Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(1*R*,2*R*)-*N*,*N*′-Bis[1-(2-pyridyl)ethylidene]cyclohexane-1,2-diamine

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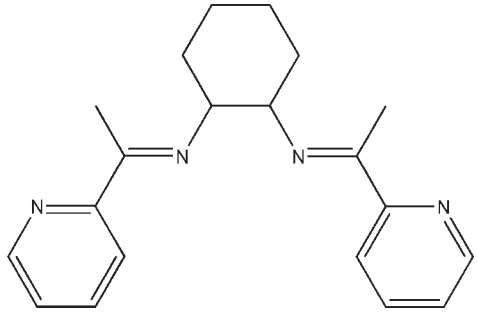
Received 7 April 2010; accepted 13 April 2010

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.040; wR factor = 0.121; data-to-parameter ratio = 18.6.

In the title compound, $C_{20}H_{24}N_4$, the cyclohexane ring adopts a chair conformation with the two imine groups linked at equatorial positions. The two halves of the molecule are related by a crystallographic twofold rotation axis. The dihedral angle between the pyridine rings is 75.73 (3)°.

Related literature

For the crystal structures of some Schiff bases derived from cyclohexane-1,2-diamine, see: Aslantaş *et al.* (2007); Glidewell *et al.* (2005); Liu *et al.* (2006).



Experimental

Crystal data

$C_{20}H_{24}N_4$
M_r = 320.43
Monoclinic, $C2/c$
a = 18.0605 (3) Å
b = 8.9371 (1) Å
c = 11.1076 (2) Å
β = 97.970 (1)°
V = 1775.54 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 100 K
0.49 × 0.37 × 0.35 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.965, T_{max} = 0.975
8186 measured reflections
2044 independent reflections
1833 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.121
S = 1.06
2044 reflections
110 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *publCIF* (Westrip, 2010).

The authors thank the University of Malaya for funding this study (FRGS grant No. FP009/2008 C).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2274).

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supporting information

Acta Cryst. (2010). E**66**, o1095 [https://doi.org/10.1107/S1600536810013607]

(1*R*,2*R*)-*N*,*N*′-Bis[1-(2-pyridyl)ethylidene]cyclohexane-1,2-diamine

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S1. Comment

The stucture of the title compound is presented in Fig. 1. The cyclohexane ring adopts a chair conformation with the two imines linked at equatorial positions. The two halves of the molecule are realted by a two-fold rotation. The dihedral angel between the two pyridine rings is 75.73 (3)°. The crystal structure is devoid of any inter- or intra- molecular interactions.

The bond distances and angles in the title molecule are in agreement with the corresponding bond distances and angles reported in some related structures (Aslantaş *et al.*, 2007; Glidewell *et al.*, 2005; Liu *et al.*, 2006).

S2. Experimental

A mixture of 2-acetylpyiridine (0.444 g, 4 mmol) and 1,2-diaminocyclohexane (0.224, 2 mmol) was refluxed in ethanol (50 ml) for 2 hours. The solution was then set aside overnight whereupon the yellow crystals of the title compound were formed.

S3. Refinement

Hydrogen atoms were placed at calculated positions (C—H 0.95-1.00 Å), and were treated as riding on their parent atoms with U_{iso}(H) set to 1.2-1.5 U_{eq}(C).

