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The hydrogen-bonding network in (*E*)-1,2-bis(1-ethylbenzimidazol-2-yl)ethene dihydrate

Robert T. Stibrany and Joseph A. Potenza*

Department of Chemistry and Chemical Biology, Rutgers, The State University of New Jersey, 610 Taylor Road, Piscataway, New Jersey 08854, USA

Correspondence e-mail:
potenza@rutchem.rutgers.edu

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Key indicators

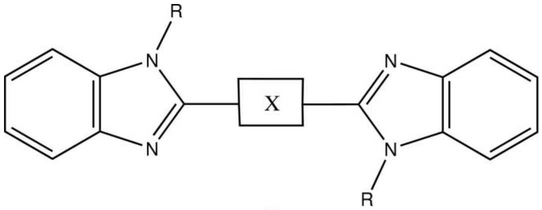
Single-crystal X-ray study
T = 297 K
Mean σ(C–C) = 0.003 Å
R factor = 0.056
wR factor = 0.138
Data-to-parameter ratio = 10.9

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

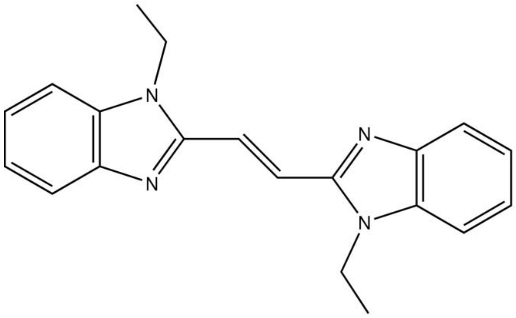
The title compound, $C_{20}H_{20}N_4 \cdot 2H_2O$, contains centrosymmetric (*E*)-1,2-bis(1-ethylbenzimidazol-2-yl)ethene molecules with parallel benzimidazole fragments separated by 0.131 (4) Å. The structure consists of layers parallel to (100). In each layer, alternating chains of water molecules, linked by $O—H\cdots O$ hydrogen bonds, and columns of alkene molecules are linked to each other by $O—H\cdots N_{imine}$ hydrogen bonds to form a two-dimensional polymeric network. An interplanar separation of 3.413 (3) Å between alkene molecules in a given column is consistent with substantial π–π interactions.

Comment

Our continuing interest in the chemistry of bis(benzimidazole) systems of type (I) for use as catalysts (Stibrany, 2001), proton sponges (Stibrany *et al.*, 2002) and geometrically constraining ligands (Stibrany *et al.*, 2004, 2005*a*) led us to prepare and characterize species with the rigid spacer X = ethene, of which (II), whose structure was published previously (Stibrany *et al.*, 2005*b*), is one example. Recrystallization of (II) from ethanol led, by the incorporation of adventitious water, to the dihydrate (II)·$2H_2O$, whose structure we report here.



(I)



(II), obtained as its dihydrate

The molecules of (II) (Fig. 1) are centrosymmetric, with the centers of symmetry located at the midpoints of the ethene fragments (sites 2*a* in space group $P2_1/c$). The benzimidazole groups are essentially planar, and the centers of symmetry ensure that, in a given molecule, the benzimidazole groups are coplanar. In this instance, owing to a slight twist about the central ethene fragment, the planes are separated by

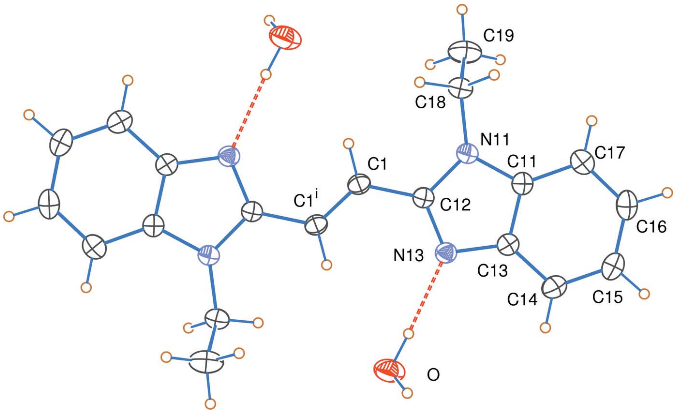


Figure 1
The structure of (II)·2H_2O, showing 25% probability displacement ellipsoids and the atom-numbering scheme. Hydrogen bonds are shown as dashed lines. Symmetry code (i) is as in Table 1.

