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Dimethyl 2,2′-[2,2′-bi(1*H*-1,3-benzimidazole)-1,1′-diyl]diacetate

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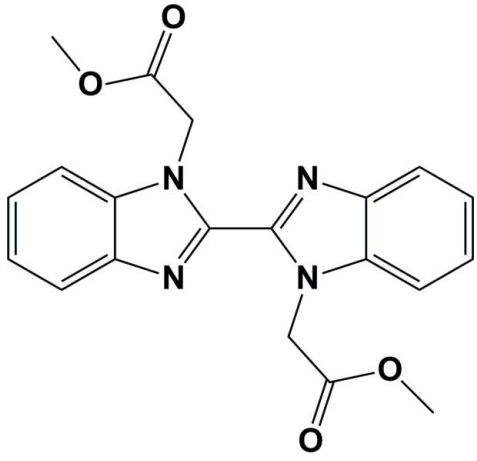
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.048; wR factor = 0.127; data-to-parameter ratio = 12.5.

The whole molecule of the title compound, $C_{20}H_{18}N_4O_4$, is generated by an inversion center. The benzimidazole ring mean plane make a dihedral angle of 89.4 (8)° with the plane passing through the acetate group (COO). In the crystal, molecules are linked *via* weak C—H···O hydrogen bonds and π–π interactions [centroid–centroid distance = 3.743 (3) Å] involving inversion-related benzimidazole groups.

Related literature

For related structures, see: Al-Mohammed *et al.* (2012); Fu & Xu (2009); Xu & Wang (2008). For the synthesis of 2,2′-bibenzimidazole, see: Tang *et al.* (2007).



Experimental

Crystal data

$C_{20}H_{18}N_4O_4$
M_r = 378.38
Triclinic, $P\overline{1}$
a = 6.904 (4) Å
b = 8.494 (5) Å
c = 8.643 (5) Å
α = 67.191 (5)°
β = 70.360 (5)°
γ = 87.172 (5)°
V = 438.1 (4) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 296 K
0.33 × 0.31 × 0.29 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.967, T_{max} = 0.971
3034 measured reflections
1600 independent reflections
1172 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.127
S = 1.06
1600 reflections
128 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8B···O1^{i}	0.97	2.59	3.364 (3)	137
C10—H10C···O1^{ii}	0.96	2.55	3.481 (4)	164

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2479).

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supporting information

Acta Cryst. (2012). E**68**, o2512 [https://doi.org/10.1107/S1600536812032266]

Dimethyl 2,2′-[2,2′-bi(1*H*-1,3-benzimidazole)-1,1′-diyl]diacetate

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S1. Comment

The whole molecule of the title compound (Fig.1) is generated by an inversion center. The benzimidazole system is essentially planar, with a dihedral angle of 0.8 (5)° between the planes of the benzene and imidazole rings. The benzimidazole ring make a dihedral angle of 89.4 (8)° with the plane passing through the acetate group (C9/O1/O2). This value is comparable to that observed in some similar structures (Al-Mohammed *et al.*, 2012; Fu *et al.*, 2009; Xu *et al.*, 2008).

In the crystal, weak intermolecular C—H···O hydrogen bonds (Table 1 and Fig. 2) and π–π stacking interactions [Cg1···Cg2^{i} = 3.743 (3) Å, where Cg1 is the centroid of ring N1/C1/C6/N2/C7; Cg2 is the centroid of ring C1-C6; symmetry code: (i) -x+1, -y+1, -z+1] stabilize the crystal structure.

