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3,3′-Bis(3-cyanopropyl)-1,1′-propylene-di(benzimidazolium) dichloride dihydrate

Mehmet Akkurt,[a] Sema Öztürk,[a]* Hasan Küçükbay,[b] Naíf Okuyucu[b] and Hoong-Kun Fun[c]

[a]Department of Physics, Faculty of Arts and Sciences, Erciyes University, 38039 Kayseri, Turkey, [b]Department of Chemistry, Faculty of Arts and Sciences, Ínönü University, 44069 Malatya, Turkey, and [c]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia

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

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.049
wR factor = 0.154
Data-to-parameter ratio = 19.3

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

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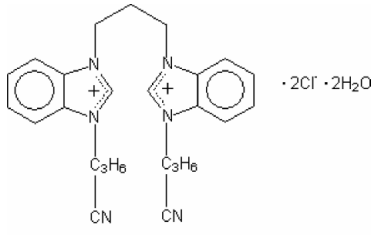
In the title compound, $C_{25}H_{28}N_6^{2+}\cdot 2Cl^-\cdot 2H_2O$, the dihedral angle between the two benzimidazole groups is 88.42 (4)°. The crystal structure is stabilized by hydrogen bonds.

Comment

Many benzimidazole compounds are known to possess versatile pharmacological activities, such as antibacterial, antifungal, antihelmintic, antiallergic, antineoplastic, local analgesic, antihistaminic, vasodilator, hypotensive and spasmolytic activities (Carlsson *et al.*, 2002; Del Poeta *et al.*, 1998; Hall *et al.*, 1998). In our previous studies (Çetinkaya *et al.*, 1996; Küçükbay *et al.*, 1995, 2001; Küçükbay & Durmaz, 1997), we also observed that many benzimidazole derivatives have shown considerable antibacterial and antifungal activity against standard strains, *viz*. *Enterococcus faecalis* (ATCC 29212), *Staphylococcus aureus* (ATCC 29213), *Escherichia coli* (ATCC25922), *Pseudomonas aeruginosa* (ATCC27853) and the yeast-like fungi *Candida albicans* and *Candida tropicalis*. In recent years, bis-benzimidazole compounds have begun to attract particular interest because of their potential use in cancer therapy in DNA-binding blocking (Turner & Denny, 1996). The aim of this study was to elucidate the crystal structure of new bis-benzimidazole derivatives and compare them with those of related benzimidazole derivatives reported previously (Çetinkaya *et al.*, 1994; Aydın *et al.*, 1998, 1999; Íngeç *et al.*, 1999; Öztürk *et al.*, 2001).



(I)

A view of the title compound, (I), is shown in Fig. 1 and selected geometric parameters are listed in Table 1. In (I), two benzimidazolium rings are connected *via* atoms C8, C9 and C10. The average bond lengths and angles involving the (N2)C8/C9/C10/(N3) group [C—C = 1.507 (3) Å, C—N = 1.476 (3) Å and C—C—C = 112.1 (6)°] are consistent with those observed in bis(1-methyl-3-ethylbenzimidazolidine-2-ylium) tetrafluoroborate (Aydın *et al.*, 1998). Within the five-membered ring, the bond lengths indicate delocalized bonding, the N to central C1 and C11 atoms averaging 1.331 (3) Å. In 1-(2-ethoxyethyl)-3-(2-methoxyethyl)benz-

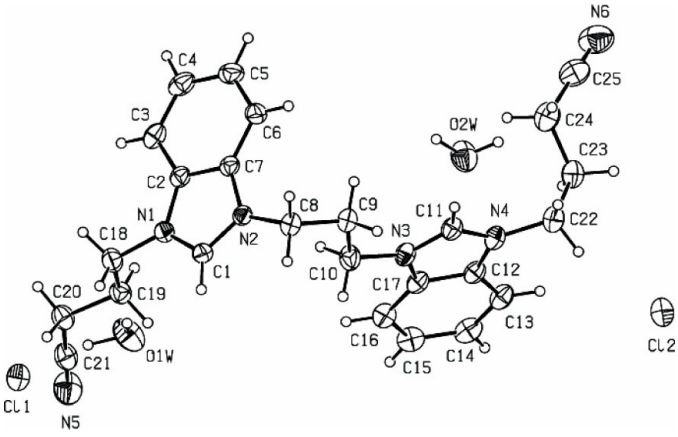


Figure 1
The structure of the title compound, (I), with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level.

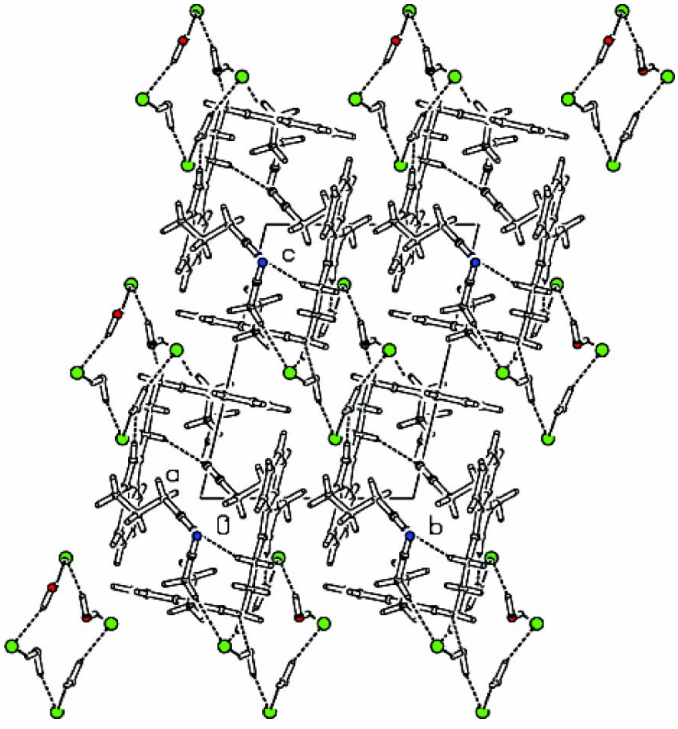


Figure 2
Projection of the crystal structure of (I) along the *a* axis. Hydrogen bonding is indicated by dotted lines.

imidazolium chloride monohydrate (Öztürk *et al.*, 2001), this value is 1.328 (7) Å.

