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Redetermination of bis(benzimidazol-2-ylmethyl)amine

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The title compound, $C_{16}H_{15}N_5$, has been redetermined from data collected at 150 K. It crystallizes in space group *Pbca*, with four molecules in the unit cell, and the molecule is disordered about a centre of symmetry.

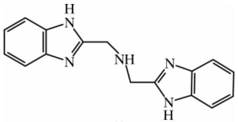
Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
Disorder in main residue
R factor = 0.034
wR factor = 0.084
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.

Comment

A room-temperature structure determination of bis(benzimidazol-2-ylmethyl)amine, (I), has been reported previously (Calderazzo *et al.*, 2003). These authors observed that the asymmetric unit contains half of the molecule and the saturated portion is disordered about a centre of symmetry. The disorder was modelled by refining the amine N atom over two positions and restraining the C—N distances to be approximately equal.



(I)

The present data collection was carried out at 150 K, and the disorder model was extended to include the saturated carbon (C8). Using this model, the refinement improved considerably and no bond-length restraints were required. Fig. 1 shows one component of the disorder, while both are shown in Fig. 2; the amine N atom has 50% occupancy of sites N3 and

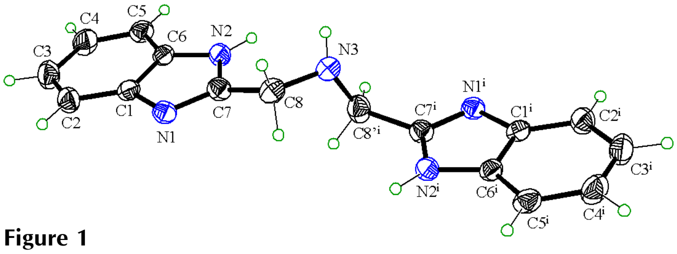


Figure 1
A perspective view of one molecule, showing 50% probability displacement ellipsoids. Only one of the two disordered components of the saturated portion of the molecule is shown. [Symmetry code: (i) $1 - x$, $-y$, $-z$].

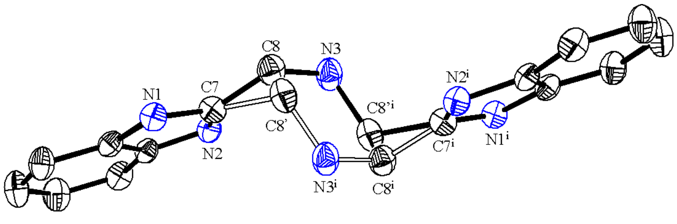


Figure 2
Perspective view, showing both components of the disorder about the centre of symmetry (50% probability displacement ellipsoids).

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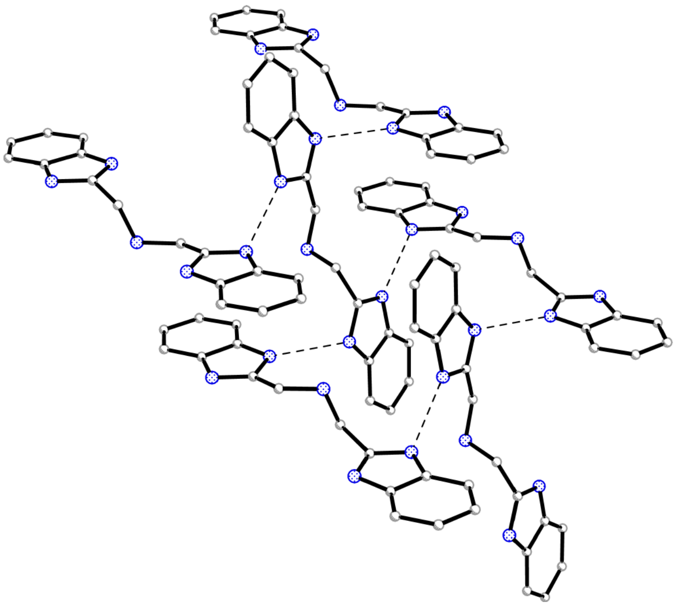

Figure 3
Hydrogen-bonding interactions between the bis(benzimidazol-2-ylmethyl)amine molecules.

