Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1,3-Bis(2-hydroxybenzyl)-2,2-dimethyl-2,3-dihydro-1*H*-benzimidazole

Shu-Ping Yang,[a]* Da-Qi Wang,[b] Li-Jun Han[c] and Hai-Tao Xia[a]

[a]Department of Chemical Engineering, Huaihai Institute of Technology, Lianyungang 222005, People's Republic of China, [b]College of Chemistry and Chemical Engineering, Liaocheng University, Shandong 252059, People's Republic of China, and [c]Department of Mathematics and Science, Huaihai Institute of Technology, Lianyungang 222005, People's Republic of China
Correspondence e-mail: yangshuping@hhit.edu.cn

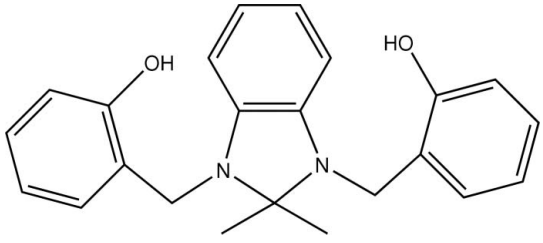
Received 13 August 2007; accepted 12 September 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.038; wR factor = 0.118; data-to-parameter ratio = 7.3.

The molecule of the title compound, $C_{23}H_{24}N_2O_2$, has a mirror plane passing through the benzimidazole ring system. The molecule adopts a bowed conformation and the two terminal benzene rings subtend a dihedral angle of 34.3 (1)°. Molecules are linked by C—H$\cdots\pi$ hydrogen bonds, forming an (010) sheet of $R_4^4(28)$ rings.

Related literature

For related literature, see: García-Báez *et al.* (2003); Ishida *et al.* (2006); Xu *et al.* (2006); Yang *et al.* (2007); Özden *et al.* (2005).



Experimental

Crystal data

$C_{23}H_{24}N_2O_2$
M_r = 360.44
Orthorhombic, $Pmn2_1$
a = 19.137 (2) Å
b = 8.0716 (15) Å
c = 6.1464 (13) Å
V = 949.4 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 298 (2) K
0.21 × 0.17 × 0.16 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan *SADABS* (Sheldrick, 1996)
T_{min} = 0.98, T_{max} = 0.99
4535 measured reflections
934 independent reflections
810 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.118
S = 1.19
934 reflections
128 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.11 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

*Cg*1 is the centroid of the C2–C7 ring.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1—H1$\cdots$N1	0.82	1.98	2.708 (4)	147
C4—H4$\cdots$*Cg*1^{i}	0.93	2.71	3.51 (2)	144

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

The authors are grateful to the Huaihai Institute of Technology Science Foundation for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2092).

References

García-Báez, E. V., Martínez-Martínez, F. J., Höpfl, H. & Padilla-Martínez, I. I. (2003). *Cryst. Growth Des.* **3**, 34–45.
Ishida, T., Suzuki, T., Hirashima, S., Mizutani, K., Yoshida, A., Ando, I., Ikeda, S., Adachi, T. & Hashimoto, H. (2006). *Bioorg. Med. Chem. Lett.* **16**, 1859–1863.
Özden, S., Atabey, D., Yildız, S. & Göker, H. (2005). *Bioorg. Med. Chem.* **13**, 1587–1597.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*a*). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Xu, Y.-J., Miao, H.-Q., Pan, W., Navarro, E. C., Tonra, J. R., Mitelman, S., Camara, M. M., Deevi, D. S., Kiselyov, A. S., Kussie, P., Wong, W. C. & Liu, H. (2006). *Bioorg. Med. Chem. Lett.* **16**, 404–408.
Yang, S.-P., Han, L.-J., Wang, D.-Q. & Ding, T.-Z. (2007). *Acta Cryst.* E**63**, o365–o367.

  doi:10.1107/S1600536807044601

supplementary materials

Acta Cryst. (2007). E**63**, o4088 [**doi:10.1107/S1600536807044601**]