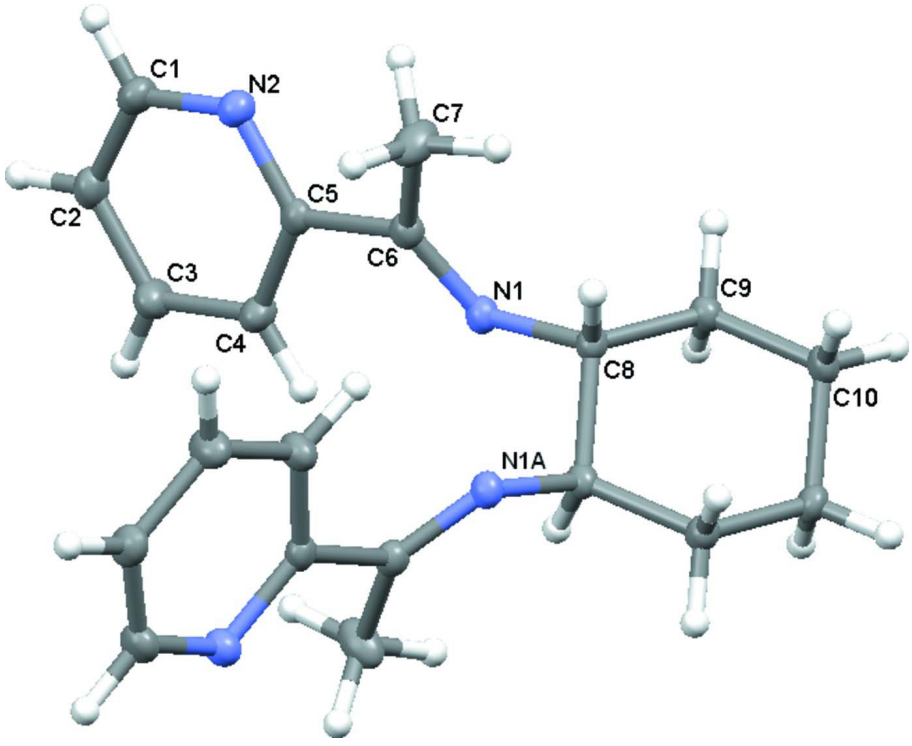

Figure 1

Thermal ellipsoid plot of the title compound at 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius. Symmetry code for the unlabeled atoms: -x, y, -z+3/2.

(1*R*,2*R*)-*N*,*N*′-Bis[1-(2- pyridyl)ethylidene]cyclohexane-1,2-diamine

Crystal data

$C_{20}H_{24}N_4$
$M_r = 320.43$
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
$a = 18.0605\ (3)$ Å
$b = 8.9371\ (1)$ Å
$c = 11.1076\ (2)$ Å
$\beta = 97.970\ (1)°$
$V = 1775.54\ (5)$ Å^3
$Z = 4$
$F(000) = 688$
$D_x = 1.199$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4367 reflections
$\theta = 2.3–30.3°$
$\mu = 0.07$ mm^{-1}
$T = 100$ K
Block, pale yellow
0.49 × 0.37 × 0.35 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.965$, $T_{max} = 0.975$
8186 measured reflections
2044 independent reflections
1833 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.3°$
$h = -23\rightarrow22$
$k = -11\rightarrow11$
$l = -14\rightarrow14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.121$
$S = 1.06$
2044 reflections
110 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
$w = 1/[\sigma^2(F_o^2) + (0.0714P)^2 + 1.0614P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-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 $F^2 > \sigma(F^2)$ 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}
N1	0.07952 (5)	0.04557 (10)	0.74621 (8)	0.0160 (2)
N2	0.19413 (5)	0.35854 (10)	0.82811 (8)	0.0188 (2)
C1	0.22757 (6)	0.47046 (13)	0.77554 (10)	0.0214 (3)
H1	0.2635	0.5294	0.8252	0.026*
C2	0.21265 (6)	0.50475 (13)	0.65292 (10)	0.0212 (3)
H2	0.2368	0.5865	0.6199	0.025*
C3	0.16173 (7)	0.41697 (13)	0.57979 (10)	0.0233 (3)
H3	0.1504	0.4372	0.4953	0.028*
C4	0.12752 (6)	0.29891 (12)	0.63160 (10)	0.0208 (3)
H4	0.0929	0.2362	0.5831	0.025*
C5	0.14487 (6)	0.27409 (11)	0.75622 (9)	0.0154 (2)
C6	0.10752 (6)	0.14970 (11)	0.81635 (9)	0.0160 (2)
C7	0.10754 (7)	0.16392 (14)	0.95159 (10)	0.0269 (3)
H7A	0.0734	0.2443	0.9679	0.040*
H7B	0.1582	0.1873	0.9909	0.040*
H7C	0.0910	0.0694	0.9838	0.040*
C8	0.03861 (5)	−0.08031 (11)	0.78929 (9)	0.0151 (2)
H8	0.0327	−0.0650	0.8766	0.018*
C9	0.08258 (6)	−0.22419 (11)	0.77538 (9)	0.0169 (2)
H9A	0.0978	−0.2272	0.6931	0.020*
H9B	0.1285	−0.2242	0.8354	0.020*
C10	0.03633 (6)	−0.36338 (12)	0.79419 (10)	0.0188 (3)
H10A	0.0656	−0.4542	0.7812	0.023*
H10B	0.0248	−0.3653	0.8787	0.023*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0176 (4)	0.0139 (4)	0.0170 (4)	−0.0002 (3)	0.0038 (3)	0.0006 (3)
N2	0.0187 (5)	0.0183 (5)	0.0191 (4)	−0.0021 (3)	0.0020 (3)	−0.0008 (3)
C1	0.0197 (5)	0.0199 (6)	0.0239 (5)	−0.0045 (4)	0.0007 (4)	−0.0011 (4)
C2	0.0208 (5)	0.0178 (5)	0.0253 (6)	−0.0030 (4)	0.0040 (4)	0.0038 (4)
C3	0.0283 (6)	0.0224 (6)	0.0186 (5)	−0.0045 (4)	0.0011 (4)	0.0038 (4)
C4	0.0244 (6)	0.0186 (5)	0.0184 (5)	−0.0052 (4)	0.0002 (4)	0.0000 (4)
C5	0.0154 (5)	0.0127 (5)	0.0184 (5)	0.0017 (4)	0.0039 (4)	−0.0007 (4)
C6	0.0155 (5)	0.0157 (5)	0.0173 (5)	0.0013 (4)	0.0039 (4)	0.0001 (4)
C7	0.0362 (7)	0.0279 (6)	0.0176 (5)	−0.0114 (5)	0.0075 (5)	−0.0030 (4)
C8	0.0179 (5)	0.0134 (5)	0.0140 (4)	−0.0012 (4)	0.0027 (4)	0.0004 (4)
C9	0.0176 (5)	0.0152 (5)	0.0179 (5)	0.0008 (4)	0.0023 (4)	0.0010 (4)
C10	0.0215 (6)	0.0136 (5)	0.0211 (5)	0.0015 (4)	0.0020 (4)	0.0019 (4)

