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Crystal structure of (1*E*,1′*E*)-*N*,*N*′-(ethane-1,2-diyl)bis[(pyridin-2-yl)-methanimine]

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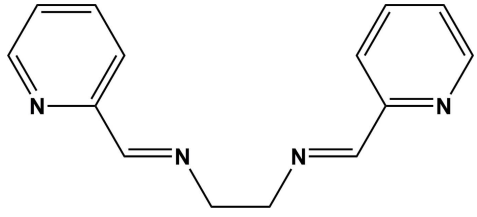
The whole molecule of the title compound, $C_{14}H_{14}N_4$, is generated by twofold rotation symmetry. The twofold axis bisects the central $-CH_2$-CH_2- bond and the planes of the pyridine rings are inclined to one another by 65.60 (7)°. In the crystal, there are no significant intermolecular interactions present.

Keywords: crystal structure; pyridinecarbaldehydes; 1,2-diaminopyridine; Schiff base; chelating ligands.

CCDC reference: 1402701

1. Related literature

For the use of Schiff bases, derived from pyridine-carbaldehydes, in synthetic chemistry, see: Marjani *et al.* (2009). For 1,2-diaminopyridine-derived Schiff bases as bidentate or polydentate chelating ligands and their possible medical applications, see: Warad *et al.* (2014).



2. Experimental

2.1. Crystal data

$C_{14}H_{14}N_4$
M_r = 238.29
Monoclinic, $C2/c$
a = 19.347 (5) Å
b = 5.9339 (12) Å
c = 13.165 (2) Å
β = 122.266 (8)°
V = 1278.0 (5) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 0.61 mm^{-1}
T = 296 K
0.30 × 0.27 × 0.25 mm

2.2. Data collection

Bruker X8 Proteum diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
T_{min} = 0.837, T_{max} = 0.862
1539 measured reflections
933 independent reflections
881 reflections with $I > 2\sigma(I)$
R_{int} = 0.015

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.120
S = 1.05
933 reflections
82 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.10 e Å^{-3}
$\Delta\rho_{min}$ = −0.10 e Å^{-3}

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); 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: *SHELXL97* and *PLATON* (Spek, 2009).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: SU5142).

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supporting information

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Crystal structure of (1*E*,1′*E*)-*N*,*N*′-(ethane-1,2-diyl)bis[(pyridin-2-yl)methanimine]

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S1. Structural commentary

Schiff bases derived from pyridinecarbaldehydes have received considerable interest in synthetic chemistry (Marjani *et al.*, 2009). 1,2-diamine-pyridine derived Schiff base bidentate or polydentate chelating ligand towards metal centers draw major attraction towards synthesis and medical application (Warad *et al.*, 2014). It is still challenging to design and rationally synthesize ligands with unique structures and functions.

S2. Synthesis and crystallization

To a solution of pyridine-2-carbaldehyde (1 mmol) dissolved in 10 ml of absolute ethanol was added drop wise ethane-1,2-diamine (1 mmol) in 5 ml of absolute ethanol under constant stirring for 10 min. The mixture was refluxed for 4 h and then concentrated under reduced pressure. The title compound was precipitated by the addition of 50 ml of n-hexane. It was filtered off, washed three times with 80 ml of distilled water then with diethyl ether to give the title compound (yield: 86%). Single crystals suitable for X-ray analysis were obtained within two days by slow evaporation of a solution in dichloromethane.

S3. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. The H atoms were fixed geometrically (C—H = 0.93 – 0.97 Å) and allowed to ride on their parent atoms with U_{iso}(H) = 1.2U_{eq}(C).