1,3-Bis(2-hydroxybenzyl)-2,2-dimethyl-2,3-dihydro-1*H*-benzimidazole

S.-P. Yang, D.-Q. Wang, L.-J. Han and H.-T. Xia

Comment

Benzimidazole derivatives display wide-ranging biological activities, such as inhibitors of hepatitis C virus NS5B polymerase (Ishida *et al.*, 2006) and heparanase (Xu *et al.*, 2006) and antimicrobial activities (Özden *et al.*, 2005). The crystal structure of a benzimidazole derivative has been recently reported (Yang *et al.*, 2007). Here we report the crystal structure of a new benzimidazole derivative, 1,3-Bis(2-hydroxybenzyl) −2,2-dimethyl-2,3-dihydrogen 1*H*-benzimidazole,(I)·(Fig. 1)

The molecule presents a mirror symmetry plane through the central phenyl ring, and two intramolecular O—H···N hydrogen-bonds define two *S*(6) patterns (García-Báez *et al.*,2003). The molecule adopts a "hawk "conformation; the two benzyl rings located at both sides of the benzimidazole rings subtend a dihedral angle of 34.3 (1)°. (Table 1 and Fig.1). The geometric parameters for (I) are normal.

The molecules are linked by four C—H···π hydrogen bonds. The C4 atom in the molecules at (*x*,*y*,*z*) acts as hydrogen-bond donor to the $Cg1^{i}$ centroid of the ring C2—C7, i: 1/2 − *x*,1 − *y*,-1/2 + *z*], generating a [010] sheet of $R_4^4(28)$ rings by translation and reflection of the hydrogen-bond, the sheets lying in the domain 0.26 < *y* < 1.26. (Table 1 and Fig. 2)

Experimental

The reaction mixture containing salicylaldehyde (21 ml, 20 mmol) and *o*-phenylene diamine (1.08 g, 10 mmol) was refluxed for about 4 h in ethanol (30 ml), then sodium borohydride (1.52 g, 40 mmol) was added and the reaction mixture was refluxed continuely for 4 h, then acetone (20 ml) and water (40 ml) were added in turn, and the reaction mixture was cooled and the product filtered off, washed with ethanol and dried. Yellow crystals of (I) suitable for X-ray structure analysis were obtained by recrystallizing the crude product from ethanol (m.p.465–467 K).

Refinement

Due to the abscense of significant anomalous dispersion effects, Friedel pairs were merged, for what the number of independent reflections is rather low.

All H atoms were positioned geometrically and refined as riding on their parent atoms,with C—H = 0.96Å and U_{iso}(H)= 1.5U_{eq}(C) for methyl H atoms, O—H = 0.82Å and U_{iso}(H)= 1.2U_{eq}(C) for hydroxy H atoms, C—H = 0.97Å and U_{iso}(H)= 1.2U_{eq}(C) for methylene H atoms, and C—H = 0.93Å and U_{iso}(H) = 1.2U_{eq}(C) for all other H atom.

Figures

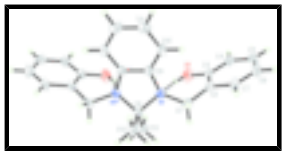


Fig. 1.
The molecular structure of (I), showing the atom-numbering scheme and intramolecular hydrogen-bonds *S*(6) pattern. Displacement ellipsoids are drawn at the 30% probability level. Dashed lines indicate hydrogen bonds·[Symmetry codes: (*)-*x*,*y*,*z*]

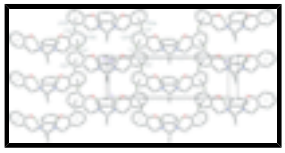


Fig. 2
Packing diagram of (I), showing the formation of a [010] sheet of $R_4^4(28)$ rings linked by a C—H···π hydrogen bond. For clarity, H atoms not involved in the motif shown have been omitted. Dashed lines indicate hydrogen bonds·[Symmetry codes: (*)-*x*,*y*,*z*; (&)-1/2 + *x*,1 − *y*,-1/2 + *z*; (&)-*x*,*y*,-1 + *z*; (#)1/2 − *x*,1 − *y*,-1/2 + *z*; (@)*x*,*y*,-1 + *z*].

