

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

1,3-Bis(1*H*-benzimidazol-2-yl)-2-oxa-
propaneYing Chen,^a Jixi Guo,^b Ruirui Yun^a and Huilu Wu^{a*}

^aSchool of Chemical and Biological Engineering, Lanzhou Jiaotong University, Lanzhou 730070, People's Republic of China, and ^bInstitute of Applied Chemistry, Xinjiang University, Urumqi 830046, Xinjiang, People's Republic of China
Correspondence e-mail: wuhuilu@163.com

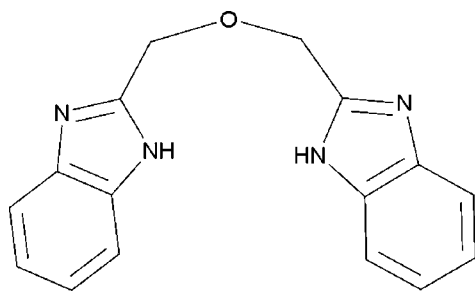
Received 21 March 2009; accepted 28 March 2009

Key indicators: single-crystal X-ray study; $T = 153$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; disorder in main residue; R factor = 0.043; wR factor = 0.112; data-to-parameter ratio = 14.1.

The title molecule, $\text{C}_{16}\text{H}_{14}\text{N}_4\text{O}$, lies on a crystallographic inversion center. The $-\text{CH}_2-$ groups and the O atom are disordered over two sites with equal occupancy, the disorder of the O atom being symmetry imposed. In the crystal structure, molecules are linked into a two-dimensional network parallel to (001) *via* intermolecular $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds.

Related literature

For the applications of bis(2-benzimidazolyl)alkanes, see: Cai *et al.* (2003); Min & Suh (2000); Roderick *et al.* (1972). For the isostructural amine analog, see: Tarazon Navarro & McKee (2003).



Experimental

Crystal data

 $\text{C}_{16}\text{H}_{14}\text{N}_4\text{O}$ $M_r = 278.31$ Orthorhombic, *Pbca* $a = 8.2143$ (4) Å $b = 9.6296$ (3) Å $c = 16.8088$ (7) Å $V = 1329.58$ (9) Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 0.09$ mm⁻¹ $T = 153$ K $0.19 \times 0.13 \times 0.09$ mm

Data collection

Rigaku R-Axis Spider diffractometer

Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995) $T_{\min} = 0.983$, $T_{\max} = 0.992$

11836 measured reflections

1525 independent reflections

1189 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.045$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.043$ $wR(F^2) = 0.112$ $S = 1.08$

1525 reflections

108 parameters

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\max} = 0.22$ e Å⁻³ $\Delta\rho_{\min} = -0.20$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N}2-\text{H}2\text{N}\cdots\text{N}1^i$	0.953 (19)	1.951 (19)	2.8803 (16)	164.2 (15)

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

Data collection: *RAPID-AUTO* (Rigaku/MSC, 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; 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*.

The authors acknowledge the financial support and a grant from the Qing Lan Talent Engineering Funds of Lanzhou Jiaotong University. A grant from the Middle-Young Age Science Foundation of Gansu Province (grant No. 3YS061-A25-023,24) is also acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2795).

References

- Cai, Y. P., Chen, C. L., Zhang, L., Shi, J. L., Xu, A. W., Su, C. Y. & Kang, B. S. (2003). *Inorg. Chim. Acta*, **342**, 107–113.
 Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
 Min, K. S. & Suh, M. P. (2000). *J. Am. Chem. Soc.* **122**, 6834–6840.
 Tarazon Navarro, A. & McKee, V. (2003). *Acta Cryst.* **E59**, o1199–1201.
 Rigaku/MSC (2004). *RAPID-AUTO*. Rigaku/MSC, The Woodlands, Texas, USA.
 Roderick, W. R., Nordeen, C. W., Von Esch, A. M. & Appell, R. N. J. (1972). *J. Med. Chem.* **15**, 655–658.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supplementary materials

Acta Cryst. (2009). E65, o948 [doi:10.1107/S1600536809011507]

1,3-Bis(1*H*-benzimidazol-2-yl)-2-oxapropane

Y. Chen, J. Guo, R. Yun and H. Wu

Comment

Interest in bis(2-benzimidazolyl)alkanes and their derivatives are widespread and has originated from their wide-ranging anti-viral activity and their importance in selective ion-exchange resins (Cai *et al.*, 2003; Min *et al.*, 2000; Roderick *et al.*, 1972). The molecular structure of the title compound is shown in Fig. 1 and is isostructural with the amine analog (Tarazon Navarro & McKee, 2003). In the crystal structure, molecules are linked into a two-dimensional network parallel to the (001) plane via intermolecular N-H...N hydrogen bonds (Fig. 2).

