Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

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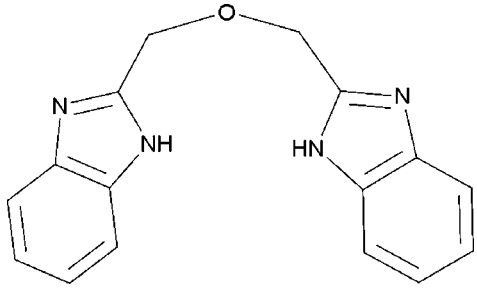
Received 21 March 2009; accepted 28 March 2009

Key indicators: single-crystal X-ray study; T = 153 K; mean σ(C–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, $C_{16}H_{14}N_4O$, lies on a crystallographic inversion center. The $-CH_2-$ 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 N—H···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

$C_{16}H_{14}N_4O$
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) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 153 K
0.19 × 0.13 × 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_{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—H···A	D—H	H···A	D···A	D—H···A
N2—H2*N*···N1^{i}	0.953 (19)	1.951 (19)	2.8803 (16)	164.2 (15)

Symmetry code: (i) $-x+\frac{3}{2}, y+\frac{1}{2}, z$.

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).

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supporting information

Acta Cryst. (2009). E**65**, o948 [doi:10.1107/S1600536809011507]

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

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S1. 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).

S2. 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 NH_4OH. The white precipitate was collected, washed with MeOH and absolute Et_2O, 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)

S3. Refinement

H atoms were included in calclulated positions and refined in a riding-model approximation with C—H distances ranging from 0.95 to 0.96Å and U_{iso}(H) = 1.2 U_{eq}(C). The H atom bonded to N2 was refined independently with an isotropic displacement parameter. The -CH_2 groups and the O atom are disordered over two sites with equall occupancy, the disorder of the O atom being symmetry imposed. The anisotropic displacement parameters of C8 and C8* were constrained to be equall using the EADP instruction in SHELXL (Sheldrick, 2008).

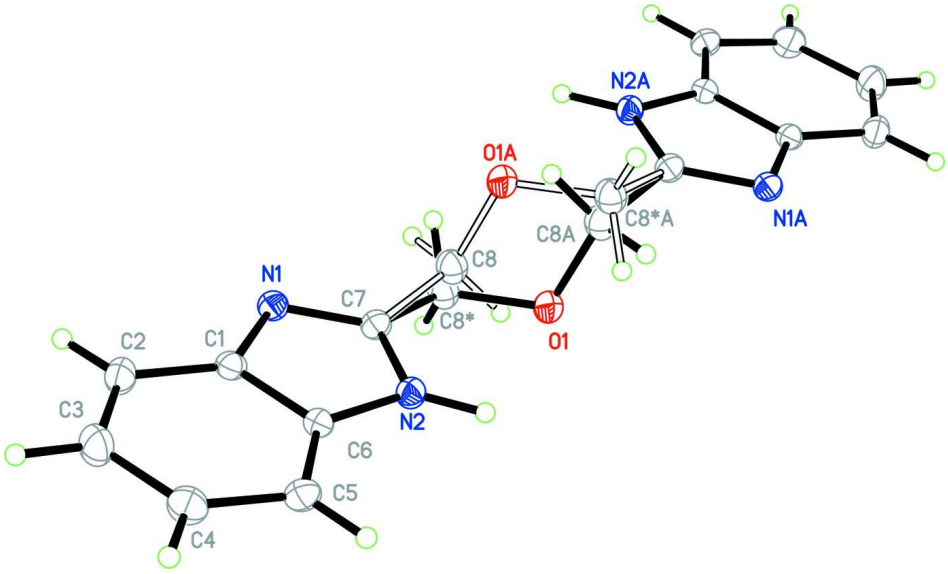

Figure 1

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.

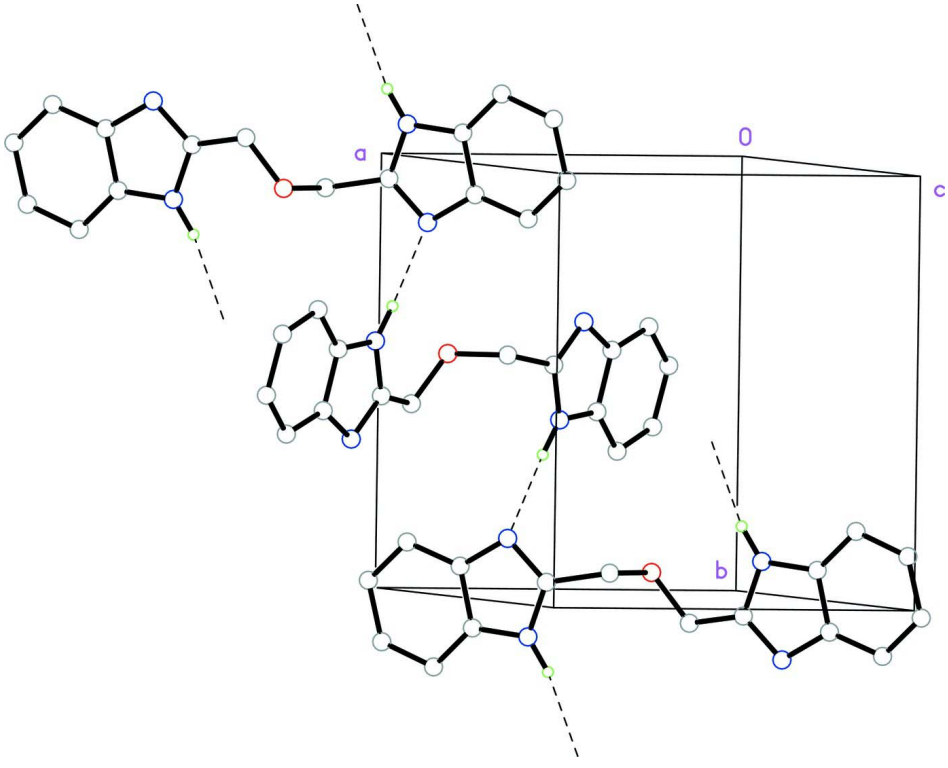

Figure 2

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

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

Crystal data

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

Data collection

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

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.112$
$S = 1.08$
1525 reflections
108 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0566P)^2 + 0.3224P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
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 $F^2 > 2\text{sigma}(F^2)$ 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}	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 (Å²)

	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)
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 (Å, º)

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 code: (i) −*x*+2, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*N*···N1^{ii}	0.953 (19)	1.951 (19)	2.8803 (16)	164.2 (15)

Symmetry code: (ii) −*x*+3/2, *y*+1/2, *z*.