In the molecule, the fused six- and five-membered rings are essentially planar [maxiumum deviations are 0.022 (2) Å for C6 and 0.026 (2) Å for C14], but the cyano groups of the two benzimidazole groups are bent out of plane on opposite sides of the fused rings. The benzimidazole groups are nearly normal to each other, the dihedral angle between their planes being 88.42 (4)°. The short contacts between the molecules and hydrogen bonds, calculated using *PARST* (Nardelli, 1995), are listed in Table 2.

Experimental

All experiments were performed under argon using freshly distilled dry solvents. To a solution of 1,1′-propylenedibenzimidazole (1.15 g, 4.17 mmol) in dimethylformamide (5 ml) 3-cyanopropyl chloride (0.8 ml, 8.34 mmol) was added and the mixture was refluxed for 3 h. Then all volatiles were driven off and the resulting crude product was crystallized from EtOH/Et_2O (3:1) as colorless crystals (1.69 g, 84%). M.p.: 392–393 K. ^{1}H NMR (DMSO): δ 2.33 (*m*, $NCH_2CH_2CH_2CN$, 4H), 2.78 (*t*, $NCH_2CH_2CH_2CN$, 4H), 2.79 (*m*, $NCH_2CH_2CH_2N$, 2H), 4.46 (*t*, $NCH_2CH_2CH_2CN$, 4H), 4.82 (*t*, $NCH_2CH_2CH_2N$, 4H), 7.69–8.23 (*m*, Ar-H, 8H), 10.54 (*s*, CH, 2H).^{13}C NMR (DMSO): δ 15.68, 26.37, 29.82, 45.85, 47.48, 109.21, 115.64, 121.44, 128.36, 133.06, 144.89. Analysis calculated for $C_{28}H_{32}Cl_2N_6$: C 62.11, H 5.80, N 17.39%; found: C 62.13, H 5.80, N 17.47%.

Crystal data

$C_{25}H_{28}N_6^{2+}\cdot 2Cl^-\cdot 2H_2O$
M_r = 519.47
Triclinic, $P\overline{1}$
a = 10.5530 (17) Å
b = 11.0533 (18) Å
c = 13.494 (2) Å
α = 71.698 (3)°
β = 75.434 (3)°
γ = 63.279 (2)°
V = 1323.5 (4) Å^3
Z = 2
D_x = 1.304 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8119 reflections
θ = 2.4–28.3°
μ = 0.28 mm^{-1}
T = 293 K
Slab, colorless
0.44 × 0.26 × 0.24 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.887, T_{max} = 0.936
8119 measured reflections
6112 independent reflections
4561 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 28.3°
$h = -13 \rightarrow 14$
$k = -14 \rightarrow 14$
$l = -17 \rightarrow 7$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.154
S = 1.00
6112 reflections
316 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0902P)^2 + 0.219P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C2	1.393 (3)	N4—C22	1.471 (3)
N1—C18	1.471 (3)	N5—C21	1.138 (4)
N1—C1	1.328 (3)	N6—C25	1.149 (4)
N2—C8	1.474 (3)	C8—C9	1.517 (3)
N2—C7	1.395 (2)	C9—C10	1.497 (3)
N2—C1	1.328 (3)	C18—C19	1.505 (3)
N3—C11	1.328 (3)	C19—C20	1.530 (3)
N3—C17	1.390 (2)	C20—C21	1.454 (3)
N3—C10	1.477 (3)	C22—C23	1.512 (3)
N4—C11	1.338 (2)	C23—C24	1.514 (4)
N4—C12	1.399 (3)	C24—C25	1.477 (4)
C7—N2—C8—C9	−63.9 (2)	C8—C9—C10—N3	−179.30 (18)
C17—N3—C10—C9	−176.08 (18)	C18—C19—C20—C21	−171.87 (17)
N2—C8—C9—C10	−52.0 (2)	C22—C23—C24—C25	170.5 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
O2W—H101···Cl2^{i}	0.89	2.33	3.178 (2)	160
O2W—H102···Cl1	0.98	2.45	3.391 (2)	161
O1W—H201···Cl1	1.02	2.19	3.163 (2)	158
O1W—H202···Cl2	1.07	2.11	3.159 (2)	168
C1—H1A···O1W^{i}	0.93	2.44	3.121 (3)	130
C10—H10B···N2	0.97	2.57	2.912 (3)	101
C10—H10B···N6^{ii}	0.97	2.57	3.503 (4)	161
C11—H11A···O2W^{iii}	0.93	2.15	3.082 (3)	178
C14—H14A···Cl1^{iv}	0.93	2.83	3.740 (2)	166
C20—H20A···Cl1^{i}	0.97	2.71	3.666 (2)	168

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

The H atoms of the water molecules were located in a difference map, and those bound to the C atoms were geometrically positioned. They were allowed to ride on their parent atoms.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 1990) and *WinGX* (Farrugia, 1999).

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