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3,3′-Bis(3-methylbut-2-enyl)-1,1′-propylenedibenzimidazolium 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 = 0.038
wR factor = 0.093
Data-to-parameter ratio = 16.6

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

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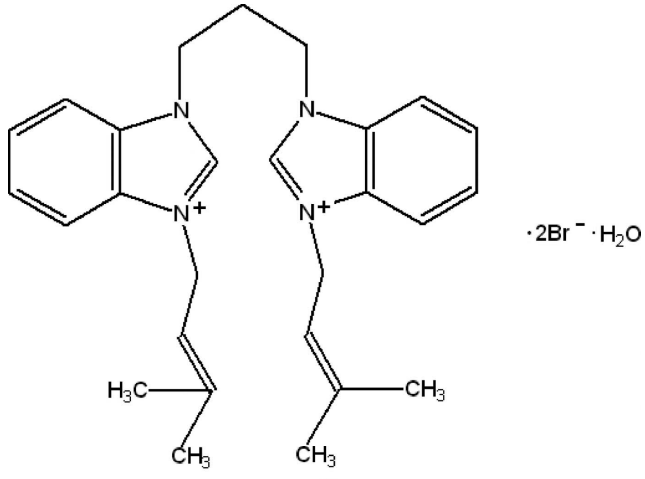
The title compound, $C_{27}H_{34}N_4^{2+}\cdot 2Br^-\cdot H_2O$, was synthesized from 1,1′-propylendibenzimidazole and 1-bromo-3-methylbut-2-ene in dimethylformamide solution. The compound crystallizes with one water molecule and two Br^- ions in the asymmetric unit. The crystal structure is stabilized by inter- and intramolecular O—H···Br and C—H···Br hydrogen-bonding interactions.

Comment

Dibenzimidazole compounds display a wide range of pharmacological activities, such as antitumour, diuretic, fungicidal, bactericidal, antihelmintic, anti-allergic, vasodilator, antihistaminic, anti-ulcer and local analgesic properties (Del Poeta *et al.*, 1998; Soderlind *et al.*, 1999; Singh & Lown, 2000; Küçükbay *et al.*, 2003, 2004). Therefore, it seemed of interest to synthesize new bisbenzimidazole compounds. The aim of the present study was to synthesize and elucidate the crystal structure of the new bisbenzimidazole compound, (I), and compare the results with those obtained from our previous studies of related bisbenzimidazole derivatives (Öztürk *et al.*, 2003; Akkurt *et al.*, 2003, 2006*a*,*b*).



(I)

The molecular structure of (I) is shown in Fig. 1, with the atom-numbering scheme. The arrangement of the molecules in the unit cell is shown in Fig. 2. The benzimidazole ring systems *A* (N1/N2/C1–C7) and *B* (N3/N4/C16–C22) are essentially planar, with maximum deviations of 0.014 (3) Å for atom C6 in *A*, and 0.019 (2) Å for atom N3 in *B*. The dihedral angle between the least-squares planes of the *A* and *B* benzimidazole ring systems is 27.33 (11)°. The values of the observed bond lengths and angles in (I) have normal values