Experimental

21.44 g (160 mmol) of diglycolic acid was combined with 34.56 g (320 mmol) of *o*-phenylenediamine in 350 ml of 5 N HCl. The solution was refluxed for 24 h. The resulting solution was neutralized with NH₄OH. The white precipitate was collected, washed with MeOH and absolute Et₂O, and dried *in vacuo*. The dried precipitate was dissolved in DMF to a green solution and through the ether diffusion exhalation crystal after three days at room temperature. The colorless crystals suitable for X-ray diffraction studies were obtained after four weeks. Yield, 29.36 g (66%). (found: C, 68.78; H, 5.09; N, 20.51 Calcd. for C₁₆H₁₄N₄O: C, 69.05; H, 5.07; N, 20.13)

Refinement

H atoms were included in calculated positions and refined in a riding-model approximation with C—H distances ranging from 0.95 to 0.96 Å and $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$. The H atom bonded to N2 was refined independently with an isotropic displacement parameter. The -CH₂ groups and the O atom are disordered over two sites with equal occupancy, the disorder of the O atom being symmetry imposed. The anisotropic displacement parameters of C8 and C8* were constrained to be equal using the EADP instruction in SHELXL (Sheldrick, 2008).

Figures

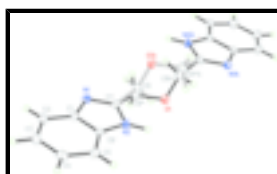


Fig. 1. The molecular structure of the title compound with displacement ellipsoids shown at the 30% probability level. Open bonds indicate the disorder component. Symmetry code (A): $-x+2, -y+1, -z+1$.

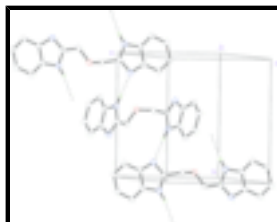


Fig. 2. Part of the crystal structure of the title compound with hydrogen bonds shown as dashed lines. Only H atoms involved in hydrogen bonds are shown. The disorder is not shown.

1,3-Bis(1*H*-benzimidazol-2-yl)-2-oxapropane

Crystal data

$C_{16}H_{14}N_4O$	$F_{000} = 584$
$M_r = 278.31$	$D_x = 1.390 \text{ Mg m}^{-3}$
Orthorhombic, <i>Pbca</i>	Mo $K\alpha$ radiation
Hall symbol: -P 2ac 2ab	$\lambda = 0.71073 \text{ \AA}$
$a = 8.2143 (4) \text{ \AA}$	Cell parameters from 1231 reflections
$b = 9.6296 (3) \text{ \AA}$	$\theta = 3.5\text{--}25.5^\circ$
$c = 16.8088 (7) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$V = 1329.58 (9) \text{ \AA}^3$	$T = 153 \text{ K}$
$Z = 4$	Prism, colourless
	$0.19 \times 0.13 \times 0.09 \text{ mm}$