S2. Experimental

The synthesis of 2,2′-bibenzimidazole [systematic name: 1*H*,1′*H*-2,2′-bibenzo[d]imidazole] has been reported (Tang *et al.*, 2007). A mixture of 2,2′-bibenzimidazole (11.71 g, 50 mmol) and NaOH (4.00 g, 100 mmol) in DMSO (40 mL) was stirred at 278 K for 2 h, and then methyl chloroacetate (10.85 g, 100 mmol) was added. The mixture was cooled to room temperature after stirring at 353 K for 24 h, and then poured into 200 mL of water. A yellow solid formed immediately, which was isolated by filtration. The crude product was then crystallized from methanol. Single crystals of the title compound, suitable for X-ray analysis, were obtained by slow evaporation of a solution in methanol.

S3. Refinement

The C-bound H-atoms were included in calculated positions and treated as riding atoms: C-H = 0.93, 0.96 and 0.97 Å for CH, CH_3 and CH_2 H-atoms, respectively, with U_{iso}(H) = k × U_{eq}(parent C-atom), where k = 1.5 for CH_3 H-atoms and = 1.2 for other H-atoms.

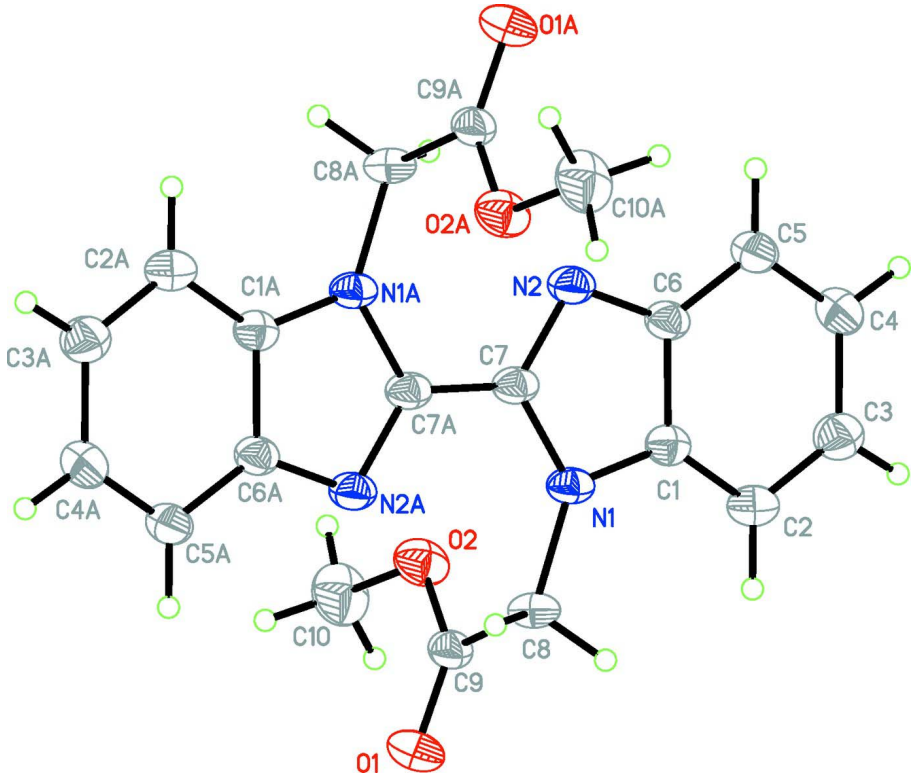

Figure 1

The molecular structure of the title molecule with the atom numbering [symmetry code for suffix A: -*x*, -*y*+1, -*z*+1]. Displacement ellipsoids are drawn at the 30% probability level