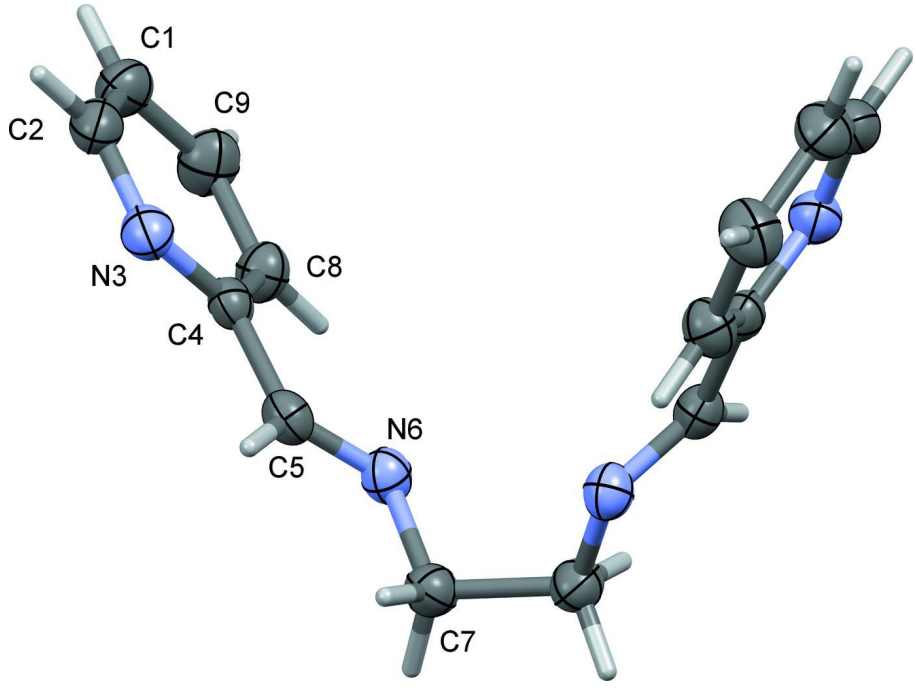

Figure 1

View of the molecular structure of the title compound, with atom labelling. Displacement ellipsoids are drawn at the 50% probability level. Unlabelled atoms are related to the labelled atoms by twofold rotation symmetry (symmetry code: -*x* + 1, *y*, -*z* - 1/2).

(1*E*,1′*E*)-*N*,*N*′-(Ethane-1,2-diyl)bis[(pyridin-2-yl)methanimine]

Crystal data

$C_{14}H_{14}N_4$
M_r = 238.29
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
a = 19.347 (5) Å
b = 5.9339 (12) Å
c = 13.165 (2) Å
β = 122.266 (8)°
V = 1278.0 (5) Å^3
Z = 4

F(000) = 504
D_x = 1.238 Mg m^{-3}
Cu *Kα* radiation, *λ* = 1.54178 Å
Cell parameters from 881 reflections
θ = 5.4–63.8°
μ = 0.61 mm^{-1}
T = 296 K
Block, colourless
0.30 × 0.27 × 0.25 mm

Data collection

Bruker X8 Proteum diffractometer
Radiation source: Rotating Anode
Graphite monochromator
Detector resolution: 18.4 pixels mm^{-1}
φ and *ω* scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
T_{min} = 0.837, T_{max} = 0.862

1539 measured reflections
933 independent reflections
881 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
θ_{max} = 63.8°, θ_{min} = 5.4°
h = −8→21
k = −6→5
l = −15→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.120$
$S = 1.05$
933 reflections
82 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
$w = 1/[\sigma^2(F_o^2) + (0.0763P)^2 + 0.2763P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.10$ e Å^{-3}
$\Delta\rho_{min} = -0.10$ e Å^{-3}