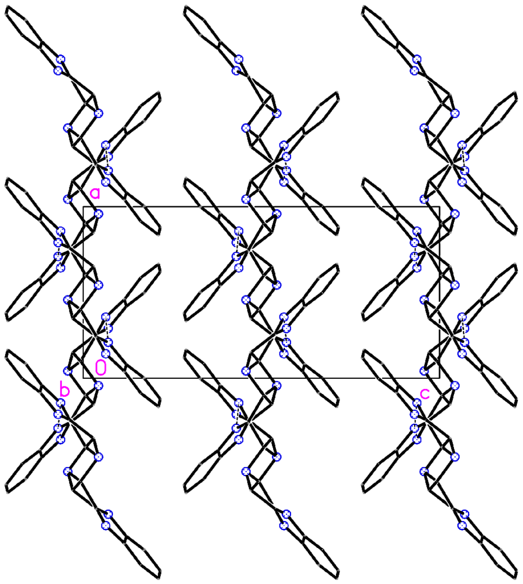

Figure 4
The packing, projected perpendicular to *b*, including both disorder components.

N3^i, which are related by a centre of symmetry [symmetry code: (i) $1 - x, -y, -z$]. The saturated C atom is disordered equally over sites C8 and C8′ and these give rise to the symmetry-related sites C8^i and C8$'^i$, respectively.

The two benzimidazole rings are parallel, but the molecule is stepped so the planes are *ca.* 1.67 Å apart. The amine is not involved in any hydrogen bonding but benzimidazole NH groups are each linked to the imine N atom of an adjacent molecule (Table 1). The resulting network consists of stepped, hydrogen-bonded layers perpendicular to *c*, as shown in Figs. 3 and 4. This arrangement also permits π–π stacking within layers (Fig. 4), whereby parallel pairs of benzimidazole rings are stacked 3.65 Å apart, and edge-to-face interactions exist between the layers.

The C7—N1 and C7—N2 bond lengths are 1.318 (2) and 1.357 (2) Å, respectively, suggesting partial delocalization of the N—C—N section of the ring. This is consistent with the observed hydrogen-bonding pattern.

Experimental

The title bisbenzimidazole was prepared from the condensation of iminodiacetic acid and 1,2-diaminobenzene, as described in the literature (Adams *et al.*, 1990). Colourless crystals were obtained by recrystallization from methanol.

Crystal data

$C_{16}H_{15}N_5$
$M_r = 277.33$
Orthorhombic, *Pbca*
$a = 8.2512$ (8) Å
$b = 9.5494$ (9) Å
$c = 17.1248$ (17) Å
$V = 1349.3$ (2) Å^3
$Z = 4$
$D_x = 1.365$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3898 reflections
$\theta = 2.4$–23.6°
$\mu = 0.09$ mm^{-1}
$T = 150$ (2) K
Lozenge, colourless
0.39 × 0.10 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000)
$T_{\min} = 0.990$, $T_{\max} = 0.991$
8858 measured reflections
1188 independent reflections
1041 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.023$
$\theta_{\max} = 25.0°$
$h = -9 \rightarrow 9$
$k = -11 \rightarrow 11$
$l = -19 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.084$
$S = 1.06$
1188 reflections
109 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0345P)^2 + 0.574P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.16$ e Å^{-3}
$\Delta\rho_{\min} = -0.21$ e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2N···N1^{ii}	0.92	1.97	2.8816 (15)	169

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

H atoms bonded to C were placed at calculated positions, with C—H distances of 0.95 and 0.98 Å for H atoms bonded to sp^2 and methylene C atoms, respectively. They were refined using a riding model, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$. H atoms bonded to N atoms were located from difference maps, assigned a fixed $U_{\rm iso} = 0.05$, and not further refined.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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