Data collection

Rigaku R-Axis Spider diffractometer	1525 independent reflections
Radiation source: fine-focus sealed tube	1189 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.045$
$T = 153 \text{ K}$	$\theta_{\text{max}} = 27.5^\circ$
φ and ω scans	$\theta_{\text{min}} = 3.5^\circ$
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	$h = -10 \rightarrow 10$
$T_{\text{min}} = 0.983$, $T_{\text{max}} = 0.992$	$k = -12 \rightarrow 11$
11836 measured reflections	$l = -20 \rightarrow 21$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.043$	$w = 1/[\sigma^2(F_o^2) + (0.0566P)^2 + 0.3224P]$
$wR(F^2) = 0.112$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.08$	$(\Delta/\sigma)_{\text{max}} < 0.001$
1525 reflections	$\Delta\rho_{\text{max}} = 0.22 \text{ e \AA}^{-3}$
108 parameters	$\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: SHELXL97 (Sheldrick, 2008), $F_c^* = kF_c[1 + 0.001 \times F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
Secondary atom site location: difference Fourier map	Extinction coefficient: 0.015 (3)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
O1	0.9632 (3)	0.5599 (2)	0.53873 (12)	0.0332 (5)	0.50
N1	0.64853 (14)	0.36549 (11)	0.43694 (6)	0.0256 (3)	
N2	0.71007 (14)	0.59185 (11)	0.43361 (7)	0.0254 (3)	
H2N	0.770 (2)	0.675 (2)	0.4419 (10)	0.049 (5)*	
C1	0.54372 (16)	0.43254 (13)	0.38456 (8)	0.0245 (3)	
C2	0.41904 (18)	0.37997 (14)	0.33731 (9)	0.0329 (4)	
H2A	0.3907	0.2844	0.3391	0.039*	
C3	0.3378 (2)	0.47100 (16)	0.28783 (10)	0.0391 (4)	
H3A	0.2525	0.4372	0.2549	0.047*	
C4	0.3787 (2)	0.61227 (16)	0.28509 (9)	0.0366 (4)	
H4A	0.3217	0.6717	0.2496	0.044*	
C5	0.49977 (17)	0.66712 (14)	0.33267 (8)	0.0303 (3)	
H5A	0.5257	0.7633	0.3319	0.036*	
C6	0.58166 (16)	0.57462 (13)	0.38169 (7)	0.0237 (3)	
C7	0.74397 (17)	0.46457 (12)	0.46425 (8)	0.0254 (3)	
C8	0.8957 (12)	0.4403 (12)	0.5122 (5)	0.0319 (12)	0.50
H8A	0.8872	0.3484	0.5339	0.038*	0.50
H8B	0.9052	0.5059	0.5549	0.038*	0.50
C8*	0.8603 (12)	0.4441 (12)	0.5307 (5)	0.0319 (12)	0.50
H8*A	0.8049	0.4226	0.5795	0.038*	0.50
H8*B	0.9273	0.3657	0.5177	0.038*	0.50

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0271 (11)	0.0310 (10)	0.0415 (11)	-0.0002 (8)	-0.0057 (9)	-0.0077 (8)
N1	0.0253 (6)	0.0221 (6)	0.0293 (6)	0.0006 (4)	0.0002 (5)	0.0014 (4)
N2	0.0249 (6)	0.0218 (6)	0.0297 (6)	-0.0011 (5)	-0.0015 (5)	0.0011 (4)
C1	0.0220 (7)	0.0231 (7)	0.0283 (7)	0.0009 (5)	0.0033 (5)	0.0007 (5)
C2	0.0314 (8)	0.0266 (7)	0.0406 (8)	-0.0031 (6)	-0.0052 (6)	-0.0021 (6)
C3	0.0342 (9)	0.0363 (8)	0.0469 (9)	-0.0002 (6)	-0.0148 (7)	-0.0009 (6)
C4	0.0346 (8)	0.0344 (8)	0.0407 (8)	0.0067 (6)	-0.0091 (7)	0.0039 (6)

supplementary materials

C5	0.0312 (8)	0.0225 (7)	0.0372 (7)	0.0037 (5)	-0.0002 (6)	0.0016 (5)
C6	0.0208 (7)	0.0230 (6)	0.0273 (6)	0.0008 (5)	0.0010 (5)	-0.0009 (5)
C7	0.0262 (7)	0.0221 (7)	0.0279 (6)	0.0010 (5)	-0.0001 (5)	0.0012 (5)
C8	0.029 (4)	0.0320 (9)	0.034 (4)	-0.001 (2)	-0.004 (2)	0.002 (2)
C8*	0.029 (4)	0.0320 (9)	0.034 (4)	-0.001 (2)	-0.004 (2)	0.002 (2)

Geometric parameters (\AA , $^\circ$)

