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## Key indicators

Single-crystal X-ray study  
 $T = 100$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å  
 $R$  factor = 0.041  
 $wR$  factor = 0.106  
Data-to-parameter ratio = 11.9For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.*trans*-1,2-Bis(1-methylbenzimidazol-2-yl)-  
cyclohexane monohydrate

Chiral molecules of the title compound,  $\text{C}_{22}\text{H}_{24}\text{N}_4 \cdot \text{H}_2\text{O}$ , have approximate twofold symmetry, essentially planar benzimidazole fragments, and a ladder-like appearance in profile, with the benzimidazole planes exhibiting a dihedral angle of  $68.75(4)^\circ$ . Geometric parameters are consistent with those of comparable species containing benzimidazole or cyclohexyl fragments. Adventitious water molecules of solvation form strong  $\text{O}-\text{H} \cdots \text{N}(\text{imine})$  hydrogen bonds to yield infinite  $2_1$  columns along the  $c$ -axis direction. The columns are related by glide-plane symmetry operations and are linked by  $\text{C}-\text{H} \cdots \pi$  interactions to yield racemic crystals.

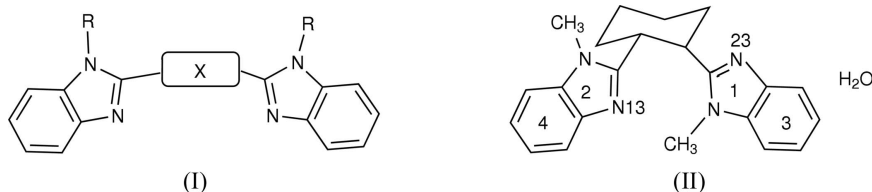
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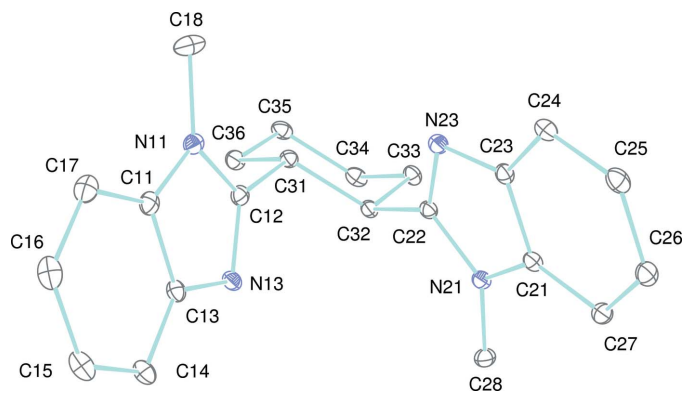
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## Comment

Bis(2-benzimidazol-2-yl) species, (I), bridged by a variety of alkyl or aryl spacers,  $X$ , are remarkably versatile. They have, for example, been tested as anti-tumour and antimicrobial agents (Baraldi *et al.*, 2004), and as topoisomerase inhibitors (Walker & Saravia, 2004). Furthermore, they have been used as polymerization catalysts (Stibrany, 2001), as agents to study electron self-exchange (Knapp *et al.*, 1990; Xie *et al.*, 1999), in selective ion-exchange resins (van Berkel *et al.*, 1995), as proton sponges (Stibrany *et al.*, 2002), and as geometrically constraining ligands (Stibrany *et al.*, 2004). Here, we report the structure of the title compound, (II), a bis(benzimidazole) ligand with a rigid two-carbon bridge and two torsional degrees of freedom.



Molecules of (II) (Fig. 1) exhibit approximate diad axes, which pass through the midpoints of the C31–C32 and C34–C35 bonds of the cyclohexyl fragments. Both benzimidazole fragments are essentially planar, as expected, and they are oriented in such a way as to give the molecule a ladder-like appearance in profile (Fig. 2). The dihedral angle between the benzimidazole planes [ $68.75(4)^\circ$ ] is approximately  $5^\circ$  larger than that reported for bis(benzimidazole-2-yl)methane [ $63.53(2)^\circ$ ; Duan *et al.*, 2005], a molecule related to (II) but with  $R = \text{H}$  and with a one-carbon atom bridge  $X = \text{CH}_2$ . Geometric parameters for (II) (Table 1) agree well with those reported for other structures of bis(benzimidazole)s with rigid two-carbon bridges (Stibrany *et al.*, 2005). In particular, in the  $\text{N}-\text{C}-\text{N}$  fragments, the  $\text{C}-\text{N}(\text{imine})$  distances are approxi-



**Figure 1**

The molecular structure of (II), showing 25% probability displacement ellipsoids and the atom-numbering scheme. H atoms and the solvent water molecule have been omitted for clarity.

mately 0.05 Å shorter than the C–N(amine) distances, consonant with the partial double-bond character of the former bonds.

