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2,2′-(Butane-1,4-diyl)dibenzimidazolium dichloride dihydrate

Liliana Dobrzańska‡ and Gareth O. Lloyd*

Department of Chemistry, University of Stellenbosch, Private Bag X1, Matieland, South Africa

‡ Permanent address: Faculty of Chemistry, Nicolaus Copernicus University, Toruń, Poland.

Correspondence e-mail: gol@sun.ac.za

Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.033
wR factor = 0.087
Data-to-parameter ratio = 17.7

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

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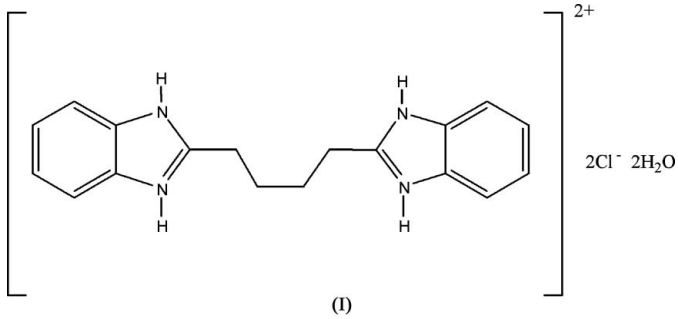
In the title compound, $C_{18}H_{24}Cl_2N_4O_2$, a quarternary adduct consisting of two water molecules and two chloride ions binds the centrosymmetric 2,2′-(butane-1,4-diyl)dibenzimidazolium cations into two-dimensional hydrogen-bonded layers.

Comment

As a continuation of our work with imidazole-functionalized ligands we prepared the ligand 1,4-bis(benzimidazol-2-yl)butane using the modified Phillips reaction (Dobrzańska, 2005; Dobrzańska, Lloyd *et al.*, 2005, 2006; Dobrzańska, Raubenheimer & Barbour, 2005). It is often stated that the unpurified reaction product that precipitates from the acidified reaction mixture on cooling is the hydrochloride of the ligand. In this report, we show that the product is, in fact, the dihydrochloride dihydrate, (I) (Fig. 1).



(I)

The conformations of the $-CH_2-$ groups of the 1,4-bis(benzimidazol-2-yl)butane ligand of (I) are all *anti*. This differs from the previously reported structure of the pure compound but is similar to some of the conformations observed in the relatively few examples involving the coordination chemistry of this compound (Chen *et al.*, 2002, 2005; Yang *et al.*, 2005). Owing to the *anti* conformations, the entire molecule is relatively flat, the largest deviation from planarity being reflected in the torsion angle C2—C10—C11—C11^{iii} = 177.23 (14)° [symmetry code: (iii) $1-x, -y, 2-z$]. The most prominent hydrogen-bonding feature in this structure is a four-membered hydrogen-bonded ring consisting of two water molecules and two chloride anions (Table 1 and Fig. 2). These rings bind *via* N—H···Cl and N—H···O(water) hydrogen bonds to four 1,4-bis(benzimidazol-2-yl)butane ligands, resulting in the formation of two-dimensional hydrogen-bonded layers running parallel to (111) (Fig. 3). These two-dimensional layers interact strongly with one another through π–π interactions.

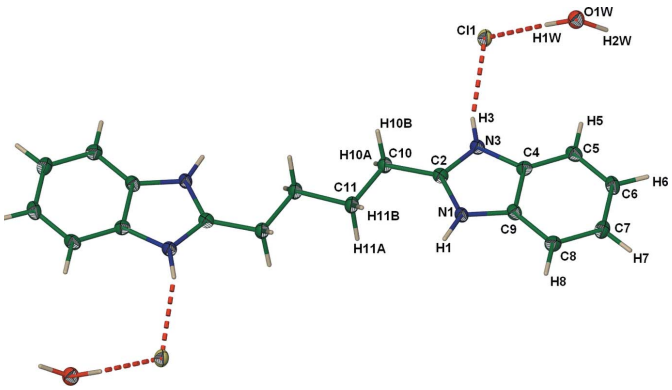


Figure 1
The structure of (I), showing atom labels and 70% probability displacement ellipsoids for non-H atoms. H atoms are shown in capped-stick representation. Unlabelled atoms are related by the symmetry operation $(1-x, -y, 2-z)$. Hydrogen bonds are shown as dashed lines.