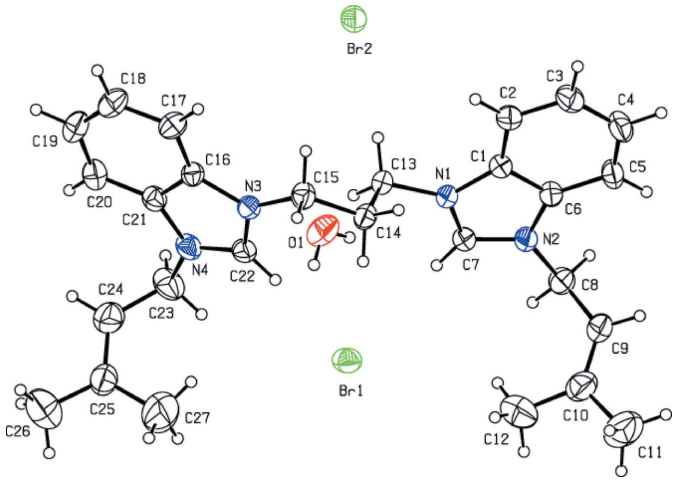


Figure 1
A view of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

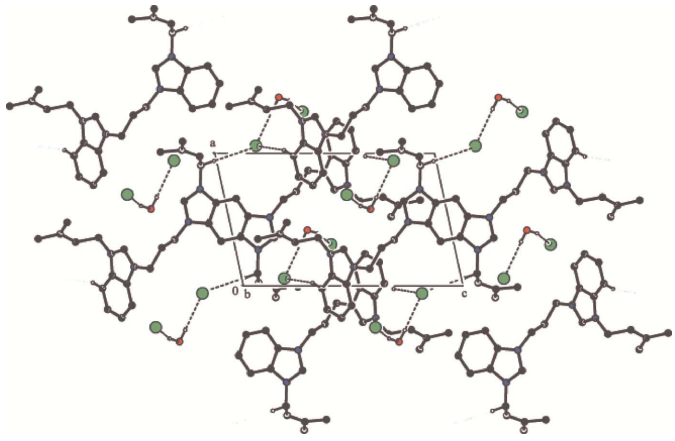


Figure 2
A view of the packing and hydrogen bonding (dashed lines) in (I), along the *b* axis. H atoms not involved in the hydrogen bonding have been omitted for clarity.

and they are in good agreement with those observed in the literature (Allen *et al.*, 1987; Akkurt *et al.*, 2005; Karaca *et al.*, 2005; Pınar *et al.*, 2006).

The crystal structure of (I) is stabilized by inter- and intramolecular O—H···Br and C—H···Br hydrogen-bonding interactions. The details of these interactions can be seen in Table 1.

Experimental

1,1′-Propylendibenzimidazole was synthesized according to the literature method of Küçükbay *et al.* (1995). A mixture of 1,1′-propylendibenzimidazole (1.0 g, 3.62 mmol) and 1-bromo-3-methyl-2-butene (1.2 g, 7.73 mmol) in dimethylformamide (DMF; 5 ml) was heated under reflux for 5 h. The mixture was then cooled and the volatiles were removed under vacuum. The residue was crystallized from a DMF–EtOH (1:3) mixture (yield 1.83 g, 85%; m.p. 477–478 K). Analysis calculated for $C_{27}H_{36}N_4Br_2O$: C 54.73, H 6.08, N 9.46%; found: C 54.84, H 6.09, N 9.60%.

Crystal data

$C_{27}H_{34}N_4^{2+}\cdot 2Br^-\cdot H_2O$
M_r = 592.40
Triclinic, $P\overline{1}$
a = 8.7777 (4) Å
b = 12.0851 (7) Å
c = 14.4078 (7) Å
α = 106.966 (4)°
β = 96.868 (4)°
γ = 104.747 (4)°
V = 1382.30 (13) Å^3
Z = 2
D_x = 1.423 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 2.96 mm^{-1}
T = 296 K
Prism, colourless
0.62 × 0.49 × 0.32 mm

Data collection

Stoe IPDS II diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.261, T_{max} = 0.451
25534 measured reflections
5267 independent reflections
4128 reflections with $I > 2\sigma(I)$
R_{int} = 0.063
θ_{max} = 26.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.093
S = 1.04
5267 reflections
317 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0473P)^2 + 0.3788P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···Br1	0.79 (6)	2.62 (6)	3.313 (4)	146 (5)
O1—H1B···Br2^{i}	0.82 (5)	2.55 (5)	3.360 (3)	169 (5)
C7—H7···Br1	0.93	2.80	3.586 (3)	142
C8—H8B···Br2^{ii}	0.97	2.89	3.819 (3)	160
C13—H13B···Br2	0.97	2.91	3.832 (3)	159
C20—H20···Br2^{iii}	0.93	2.91	3.812 (3)	163
C22—H22···Br1	0.93	2.62	3.523 (3)	164

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+1, -y+1, -z$; (iii) $-x+2, -y+2, -z+1$.

Carbon-bound H atoms were positioned geometrically, with C—H = 0.93–0.97 Å, and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}($C_{aromatic}$ or $C_{methylene}$) or 1.5U_{eq}(C_{methyl}). 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: *SIR97* (Altomare *et al.*, 1999); 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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