O1—C8*	1.405 (12)	C4—C5	1.381 (2)
O1—C8 ⁱ	1.441 (7)	C4—H4A	0.9500
N1—C7	1.3175 (17)	C5—C6	1.3874 (18)
N1—C1	1.3904 (17)	C5—H5A	0.9500
N2—C7	1.3583 (16)	C7—C8*	1.483 (12)
N2—C6	1.3790 (17)	C7—C8	1.502 (12)
N2—H2N	0.953 (19)	C8—O1 ⁱ	1.441 (7)
C1—C2	1.3914 (19)	C8—H8A	0.9600
C1—C6	1.4041 (19)	C8—H8B	0.9600
C2—C3	1.380 (2)	C8*—O1 ⁱ	1.862 (7)
C2—H2A	0.9500	C8*—H8*A	0.9600
C3—C4	1.402 (2)	C8*—H8*B	0.9600
C3—H3A	0.9500		
C8—O1—C8 ⁱ	97.6 (5)	N1—C7—C8	124.6 (5)
C8*—O1—C8 ⁱ	115.2 (7)	N2—C7—C8	120.9 (5)
C8—O1—C8* ⁱ	95.4 (5)	O1—C8—C7	112.6 (8)
C8*—O1—C8* ⁱ	113.1 (3)	O1 ⁱ —C8—C7	110.5 (6)
C8 ⁱ —O1—H8B	136.0	O1—C8—H8A	133.4
C8* ⁱ —O1—H8B	134.6	O1 ⁱ —C8—H8A	106.5
C7—N1—C1	104.64 (10)	C7—C8—H8A	106.7
C7—N2—C6	106.76 (11)	O1 ⁱ —C8—H8B	112.3
C7—N2—H2N	126.8 (11)	C7—C8—H8B	111.5
C6—N2—H2N	126.2 (11)	H8A—C8—H8B	109.1
N1—C1—C2	130.41 (12)	O1—C8—H8*A	93.2
N1—C1—C6	109.68 (12)	O1 ⁱ —C8—H8*A	158.6
C2—C1—C6	119.88 (12)	C7—C8—H8*A	90.6
C3—C2—C1	117.95 (13)	H8A—C8—H8*A	62.0
C3—C2—H2A	121.0	H8B—C8—H8*A	60.4
C1—C2—H2A	121.0	O1—C8—H8*B	128.0
C2—C3—C4	121.35 (14)	O1 ⁱ —C8—H8*B	78.4
C2—C3—H3A	119.3	C7—C8—H8*B	119.4
C4—C3—H3A	119.3	H8B—C8—H8*B	119.7
C5—C4—C3	121.63 (14)	O1—C8*—C7	110.8 (7)
C5—C4—H4A	119.2	O1—C8*—H8A	128.6
C3—C4—H4A	119.2	C7—C8*—H8A	108.8
C4—C5—C6	116.58 (13)	H8A—C8*—H8B	125.3
C4—C5—H5A	121.7	O1—C8*—H8*A	111.9
C6—C5—H5A	121.7	C7—C8*—H8*A	111.5

N2—C6—C5	132.03 (12)	O1 ⁱ —C8*—H8*A	154.1
N2—C6—C1	105.38 (11)	O1—C8*—H8*B	107.5
C5—C6—C1	122.58 (13)	C7—C8*—H8*B	107.6
N1—C7—N2	113.54 (12)	H8B—C8*—H8*B	115.5
N1—C7—C8*	123.3 (5)	H8*A—C8*—H8*B	107.3
N2—C7—C8*	122.5 (5)		

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

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2N \cdots N1 ⁱⁱ	0.953 (19)	1.951 (19)	2.8803 (16)	164.2 (15)

Symmetry codes: (ii) $-x+3/2, y+1/2, z$.

Fig. 1

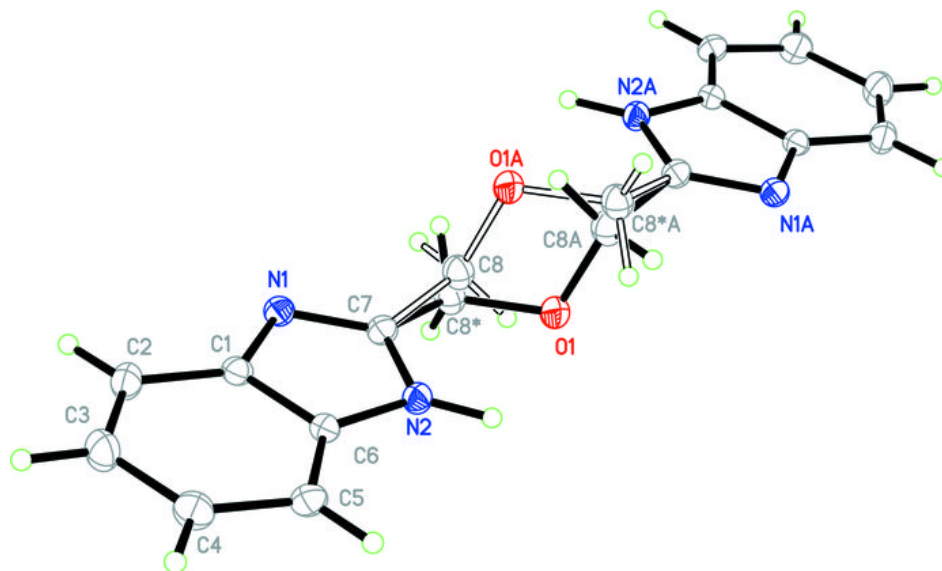


Fig. 2