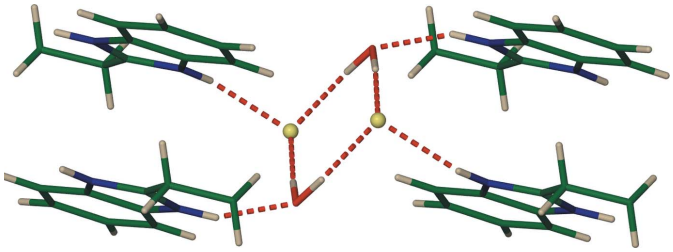


Figure 2
The hydrogen-bonding pattern involving the water, chloride and benzimidazole groups in (I). All molecules, except chloride ions, which are shown as spheres, are shown in capped-stick representation. Hydrogen bonds are shown as dashed lines.

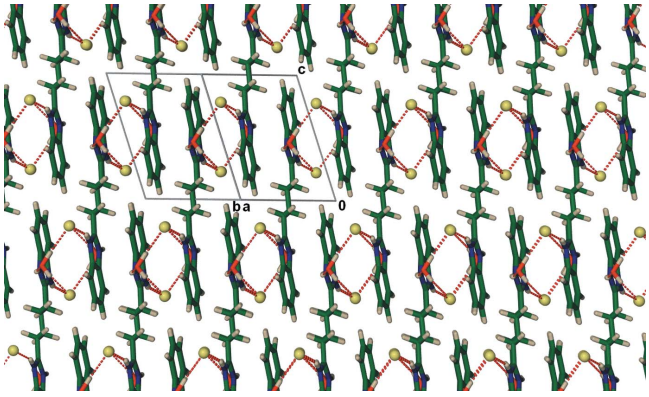


Figure 3
Molecular packing of the two-dimensional hydrogen-bonded layers *via* π–π interactions, with the molecules shown in capped-stick representation, viewed parrallel to [110].

Experimental

Compound (I) was prepared from a 2:1 molar ratio of hexanedioic acid (1.00 g) and 1,2-diaminobenzene (1.64 g) using a modified Phillips reaction [reflux in 4 *M* HCl (60 ml) for 24 h] and was recrystallized from a 4 *M* HCl solution. Single crystals suitable for X-ray analysis were then obtained by slow evaporation of a 4 *M* HCl solution at 323 K (Isele *et al.*, 2002; Preston, 1974; Wang & Joullié, 1957).

Crystal data

$C_{18}H_{20}N_4^{2+}\cdot 2Cl^-\cdot 2H_2O$
$M_r = 399.31$
Triclinic, $P\overline{1}$
$a = 6.9671\ (5)$ Å
$b = 8.6567\ (7)$ Å
$c = 8.8036\ (7)$ Å
$\alpha = 69.342\ (1)^\circ$
$\beta = 79.171\ (1)^\circ$
$\gamma = 69.886\ (1)^\circ$
$V = 465.18\ (6)$ Å^3
$Z = 1$
$D_x = 1.425$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1899 reflections
$\theta = 3.1$–27.8°
$\mu = 0.37$ mm^{-1}
$T = 100\ (2)$ K
Block, colourless
0.38 × 0.30 × 0.23 mm

Data collection

Bruker APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
5344 measured reflections
2129 independent reflections
1939 reflections with $I > 2\sigma(I)$
$R_{int} = 0.053$
$\theta_{max} = 28.3^\circ$
$h = -9 \rightarrow 8$
$k = -11 \rightarrow 11$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.087$
$S = 1.07$
2129 reflections
120 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0388P)^2 + 0.1613P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···Cl1	0.88	2.28	3.1169 (13)	159
O1*W*—H1*W*···Cl1	0.98	2.18	3.1594 (11)	176
O1*W*—H2*W*···Cl1^{i}	0.97	2.19	3.1563 (12)	174
N1—H1···O1*W*^{ii}	0.88	1.94	2.7433 (17)	151

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

All non-water H atoms were positioned geometrically (C—H = 0.95 and 0.99 Å for aromatic CH and CH_2, respectively, and N—H = 0.88 Å) and constrained to ride on their parent atoms; U_{iso}(H) values were set to $1.2U_{eq}$(C,N). Water H atoms were restrained to 0.95 (2) Å and U_{iso}(H) values were allowed to refine independently.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001; Atwood & Barbour, 2003); software used to prepare material for publication: *X-SEED*.

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