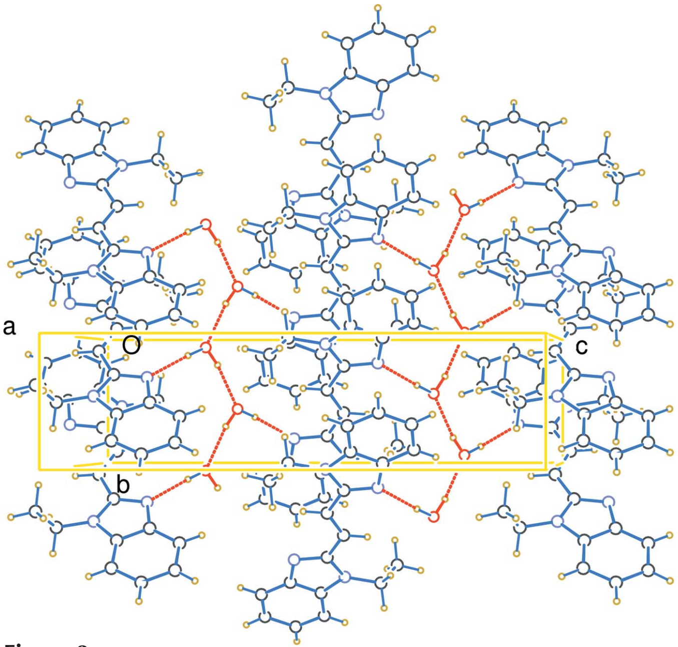


Figure 2
View, approximately perpendicular to (100), of the structure. Hydrogen bonds are shown as dashed lines.

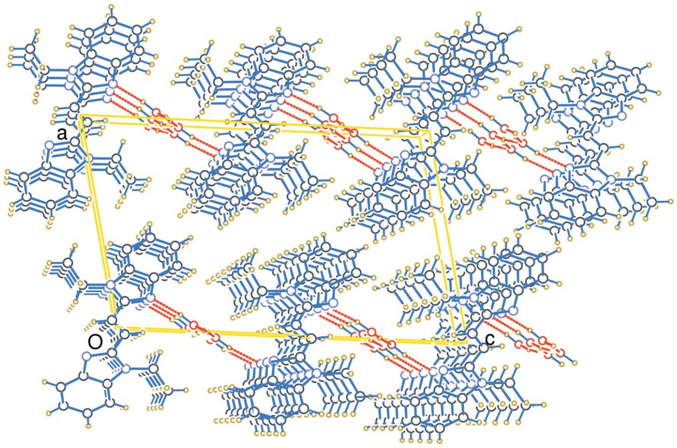


Figure 3
View, along the *b* axis, of the structure. Hydrogen bonds are shown as dashed lines.

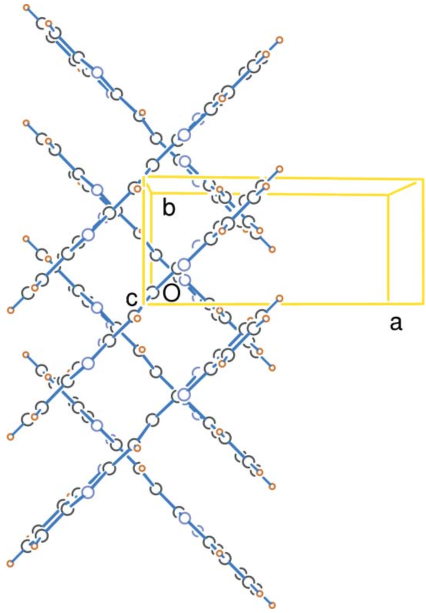


Figure 4
View, along the *c* axis, showing two columns of molecules of (II) in profile.

0.131 (4) Å, a value significantly smaller than the value of 0.211 (4) Å reported for neat (II), which is also centrosymmetric. The geometric parameters of molecule (II) in the dihydrate (Table 1) compare favorably to those reported for the unsolvated species (Stibrany *et al.*, 2005*b*), as well as those reported for a related bis(*N*-methylbenzimidazole) species (Morkovnik *et al.*, 1995). In particular, in these three species, the C12—N_{imine} distances are significantly shorter than the C12—N_{amine} distances, as expected, and the N13—C13—C11 angles are significantly larger than the N11—C11—C13 angles.

