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Bis(benzimidazol-2-yl)methane

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.042
wR factor = 0.130
Data-to-parameter ratio = 13.4

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

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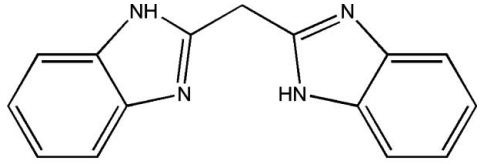
In the title molecule, $C_{15}H_{12}N_4$, the two essentially planar benzimidazolyl moieties make a dihedral angle of 63.53 (2)°. Intermolecular N—H···N hydrogen bonds stabilize the crystal packing.

Comment

Bis(benzimidazol-2-yl)methane and its derivatives are the subject of intensive study because they exhibit antimicrobial activities (Agh-Atabay *et al.*, 2003), execute control of liver diseases (Kotomo *et al.*, 1992), and may serve as inhibitors of cell death (Bitler *et al.*, 2000) and HCV NS3 serine protease (Yeung *et al.*, 2001). They are also employed as ligands (Gupta *et al.*, 2001). In this paper, we report the crystal structure of the title compound, (I) (Fig. 1).



(I)

In (I), the bond lengths and angles of the benzimidazole moieties (Table 1) are in agreement with published values (Eryigit & Kendi, 1998; Chen *et al.*, 2002). The benzimidazolyl moieties C1–C7/N1/N2 and C9–C15/N3/N4 are each essentially planar, making a dihedral angle of 63.53 (2)°.

The crystal packing of (I) (Fig. 2) is stabilized by intermolecular N—H···N hydrogen-bond interactions (Table 2).

Experimental

The title compound can be synthesized from 1,2-phenylenediamine and malonic acid in ethylene glycol as solvent at reflux for 24 h (Lane,

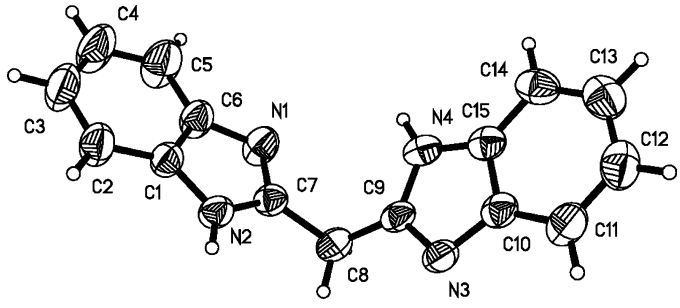


Figure 1
A view of (I), with displacement ellipsoids drawn at the 40% probability level.

1953), in a yield of 56%, or in PPA (poly phosphorous acid) as solvent at 453 K for 2.5–4 h (Vyas *et al.*, 1980), in a yield of 85%. However, we used 1,2-phenylenediamine (0.02 mol) and malonamide (0.01 mol) at 453–463 K under solvent-free conditions for 1 h, providing a convenient protocol for the preparation of this class of heterocycles. Purification was achieved by recrystallization from methanol in 92% isolated yield. Crystals of (I) suitable for X-ray diffraction analysis were obtained by slow evaporation of a methanol solution at room temperature over two weeks.

Crystal data

$C_{15}H_{12}N_4$
M_r = 248.29
Tetragonal, $I4_1/a$
a = 18.296 (4) Å
c = 15.728 (3) Å
V = 5264.6 (18) Å^3
Z = 16
D_x = 1.253 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4441 reflections
θ = 2.2–25.8°
μ = 0.08 mm^{-1}
T = 298 (2) K
Block, orange
0.40 × 0.31 × 0.27 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.950, T_{max} = 0.979
10662 measured reflections
2307 independent reflections
1798 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 25.1°
$h = -15 \rightarrow 21$
$k = -17 \rightarrow 21$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.130
S = 1.00
2307 reflections
172 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.075P)^2 + 1.5659P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C7	1.312 (2)	N3—C10	1.388 (2)
N1—C6	1.391 (2)	N4—C9	1.340 (2)
N2—C7	1.333 (2)	N4—C15	1.381 (2)
N2—C1	1.370 (2)	C7—C8	1.491 (2)
N3—C9	1.315 (2)	C8—C9	1.493 (2)
C7—N1—C6	105.43 (14)	C9—N4—C15	106.79 (13)
C7—N2—C1	106.98 (13)	C7—C8—C9	114.19 (14)
C9—N3—C10	105.81 (13)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···N3^{i}	0.86	1.94	2.7869 (18)	169
N4—H4A···N1^{ii}	0.86	2.00	2.823 (2)	160

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

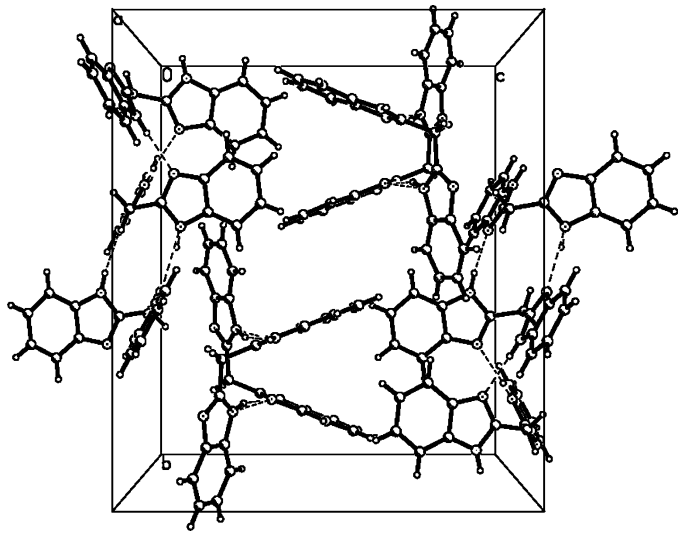


Figure 2
A packing diagram for the title compound. The intermolecular N—H···N hydrogen bonds are shown by dashed lines.

All H atoms were placed in calculated positions, with C—H = 0.93–0.97 Å and N—H = 0.86 Å, and were included in the final cycles of refinement using a riding model, with $U_{iso}(H) = 1.2U_{eq}$ of the parent atom.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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