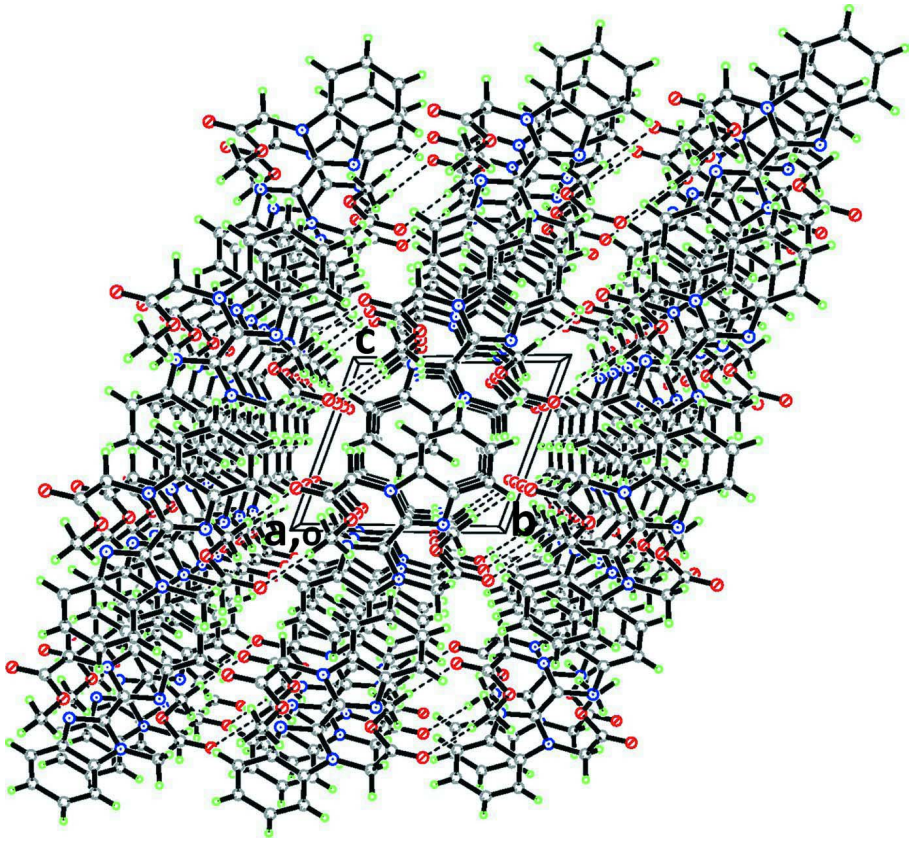

Figure 2

The crystal packing of the title compound, viewed along the *b* axis. Weak C—H···O interaction are shown as dashed lines.

Dimethyl 2,2′-[2,2′-bi(1*H*-1,3-benzimidazole)-1,1′-diyl]diacetate

Crystal data

$C_{20}H_{18}N_4O_4$
$M_r = 378.38$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 6.904$ (4) Å
$b = 8.494$ (5) Å
$c = 8.643$ (5) Å
$\alpha = 67.191$ (5)°
$\beta = 70.360$ (5)°
$\gamma = 87.172$ (5)°
$V = 438.1$ (4) Å^3
$Z = 1$
$F(000) = 198$
$D_x = 1.434$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 853 reflections
$\theta = 2.6$–$23.8°$
$\mu = 0.10$ mm^{-1}
$T = 296$ K
Block, brown
0.33 × 0.31 × 0.29 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.967$, $T_{max} = 0.971$
3034 measured reflections
1600 independent reflections
1172 reflections with $I > 2\sigma(I)$