1,3-Bis(2-hydroxybenzyl)-2,2-dimethyl-2,3-dihydro-1*H*-benzimidazole

Crystal data

$C_{23}H_{24}N_2O_2$	F_{000} = 384
M_r = 360.44	D_x = 1.261 Mg m^{-3}
Orthorhombic, $Pmn2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac -2	Cell parameters from 2085 reflections
a = 19.137 (2) Å	θ = 2.5–26.2º
b = 8.0716 (15) Å	μ = 0.08 mm^{-1}
c = 6.1464 (13) Å	*T* = 298 (2) K
V = 949.4 (3) Å^3	Block, yellow
Z = 2	0.21 × 0.17 × 0.16 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer	934 independent reflections
Radiation source: fine-focus sealed tube	810 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.032
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.1º
Absorption correction: multi-scan SADABS (Sheldrick, 1996)	*h* = −22→22
T_{min} = 0.98, T_{max} = 0.99	*k* = −7→9
4535 measured reflections	*l* = −7→7

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.038	$w = 1/[\sigma^2(F_o^2) + (0.0649P)^2 + 0.1011P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.118	$(\Delta/\sigma)_{max} < 0.001$

S = 1.19	$\Delta\rho_{max}$ = 0.11 e Å^{-3}
934 reflections	$\Delta\rho_{min}$ = −0.19 e Å^{-3}
128 parameters	Extinction correction: none
1 restraint	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.05992 (13)	0.7643 (3)	0.2677 (4)	0.0380 (7)
O1	0.11741 (12)	0.5707 (3)	−0.0445 (4)	0.0562 (7)
H1	0.0866	0.6049	0.0366	0.084*
C1	0.13090 (16)	0.7587 (4)	0.3599 (6)	0.0475 (9)
H1A	0.1343	0.6644	0.4572	0.057*
H1B	0.1387	0.8581	0.4453	0.057*
C2	0.18718 (17)	0.7456 (4)	0.1898 (6)	0.0415 (8)
C3	0.17888 (16)	0.6515 (4)	0.0007 (6)	0.0403 (8)
C4	0.23433 (18)	0.6332 (4)	−0.1444 (7)	0.0501 (9)
H4	0.2283	0.5712	−0.2706	0.060*
C5	0.2979 (2)	0.7061 (5)	−0.1026 (7)	0.0596 (11)
H5	0.3345	0.6926	−0.2004	0.072*
C6	0.3079 (2)	0.7990 (5)	0.0828 (9)	0.0641 (11)
H6	0.3510	0.8476	0.1113	0.077*
C7	0.25254 (17)	0.8190 (5)	0.2264 (7)	0.0505 (10)
H7	0.2590	0.8830	0.3506	0.061*
C8	0.0000	0.7094 (6)	0.4089 (7)	0.0388 (11)
C9	0.03658 (16)	0.9138 (4)	0.1698 (5)	0.0361 (7)
C10	0.07437 (17)	1.0423 (4)	0.0804 (6)	0.0430 (8)
H10	0.1230	1.0425	0.0812	0.052*
C11	0.03590 (19)	1.1734 (4)	−0.0123 (7)	0.0553 (10)
H11	0.0596	1.2619	−0.0748	0.066*
C12	0.0000	0.8038 (8)	0.6262 (8)	0.0549 (14)
H12A	−0.0386	0.7670	0.7135	0.082*
H12B	0.0000	0.9205	0.5993	0.082*