Geometric parameters (Å, º)

N1—C6	1.2726 (14)	C6—C7	1.5075 (15)
N1—C8	1.4623 (12)	C7—H7A	0.9800
N2—C5	1.3422 (14)	C7—H7B	0.9800
N2—C1	1.3432 (14)	C7—H7C	0.9800
C1—C2	1.3856 (16)	C8—C9	1.5304 (14)
C1—H1	0.9500	C8—C8^{i}	1.5392 (19)
C2—C3	1.3833 (16)	C8—H8	1.0000
C2—H2	0.9500	C9—C10	1.5288 (14)
C3—C4	1.3874 (15)	C9—H9A	0.9900
C3—H3	0.9500	C9—H9B	0.9900
C4—C5	1.3940 (15)	C10—C10^{i}	1.526 (2)
C4—H4	0.9500	C10—H10A	0.9900
C5—C6	1.5039 (14)	C10—H10B	0.9900
C6—N1—C8	122.56 (9)	H7A—C7—H7B	109.5
C5—N2—C1	117.45 (9)	C6—C7—H7C	109.5
N2—C1—C2	123.65 (10)	H7A—C7—H7C	109.5
N2—C1—H1	118.2	H7B—C7—H7C	109.5
C2—C1—H1	118.2	N1—C8—C9	108.70 (8)
C3—C2—C1	118.33 (10)	N1—C8—C8^{i}	105.88 (7)
C3—C2—H2	120.8	C9—C8—C8^{i}	112.63 (6)
C1—C2—H2	120.8	N1—C8—H8	109.8
C2—C3—C4	119.06 (10)	C9—C8—H8	109.8
C2—C3—H3	120.5	C8^{i}—C8—H8	109.8
C4—C3—H3	120.5	C10—C9—C8	111.64 (8)
C3—C4—C5	118.76 (10)	C10—C9—H9A	109.3
C3—C4—H4	120.6	C8—C9—H9A	109.3
C5—C4—H4	120.6	C10—C9—H9B	109.3
N2—C5—C4	122.72 (10)	C8—C9—H9B	109.3
N2—C5—C6	116.91 (9)	H9A—C9—H9B	108.0

C4—C5—C6	120.37 (9)	C10^{i}—C10—C9	110.46 (7)
N1—C6—C5	115.68 (9)	C10^{i}—C10—H10A	109.6
N1—C6—C7	128.08 (10)	C9—C10—H10A	109.6
C5—C6—C7	116.23 (9)	C10^{i}—C10—H10B	109.6
C6—C7—H7A	109.5	C9—C10—H10B	109.6
C6—C7—H7B	109.5	H10A—C10—H10B	108.1

Symmetry code: (i) $-x$, y, $-z+3/2$.