, fungicidal, bactericidal, anthelmintic, anti-allergic, vasodilator, antihistaminic, anti-ulcer and local analgesic properties (Carlsson *et al.*, 2002; Kotovskaya *et al.*, 2006; Sondhi *et al.*, 2006). We have reported the syntheses and antimicrobial activities of many benzimidazole derivatives (Küçükbay *et al.*, 2001, 2003, 2004; Küçükbay & Durmaz, 1997). The objective of this study was to synthesize and elucidate the crystal structure of the title compound, (I), and compare the results with those obtained in our previous studies of related benzimidazole derivatives (Akkurt, *et al.*, 2005, 2006; Karaca *et al.*, 2005; Pınar *et al.*, 2006).



(I)

The values of the geometric parameters of (I) are within the ranges of normally accepted values (Allen *et al.*, 1987). In the title molecule (Fig. 1), the two benzimidazole ring systems, *A* (N1/N2/C1–C6/C14) and *B* (N3/N4/C18–C24), are essentially planar, with maximum deviations of 0.014 (4) Å for C6 in *A*

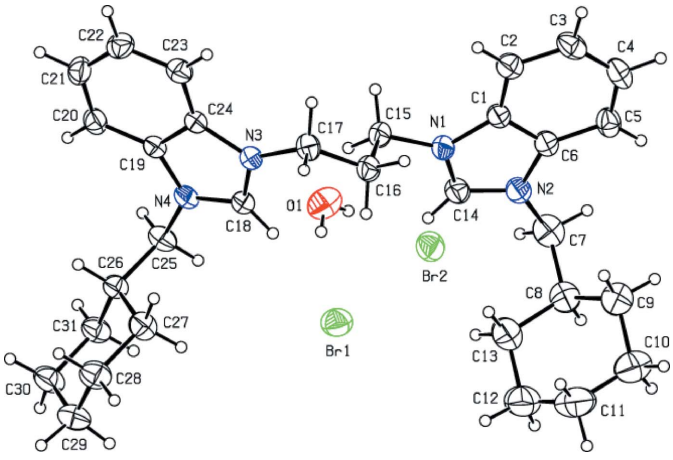


Figure 1
The asymmetric unit of (I), showing the atom-numbering scheme and 30% probability displacement ellipsoids. H atoms are shown as small spheres.

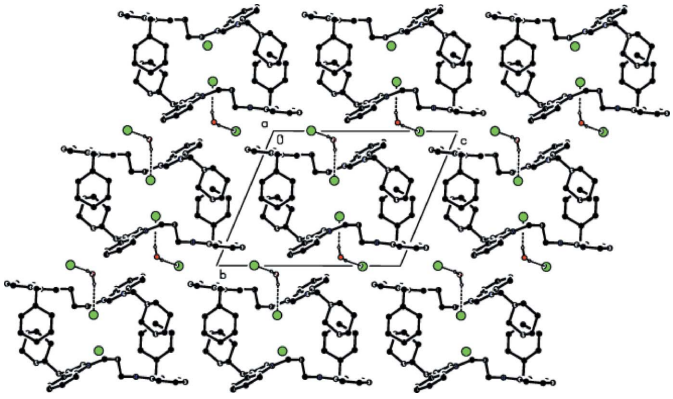


Figure 2
View along the *a* axis, showing the packing and hydrogen bonds (dashed lines) in the crystal structure of (I). H atoms not involved in hydrogen bonding have been omitted.

and 0.013 (3) Å for N3 in *B*. The dihedral angle between the planes of these benzimidazole ring systems is 31.84 (11)°. The cyclohexane rings (C8–C13 and C26–C31) have chair conformations (Boeyens, 1978), with puckering parameters Q_T = 0.540 (6) Å, θ = 180.0 (6)° and φ = 207 (23)° for the C8–C13 ring, and Q_T = 0.563 (5) Å, θ = 3.2 (4)° and φ = 12 (8)° for the C26–C31 ring (Cremer & Pople, 1975).

The crystal structure is stabilized by intermolecular O—H···Br hydrogen bonds involving the H atoms of the water molecule (Fig. 2), and also by weak intermolecular C—H···Br and C—H···N interactions (Table 1).

Experimental

1,1′-Propylenedibenzimidazole was synthesized according to the literature method of Küçükbay *et al.* (2005). A mixture of 1,1′-propylenedibenzimidazole (0.9 g, 3.26 mmol) and cyclohexylmethyl bromide (1.2 g, 6.78 mmol) in dimethylformamide (DMF, 5 ml) was heated under reflux for 3 h. The mixture was then cooled and the volatiles were removed *in vacuo*. The residue was crystallized from a DMF/EtOH (1:3) mixture (yield: 1.77 g, 83%; m.p. 454–455 K). Analysis, calculated for $C_{16}H_{25}N_3I_2$: C 37.42, H 4.87, N 8.18%; found: C 37.71, H 4.87, N 8.24%.

Crystal data

$C_{31}H_{42}N_4^{2+}\cdot 2Br^-\cdot H_2O$
M_r = 648.50
Triclinic, $P\overline{1}$
a = 9.2538 (5) Å
b = 12.5925 (8) Å
c = 15.4610 (9) Å
α = 105.049 (5)°
β = 105.593 (4)°
γ = 110.034 (4)°
V = 1502.14 (18) Å^3
Z = 2
D_x = 1.434 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 2.73 mm^{-1}
T = 296 K
Prism, colourless
0.68 × 0.55 × 0.36 mm

Data collection

STOE IPDS-II diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.258, T_{max} = 0.440
29924 measured reflections
5915 independent reflections
4567 reflections with $I > 2\sigma(I)$
R_{int} = 0.049
θ_{max} = 26.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.110
S = 1.05
5915 reflections
351 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.047P)^2 + 1.0857P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.57 e Å^{-3}
$\Delta\rho_{min}$ = −0.65 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···Br1	0.82 (3)	2.50 (3)	3.305 (5)	167 (3)
O1—H1B···Br2	0.82 (5)	2.60 (5)	3.421 (4)	173 (5)
C7—H7B···Br2	0.97	2.84	3.805 (5)	176
C18—H18···Br1	0.93	2.59	3.495 (4)	165
C27—H27B···N4	0.97	2.61	2.936 (5)	100

The C-bound H atoms were positioned geometrically, with C—H = 0.93–0.98 Å, and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C). The H atoms of the water molecule were located in a difference Fourier map and refined isotropically.

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 IPDS-II diffractometer (purchased under grant F.279 of the University Research Fund). HK and NŞ also thank İnönü University Research Fund (BAPB-2005/36 and 2005/37) for financial support for this study.

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