C13	0.0000	0.5227 (6)	0.4407 (9)	0.0558 (14)
H13A	−0.0388	0.4917	0.5311	0.084*
H13B	0.0000	0.4688	0.3020	0.084*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0354 (15)	0.0457 (16)	0.0328 (13)	0.0022 (12)	−0.0027 (11)	0.0001 (11)
O1	0.0520 (15)	0.0622 (15)	0.0545 (16)	0.0005 (12)	−0.0054 (12)	−0.0179 (13)
C1	0.049 (2)	0.055 (2)	0.0384 (18)	0.0024 (15)	−0.0090 (18)	−0.0074 (17)
C2	0.0366 (18)	0.0411 (17)	0.0469 (18)	0.0058 (14)	−0.0076 (16)	0.0004 (16)
C3	0.0365 (18)	0.0395 (17)	0.0449 (18)	0.0085 (14)	−0.0066 (15)	0.0019 (15)
C4	0.046 (2)	0.054 (2)	0.050 (2)	0.0123 (16)	0.0004 (17)	0.0021 (18)
C5	0.056 (2)	0.056 (2)	0.067 (3)	0.0104 (18)	0.014 (2)	0.020 (2)
C6	0.042 (2)	0.057 (2)	0.093 (3)	−0.0062 (17)	−0.002 (2)	0.012 (2)
C7	0.0351 (19)	0.055 (2)	0.062 (3)	0.0009 (15)	−0.0094 (18)	−0.0043 (17)
C8	0.036 (2)	0.045 (2)	0.035 (3)	0.000	0.000	−0.001 (2)
C9	0.0387 (15)	0.0391 (15)	0.0307 (15)	−0.0013 (14)	−0.0008 (15)	−0.0056 (13)
C10	0.0295 (16)	0.0492 (19)	0.0504 (19)	−0.0041 (14)	0.0008 (16)	−0.0064 (16)
C11	0.069 (2)	0.0404 (19)	0.056 (2)	−0.0060 (16)	0.005 (2)	0.0039 (16)
C12	0.049 (3)	0.082 (4)	0.035 (3)	0.000	0.000	−0.008 (3)
C13	0.056 (3)	0.055 (3)	0.056 (3)	0.000	0.000	0.008 (3)

Geometric parameters (Å, °)

N1—C9	1.420 (4)	C6—H6	0.9300
N1—C1	1.472 (4)	C7—H7	0.9300
N1—C8	1.505 (4)	C8—N1^{i}	1.505 (4)
O1—C3	1.373 (4)	C8—C13	1.520 (7)
O1—H1	0.8200	C8—C12	1.538 (7)
C1—C2	1.505 (5)	C9—C10	1.379 (5)
C1—H1A	0.9700	C9—C9^{i}	1.400 (6)
C1—H1B	0.9700	C10—C11	1.409 (5)
C2—C3	1.397 (5)	C10—H10	0.9300
C2—C7	1.402 (5)	C11—C11^{i}	1.374 (7)
C3—C4	1.394 (5)	C11—H11	0.9300
C4—C5	1.376 (5)	C12—H12A	0.9600
C4—H4	0.9300	C12—H12B	0.9564
C5—C6	1.377 (6)	C13—H13A	0.9599
C5—H5	0.9300	C13—H13B	0.9567
C6—C7	1.387 (6)		
C9—N1—C1	118.7 (3)	C6—C7—C2	122.0 (4)
C9—N1—C8	104.8 (3)	C6—C7—H7	119.0
C1—N1—C8	118.2 (3)	C2—C7—H7	119.0
C3—O1—H1	109.5	N1—C8—N1^{i}	99.3 (3)
N1—C1—C2	113.3 (3)	N1—C8—C13	111.5 (3)
N1—C1—H1A	108.9	N1^{i}—C8—C13	111.5 (3)
C2—C1—H1A	108.9	N1—C8—C12	110.8 (2)