In the crystal structure of (II), water molecules of solvation form strong O–H···N(imine) hydrogen bonds (Table 2) with the organic molecules to yield infinite chains or columns parallel to the *c* axis direction, in which successive molecules of a given type are related by the  $2_1$  symmetry operations of the space group (Fig. 3). The organic molecules of (II) are chiral and, as a result, individual ' $2_1$ ' columns contain one enantiomer exclusively. The glide-plane symmetry operations produce parallel columns containing the other enantiomer exclusively and, as a result, crystals of (II) are racemic. To complete the structure, the columns are linked by C–H··· $\pi$  interactions, some of which are shown in Table 3. There is little, if any, evidence for  $\pi$ – $\pi$  stacking.

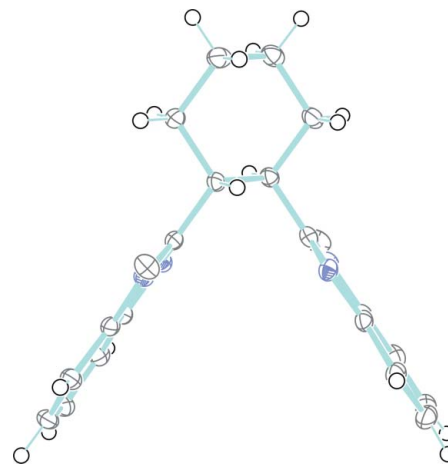
## Experimental

*trans*-1,2-Bis(1-methylbenzimidazol-2-yl)cyclohexane hydrate was prepared in two steps. Firstly, *trans*(1,2-bis(1*H*-benzimidazol-2-yl)) was prepared by the Phillips condensation method (Phillips, 1931) from *trans*-1,2-cyclohexane dicarboxylic acid and two equivalents of 1,2-phenylenediamine. Secondly, using the procedure described previously by Stibrany *et al.* (2004), methylation of the amine N atoms, effected using methyl iodide, yielded the non-hydrated product. Crystals of the title compound, (II), were obtained by slow evaporation in air of an acetone solution of the product. Incorporation of adventitious water resulted in the monohydrate [m.p. 479 K (soften), 491 K (melt)]. Spectroscopic analysis: IR (KBr pellet,  $\text{cm}^{-1}$ ): 3384 (*m*), 3051 (*w*), 2929 (*s*), 2853 (*m*), 1614 (*w*), 1468 (*s*), 1443 (*s*), 1329 (*m*), 1008 (*w*), 745 (*s*).

### Crystal data

$\text{C}_{22}\text{H}_{24}\text{N}_4\cdot\text{H}_2\text{O}$   
 $M_r = 362.47$   
 Orthorhombic,  $Pna2_1$   
 $a = 9.6596$  (7) Å  
 $b = 14.555$  (1) Å  
 $c = 13.547$  (1) Å  
 $V = 1904.6$  (2) Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.264$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation  
 Cell parameters from 6763 reflections  
 $\theta = 2.5$ – $30.6^\circ$   
 $\mu = 0.08$  mm<sup>-1</sup>  
 $T = 100$  (2) K  
 Plate, colourless  
 $0.29 \times 0.14 \times 0.03$  mm



**Figure 2**

A view of the organic molecule of (II), showing the benzimidazole planes in profile.

### Data collection

Bruker SMART CCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Bruker, 2000; Blessing, 1995)  
 $T_{\min} = 0.831$ ,  $T_{\max} = 1.00$   
 14429 measured reflections

3030 independent reflections  
 2785 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.038$   
 $\theta_{\text{max}} = 30.6^\circ$   
 $h = -12 \rightarrow 13$   
 $k = -10 \rightarrow 20$   
 $l = -19 \rightarrow 14$

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.041$   
 $wR(F^2) = 0.106$   
 $S = 1.00$   
 3030 reflections  
 254 parameters  
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.071P)^2 + 0.2238P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.37$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.16$  e Å<sup>-3</sup>

**Table 1**

Selected geometric parameters (Å, °).