In the crystal structure, each water molecule forms two strong hydrogen bonds (Table 2), one to a second water molecule related to the first by the screw axis symmetry operation of the space group, and the second to the N_{imine} atom of a benzimidazole fragment. The former bonds lead to a chain of hydrogen-bonded water molecules parallel to [010], whilst the latter link columns of molecules (II) to these chains (Fig. 2). The result is a structure which, when viewed along the *b* axis, appears as two-dimensional layers parallel to the (100) planes (Fig. 3). Each layer contains alternating columns of molecules (II) and water molecules, which are linked along the *b* axis by O—H···O hydrogen bonds and along the *c* axis by O—H—N_{imine} hydrogen bonds to form a two-dimensional polymeric network.

In profile, columns of (II) along the *b* axis appear as slipped decks of equally spaced cards (Fig. 4). Adjacent columns are related to each other by the *c*-glide plane symmetry operations. As a result, the columns contain molecules alternately canted to the left and to the right, which appear as criss-crossed arrays in profile, with the benzimidazole planes in adjacent columns approximately perpendicular to each other. The interplanar spacing of 3.413 (3) Å between the benzimidazole planes in a given column is consistent with π–π interactions (Janiak, 2000).

Experimental

The synthesis of (*E*)-1,2-bis(1-ethylbenzimidazol-2-yl)ethene has been reported previously (Stibrany *et al.*, 2005*b*). The dihydrate reported here was obtained by the slow evaporation of an ethanol solution of (II) in the absence of triethyl orthoformate, which acted as a dessicant for the preparation of crystals of neat (II).

Crystal data

$C_{20}H_{20}N_4 \cdot 2H_2O$
$M_r = 352.43$
Monoclinic, $P2_1/c$
$a = 11.0314$ (8) Å
$b = 4.8234$ (3) Å
$c = 17.8657$ (15) Å
$\beta = 102.498$ (6)°
$V = 928.09$ (12) Å^3
$Z = 2$

$D_x = 1.261$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 772 reflections
$\theta = 2.3–18.7°$
$\mu = 0.08$ mm^{-1}
$T = 297$ (1) K
Rod, pale yellow
$0.48 \times 0.09 \times 0.04$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Blessing, 1995)
$T_{min} = 0.68$, $T_{max} = 1.00$
6786 measured reflections

1629 independent reflections
1071 reflections with $I > 2\sigma(I)$
$R_{int} = 0.069$
$\theta_{max} = 25.2°$
$h = -13 \rightarrow 13$
$k = -5 \rightarrow 5$
$l = -18 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.138$
$S = 1.00$
1629 reflections
150 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0696P)^2 + 0.0554P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.14$ e Å^{-3}
$\Delta\rho_{min} = -0.12$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—C1^{i}	1.323 (5)	N11—C11	1.389 (3)
C1—C12	1.449 (3)	N13—C13	1.384 (3)
N11—C12	1.371 (3)	C11—C13	1.387 (3)
N13—C12	1.325 (3)		
C1^{i}—C1—C12	124.0 (3)	C12—N13—C13	104.70 (19)
N11—C12—C1	122.7 (2)	C13—C11—N11	105.92 (19)
N13—C12—C1	124.2 (2)	N13—C13—C11	110.4 (2)
N13—C12—N11	113.11 (19)	N11—C11—C17	131.5 (2)
C12—N11—C11	105.90 (18)	N13—C13—C14	129.8 (2)

Symmetry code: (i) $-x, -y, -z$.

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

D—H···A	D—H	H···A	D···A	D—H···A
O—H1O···N13	0.92 (3)	1.98 (3)	2.892 (3)	171 (3)
O—H2O···O^{ii}	0.87 (4)	1.92 (4)	2.779 (4)	166 (3)

Symmetry code: (ii) $-x, y + \frac{1}{2}, -z + \frac{1}{2}$.

The water H atoms, which are involved in hydrogen bonding, the ethene H atom, and both the methyl and the methylene H atoms of the ethyl fragment were refined isotropically (C—H = 0.94 (3)–1.02 (5) Å). C-bound H atoms of the benzimidazole fragment were positioned geometrically and treated as riding, with Csp^2—H distances set to 0.93 Å, and with $U_{iso}(H) = 1.2\ U_{eq}(C)$.

Data collection: *SMART WNT/2000* (Bruker, 2000); cell refinement: *SAINT-Plus* (Bruker, 2000); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-32* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL* (Bruker, 2000).

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