R_{int} = 0.027
θ_{max} = 25.5°, θ_{min} = 2.6°
h = −8→8
k = −9→10
l = −10→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.127
S = 1.06
1600 reflections
128 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0656P)^2 + 0.0023P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
C1	0.3492 (3)	0.6658 (2)	0.5117 (2)	0.0382 (5)
C2	0.5275 (3)	0.7663 (3)	0.4646 (3)	0.0485 (6)
H2	0.6033	0.8332	0.3460	0.058*
C3	0.5865 (4)	0.7619 (3)	0.6022 (3)	0.0539 (6)
H3	0.7063	0.8271	0.5762	0.065*
C4	0.4722 (4)	0.6624 (3)	0.7808 (3)	0.0533 (6)
H4	0.5173	0.6635	0.8704	0.064*
C5	0.2955 (3)	0.5636 (3)	0.8265 (3)	0.0497 (6)
H5	0.2197	0.4979	0.9454	0.060*
C6	0.2327 (3)	0.5645 (2)	0.6892 (2)	0.0397 (5)
C7	0.0742 (3)	0.5291 (2)	0.5281 (2)	0.0385 (5)
C8	0.3089 (3)	0.7314 (2)	0.2179 (2)	0.0433 (5)
H8A	0.2674	0.6583	0.1696	0.052*
H8B	0.4585	0.7504	0.1691	0.052*
C9	0.2220 (3)	0.9010 (3)	0.1547 (3)	0.0435 (5)
C10	0.0081 (4)	1.1015 (3)	0.2334 (4)	0.0720 (8)
H10A	0.1162	1.1933	0.1612	0.108*
H10B	−0.0752	1.1195	0.3382	0.108*
H10C	−0.0764	1.0989	0.1659	0.108*
N1	0.2463 (2)	0.64117 (18)	0.41008 (19)	0.0388 (4)
N2	0.0622 (3)	0.4797 (2)	0.6961 (2)	0.0435 (5)
O1	0.2651 (3)	0.9897 (2)	−0.00051 (19)	0.0731 (6)

O2	0.0980 (2)	0.94015 (17)	0.28611 (18)	0.0535 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0432 (12)	0.0319 (10)	0.0357 (10)	0.0078 (9)	−0.0109 (9)	−0.0122 (8)
C2	0.0521 (14)	0.0445 (12)	0.0380 (11)	−0.0018 (10)	−0.0083 (10)	−0.0104 (9)
C3	0.0519 (14)	0.0543 (13)	0.0493 (13)	−0.0041 (11)	−0.0148 (11)	−0.0153 (11)
C4	0.0571 (15)	0.0605 (14)	0.0434 (12)	0.0081 (12)	−0.0224 (11)	−0.0176 (11)
C5	0.0498 (14)	0.0542 (13)	0.0354 (11)	0.0099 (11)	−0.0134 (10)	−0.0094 (9)
C6	0.0429 (13)	0.0351 (10)	0.0329 (10)	0.0077 (9)	−0.0102 (9)	−0.0079 (8)
C7	0.0420 (12)	0.0299 (10)	0.0321 (10)	0.0035 (9)	−0.0063 (8)	−0.0059 (8)
C8	0.0497 (13)	0.0421 (11)	0.0280 (10)	−0.0052 (10)	−0.0030 (9)	−0.0110 (8)
C9	0.0431 (13)	0.0446 (12)	0.0335 (10)	−0.0084 (9)	−0.0131 (9)	−0.0044 (9)
C10	0.082 (2)	0.0443 (13)	0.0862 (18)	0.0167 (13)	−0.0383 (16)	−0.0158 (13)
N1	0.0442 (10)	0.0304 (8)	0.0302 (8)	0.0015 (7)	−0.0059 (7)	−0.0058 (7)
N2	0.0454 (11)	0.0396 (9)	0.0321 (9)	0.0036 (8)	−0.0079 (8)	−0.0049 (7)
O1	0.0739 (12)	0.0779 (12)	0.0375 (9)	0.0000 (10)	−0.0172 (8)	0.0073 (8)
O2	0.0684 (11)	0.0384 (8)	0.0470 (9)	0.0120 (7)	−0.0185 (8)	−0.0119 (7)

Geometric parameters (Å, º)