N11–C12	1.374 (2)	N23–C23	1.391 (2)
N21–C22	1.366 (2)	C11–C13	1.402 (3)
N13–C12	1.321 (3)	C21–C23	1.403 (3)
N23–C22	1.323 (2)	C12–C31	1.495 (2)
N11–C11	1.384 (3)	C22–C32	1.505 (2)
N21–C21	1.385 (2)	C31–C32	1.536 (2)
N13–C13	1.388 (2)		
N13–C12–N11	112.60 (16)	C22–N23–C23	104.61 (15)
N23–C22–N21	113.36 (16)	N11–C11–C13	105.36 (16)
C22–N11–C11	106.85 (15)	N21–C21–C23	105.38 (15)
C22–N21–C21	106.60 (15)	N13–C13–C11	109.91 (16)
C12–N13–C13	105.27 (15)	N23–C23–C21	110.04 (16)

**Table 2**

Hydrogen-bond geometry (Å, °).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
O1–H1O···N13 <sup>i</sup>	0.85 (3)	2.12 (3)	2.955 (2)	171 (2)
O1–H2O···N23 <sup>ii</sup>	0.85 (3)	2.14 (3)	2.983 (2)	170 (2)

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

**Table 3**

Intermolecular C—H...Cgn interactions in (II) (Å, °) with H...Cgn distances less than 3.0 Å. The ring centroid designations n correspond to the numbers given in the scheme.

C—H...Cgn	H...Cgn	C—H...Cgn	C...Cgn
C25—H25...Cg1 <sup>i</sup>	2.98	141	3.749
C26—H26...Cg3 <sup>i</sup>	2.88	156	3.748
C28—H28B...Cg1 <sup>ii</sup>	2.98	154	3.864
C28—H28C...Cg4 <sup>ii</sup>	2.91	124	3.534

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

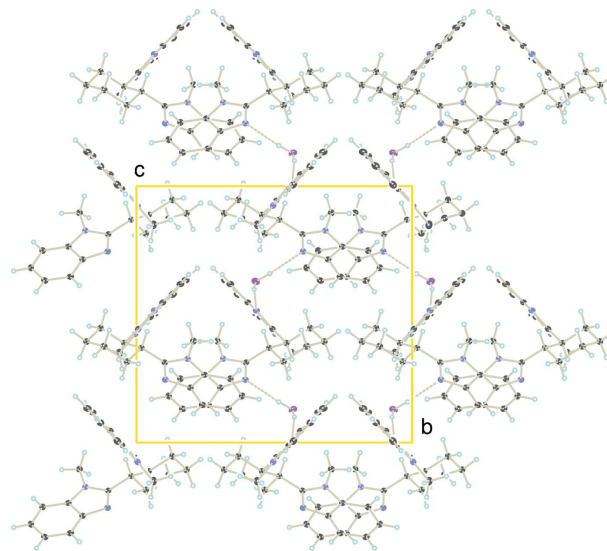
The water H atoms, which are involved in hydrogen bonding, were refined isotropically. C-bound H atoms were positioned geometrically and treated as riding, with  $Csp^2-H$  and  $Csp^3-H$  distances set to 0.93 and 0.96 Å, respectively, and with  $U_{iso}(H) = 1.2U_{eq}(C)$  and  $1.5U_{eq}(C)$ , respectively. A final difference Fourier map revealed 37 peaks larger than the absolute value of the largest negative peak. Of these, 33 of the first 35 peaks were located approximately midway along the C—C or C—N vectors, of which there are a total of 30 in the molecular framework. The 33 peaks are consistent with residual bond density, with three of the bonds exhibiting split peaks.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT-Plus* (Bruker, 2000); data reduction: *SAINT-Plus* (Bruker, 2000); 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 *ORTEP32* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL* (Bruker, 2000).

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**Figure 3**  
 A projection, along the *a* axis, of the structure of (II). O atoms are coloured purple and hydrogen bonds are indicated by dashed lines.

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