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -*R*-factor-obs *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}
N3	0.69126 (7)	0.50665 (18)	0.02615 (10)	0.0576 (4)
N6	0.55349 (7)	0.05345 (19)	−0.11311 (9)	0.0542 (4)
C1	0.67191 (10)	0.7085 (3)	0.16610 (14)	0.0703 (6)
C2	0.70819 (9)	0.6792 (2)	0.10143 (14)	0.0634 (5)
C4	0.63482 (8)	0.3583 (2)	0.01373 (11)	0.0478 (4)
C5	0.61592 (8)	0.1747 (2)	−0.07264 (11)	0.0495 (4)
C7	0.54261 (9)	−0.1287 (2)	−0.19397 (13)	0.0585 (5)
C8	0.59615 (9)	0.3750 (2)	0.07686 (12)	0.0591 (5)
C9	0.61501 (11)	0.5541 (3)	0.15343 (14)	0.0728 (6)
H1	0.68570	0.83080	0.21760	0.0840*
H2	0.55830	0.26720	0.06750	0.0710*
H4	0.74660	0.78490	0.11040	0.0760*
H5	0.65170	0.14760	−0.09810	0.0590*
H7	0.58250	−0.11360	−0.21680	0.0700*
H8	0.55230	−0.27160	−0.15260	0.0700*
H9	0.58950	0.57050	0.19620	0.0870*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N3	0.0509 (8)	0.0571 (7)	0.0602 (7)	0.0007 (5)	0.0265 (6)	0.0064 (5)
N6	0.0501 (8)	0.0609 (7)	0.0466 (6)	0.0031 (5)	0.0225 (5)	0.0005 (5)
C1	0.0714 (11)	0.0636 (9)	0.0601 (9)	−0.0011 (7)	0.0246 (8)	−0.0102 (7)
C2	0.0554 (10)	0.0564 (9)	0.0628 (9)	−0.0038 (6)	0.0212 (7)	0.0035 (6)
C4	0.0410 (8)	0.0514 (7)	0.0430 (7)	0.0068 (5)	0.0171 (6)	0.0090 (5)

C5	0.0463 (8)	0.0551 (8)	0.0473 (7)	0.0085 (6)	0.0251 (6)	0.0079 (5)
C7	0.0600 (9)	0.0532 (8)	0.0540 (8)	0.0048 (6)	0.0249 (7)	−0.0001 (6)
C8	0.0561 (9)	0.0674 (9)	0.0542 (8)	−0.0052 (7)	0.0298 (7)	−0.0034 (6)
C9	0.0743 (12)	0.0866 (11)	0.0628 (9)	−0.0047 (8)	0.0401 (9)	−0.0136 (8)

Geometric parameters (Å, º)

N3—C2	1.3384 (18)	C8—C9	1.373 (2)
N3—C4	1.343 (2)	C1—H1	0.9300
N6—C5	1.254 (2)	C2—H4	0.9300
N6—C7	1.4514 (18)	C5—H5	0.9300
C1—C2	1.373 (3)	C7—H7	0.9700
C1—C9	1.372 (3)	C7—H8	0.9700
C4—C5	1.4730 (18)	C8—H2	0.9300
C4—C8	1.387 (2)	C9—H9	0.9300
C7—C7^{i}	1.516 (2)		
C2—N3—C4	116.96 (15)	N3—C2—H4	118.00
C5—N6—C7	117.93 (15)	C1—C2—H4	118.00
C2—C1—C9	118.85 (16)	N6—C5—H5	119.00
N3—C2—C1	123.50 (16)	C4—C5—H5	119.00
N3—C4—C5	115.43 (14)	N6—C7—H7	109.00
N3—C4—C8	122.94 (12)	N6—C7—H8	109.00
C5—C4—C8	121.62 (13)	H7—C7—H8	108.00
N6—C5—C4	122.55 (15)	C7^{i}—C7—H7	109.00
N6—C7—C7^{i}	111.74 (13)	C7^{i}—C7—H8	109.00
C4—C8—C9	118.56 (16)	C4—C8—H2	121.00
C1—C9—C8	119.17 (19)	C9—C8—H2	121.00
C2—C1—H1	121.00	C1—C9—H9	120.00
C9—C1—H1	121.00	C8—C9—H9	120.00
C2—N3—C4—C5	178.17 (12)	N3—C4—C5—N6	−164.26 (12)
C2—N3—C4—C8	−1.6 (2)	C5—C4—C8—C9	−178.16 (14)
C4—N3—C2—C1	0.9 (2)	C8—C4—C5—N6	15.5 (2)
C7—N6—C5—C4	−177.50 (11)	N3—C4—C8—C9	1.6 (2)
C5—N6—C7—C7^{i}	−131.18 (14)	N6—C7—C7^{i}—N6^{i}	73.41 (17)
C9—C1—C2—N3	−0.2 (3)	C4—C8—C9—C1	−0.8 (2)
C2—C1—C9—C8	0.1 (3)		

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