C1—N1	1.377 (3)	C7—N1	1.379 (2)
C1—C2	1.381 (3)	C7—C7^{i}	1.449 (4)
C1—C6	1.398 (3)	C8—N1	1.448 (2)
C2—C3	1.368 (3)	C8—C9	1.503 (3)
C2—H2	0.9300	C8—H8A	0.9700
C3—C4	1.398 (3)	C8—H8B	0.9700
C3—H3	0.9300	C9—O1	1.194 (2)
C4—C5	1.367 (3)	C9—O2	1.321 (2)
C4—H4	0.9300	C10—O2	1.447 (3)
C5—C6	1.391 (3)	C10—H10A	0.9600
C5—H5	0.9300	C10—H10B	0.9600
C6—N2	1.383 (3)	C10—H10C	0.9600
C7—N2	1.320 (2)		
N1—C1—C2	131.59 (18)	N1—C8—C9	115.16 (16)
N1—C1—C6	105.60 (18)	N1—C8—H8A	108.5
C2—C1—C6	122.81 (19)	C9—C8—H8A	108.5
C3—C2—C1	116.29 (19)	N1—C8—H8B	108.5
C3—C2—H2	121.9	C9—C8—H8B	108.5
C1—C2—H2	121.9	H8A—C8—H8B	107.5
C2—C3—C4	122.0 (2)	O1—C9—O2	124.6 (2)
C2—C3—H3	119.0	O1—C9—C8	121.8 (2)
C4—C3—H3	119.0	O2—C9—C8	113.58 (15)
C5—C4—C3	121.4 (2)	O2—C10—H10A	109.5
C5—C4—H4	119.3	O2—C10—H10B	109.5
C3—C4—H4	119.3	H10A—C10—H10B	109.5

C4—C5—C6	117.83 (19)	O2—C10—H10C	109.5
C4—C5—H5	121.1	H10A—C10—H10C	109.5
C6—C5—H5	121.1	H10B—C10—H10C	109.5
N2—C6—C5	130.18 (18)	C1—N1—C7	106.58 (15)
N2—C6—C1	110.13 (17)	C1—N1—C8	123.60 (16)
C5—C6—C1	119.7 (2)	C7—N1—C8	129.65 (17)
N2—C7—N1	112.52 (18)	C7—N2—C6	105.16 (16)
N2—C7—C7^{i}	124.2 (2)	C9—O2—C10	116.11 (17)
N1—C7—C7^{i}	123.3 (2)		

N1—C1—C2—C3	179.33 (19)	C2—C1—N1—C8	−3.2 (3)
C6—C1—C2—C3	0.1 (3)	C6—C1—N1—C8	176.12 (16)
C1—C2—C3—C4	−0.5 (3)	N2—C7—N1—C1	−0.7 (2)
C2—C3—C4—C5	0.3 (3)	C7^{i}—C7—N1—C1	−179.9 (2)
C3—C4—C5—C6	0.2 (3)	N2—C7—N1—C8	−176.09 (18)
C4—C5—C6—N2	−179.3 (2)	C7^{i}—C7—N1—C8	4.8 (3)
C4—C5—C6—C1	−0.5 (3)	C9—C8—N1—C1	−87.6 (2)
N1—C1—C6—N2	0.0 (2)	C9—C8—N1—C7	87.1 (2)
C2—C1—C6—N2	179.38 (19)	N1—C7—N2—C6	0.7 (2)
N1—C1—C6—C5	−179.02 (17)	C7^{i}—C7—N2—C6	179.9 (2)
C2—C1—C6—C5	0.4 (3)	C5—C6—N2—C7	178.5 (2)
N1—C8—C9—O1	178.39 (19)	C1—C6—N2—C7	−0.4 (2)
N1—C8—C9—O2	−1.1 (3)	O1—C9—O2—C10	1.1 (3)
C2—C1—N1—C7	−178.9 (2)	C8—C9—O2—C10	−179.49 (17)
C6—C1—N1—C7	0.42 (19)		

Symmetry code: (i) −*x*, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8*B*···O1^{ii}	0.97	2.59	3.364 (3)	137
C10—H10*C*···O1^{iii}	0.96	2.55	3.481 (4)	164

Symmetry codes: (ii) −*x*+1, −*y*+2, −*z*; (iii) −*x*, −*y*+2, −*z*.