N1—C1—H1B	108.9	$N1^{i}$—C8—C12	110.8 (2)
C2—C1—H1B	108.9	C13—C8—C12	112.3 (4)
H1A—C1—H1B	107.7	$C10—C9—C9^{i}$	121.6 (2)
C3—C2—C7	117.7 (3)	C10—C9—N1	130.0 (3)
C3—C2—C1	122.3 (3)	$C9^{i}$—C9—N1	108.33 (16)
C7—C2—C1	119.8 (3)	C9—C10—C11	116.9 (3)
O1—C3—C4	118.2 (3)	C9—C10—H10	121.6
O1—C3—C2	121.6 (3)	C11—C10—H10	121.6
C4—C3—C2	120.2 (3)	$C11^{i}$—C11—C10	121.5 (2)
C5—C4—C3	120.5 (4)	$C11^{i}$—C11—H11	119.3
C5—C4—H4	119.7	C10—C11—H11	119.3
C3—C4—H4	119.7	C8—C12—H12A	109.4
C4—C5—C6	120.6 (4)	C8—C12—H12B	109.7
C4—C5—H5	119.7	H12A—C12—H12B	113.7
C6—C5—H5	119.7	C8—C13—H13A	109.4
C5—C6—C7	118.9 (3)	C8—C13—H13B	109.6
C5—C6—H6	120.5	H13A—C13—H13B	113.4
C7—C6—H6	120.5		
C9—N1—C1—C2	−75.7 (3)	C1—C2—C7—C6	174.7 (3)
C8—N1—C1—C2	155.8 (3)	$C9—N1—C8—N1^{i}$	35.7 (4)
N1—C1—C2—C3	−37.1 (4)	$C1—N1—C8—N1^{i}$	170.4 (2)
N1—C1—C2—C7	147.9 (3)	C9—N1—C8—C13	153.2 (3)
C7—C2—C3—O1	177.8 (3)	C1—N1—C8—C13	−72.0 (4)
C1—C2—C3—O1	2.6 (5)	C9—N1—C8—C12	−80.9 (3)
C7—C2—C3—C4	−0.1 (5)	C1—N1—C8—C12	53.8 (4)
C1—C2—C3—C4	−175.3 (3)	C1—N1—C9—C10	23.7 (5)
O1—C3—C4—C5	−177.4 (3)	C8—N1—C9—C10	158.1 (4)
C2—C3—C4—C5	0.6 (5)	$C1—N1—C9—C9^{i}$	−157.9 (2)
C3—C4—C5—C6	−0.3 (5)	$C8—N1—C9—C9^{i}$	−23.4 (2)
C4—C5—C6—C7	−0.4 (6)	$C9^{i}$—C9—C10—C11	−0.4 (4)
C5—C6—C7—C2	0.9 (6)	N1—C9—C10—C11	177.9 (3)
C3—C2—C7—C6	−0.6 (5)	$C9—C10—C11—C11^{i}$	0.4 (4)

Symmetry codes: (i) −*x*, *y*, *z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.82	1.98	2.708 (4)	147
$C4—H4···Cg1^{ii}$	0.93	2.71	3.51 (2)	144

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

Fig. 1

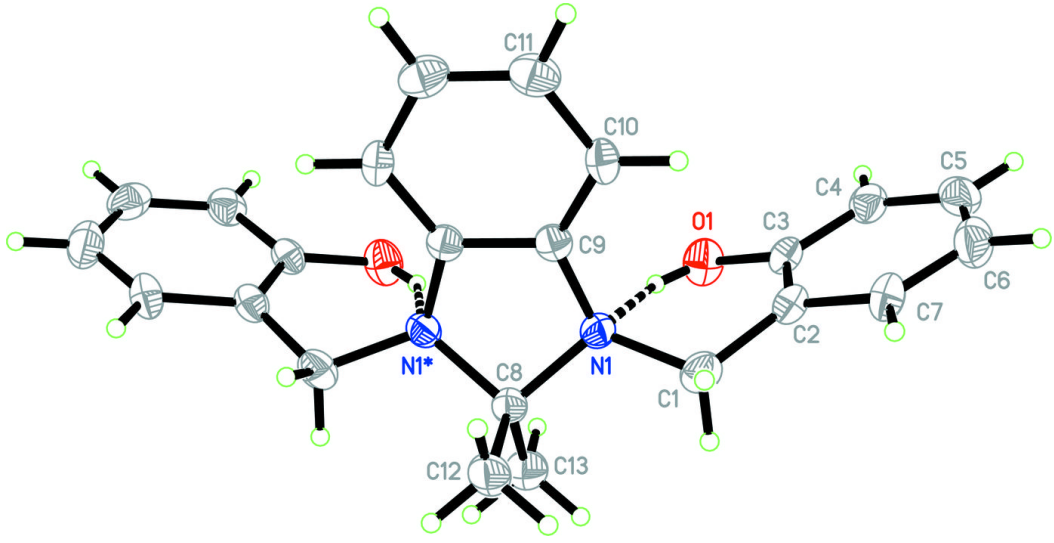


Fig. 2