N,N′-Bis[(E)-quinoxalin-2-ylmethyl-idene]ethane-1,2-diamine

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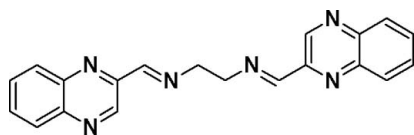Correspondence e-mail: yusuff@cusat.ac.in

In the molecule of the title compound, $C_{20}H_{16}N_6$, the central C—C bond lies on a crystallographic inversion centre. The quinoxalidine ring is nearly planar, with a maximum deviation of 0.021 (2) Å from the mean plane. The crystal structure is stabilized by intermolecular C—H···N interactions, leading to the formation of a layer-like structure, which extends along the a axis.

Related literature

For the synthesis of the Schiff base, see: Zolezzi *et al.* (1999). For the properties of Schiff base ligands, see: Gupta & Sutar (2008); Harmenberg *et al.* (1991); Mayadevi *et al.* (2003); Miller *et al.* (1999); Naylor *et al.* (1993); Sreekala & Yusuff (1994); Xavier *et al.* (2004); Yusuff & Sreekala (1991). For related structures, see: Habibi *et al.* (2006); Taylor & Kennard (1982).

Experimental

Crystal data

C_{20}H_{16}N_6

$C_{20}H_{16}N_6$	γ = 116.046 (5)°
M_r = 340.39	V = 420.1 (3) Å³
Triclinic, $P\bar{1}$	Z = 1
a = 6.888 (2) Å	Mo $K\alpha$ radiation
b = 7.381 (3) Å	μ = 0.09 mm⁻¹
c = 9.638 (4) Å	T = 298 (2) K
α = 101.674 (6)°	0.40 × 0.24 × 0.18 mm
β = 96.233 (6)°	

Data collection

Bruker SMART CCD area-detector diffractometer	Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001) T_{min} = 0.967, T_{max} = 0.995
3956 measured reflections	1239 reflections with $I > 2\sigma(I)$
1465 independent reflections	R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.071	118 parameters
$wR(F^2)$ = 0.164	H-atom parameters constrained
S = 1.27	$\Delta\rho_{max}$ = 0.13 e Å⁻³
1465 reflections	$\Delta\rho_{min}$ = −0.21 e Å⁻³

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···N2^i	0.93	2.73	3.647 (4)	168
C9—H9···N1^{ii}	0.93	2.67	3.593 (3)	169

Symmetry codes: (i) $x + 1, y, z$; (ii) $x − 1, y, z$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and/or *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

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supplementary materials

N,*N'*-Bis[(*E*)-quinoxalin-2-ylmethylidene]ethane-1,2-diamine

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Comment

Schiff bases derived from aldehydes and diamines constitute one of the most relevant synthetic ligand systems. They find application in a broad range of transition metal catalyzed reactions including lactide polymerization, epoxidation of olefins, hydroxylation and asymmetric ring opening of epoxides (Gupta & Sutar, 2008). Many drug candidates bearing quinoxaline core structures are in clinical trials in antiviral (Harmenberg *et al.*, 1991), anticancer and central nervous system therapeutic areas (Naylor *et al.*, 1993). Catalytic and antibacterial activities have been observed for the Schiff base complexes derived from Quinoxaline-2-carboxaldehyde (Yusuff & Sreekala, 1991; Sreekala & Yusuff, 1994; Mayadevi *et al.*, 2003). Ethylenediamine groups appear to be of importance for various transition metal catalysis (Miller *et al.*, 1999; Xavier *et al.*, 2004). We have recently prepared the title compound (1), and report here its structure.

The single-crystal X-ray structure determination of (1) was carried out at 298 (2) K. The structure analysis showed that the compound to form in triclinic space group P-1 with a =6.888 (2) A°, b=7.381 (3) A°, c=9.638 (4) A° and α = 101.674 (6)°, β = 96.233 (6)°, γ = 116.046 (5)° with z=1. A perspective drawing is depicted in figure 1 with the atomic numbering scheme. The C10—N3—C9, N3—C9—C8 angles are 117.9 (2)° and 121.5 (2)° respectively. The N3—C10 and N3—C9 bond lengths are 1.455 (3) A° and 1.260 (3) A° respectively. In this compound (1), the short (C–)H⋯N contacts are responsible for the stability of layer structure (figure 3) which extends along the *a* axis (Taylor & Kennard, 1982). The (C–)H⋯N distances and C—H—N angles are given in table 1.

Experimental

A hot solution of ethylenediamine (1 mmol) in methanol (25 ml) was slowly added over a hot solution of quinoxaline-2-carboxaldehyde (2 mmol) in the same solvent (50 ml). The resulting mixture on cooling yielded the crude product. The precipitated diimine was filtered off and washed with cold methanol. Light yellow single crystals of (1) were obtained from a solution of dichloromethane by slow evaporation.

Refinement

H atoms were positioned geometrically with, C—H = 0.93 A° and refined in riding mode with U_{iso} (H) = 1.2Ueq(C).
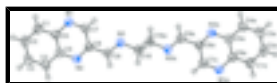
Figures

Fig. 1. An *ORTEP-3* (Farrugia, 1997) plot of the (I) compound, with the atomic labelling scheme. The shapes of the ellipsoids correspond to 50% probability contours of atomic displacement.
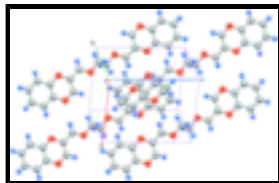
supplementary materials

Fig. 2. Unit cell packing diagram of *N,N'*-bis[(*E*)-quinoxalin-2-ylmethylidene]ethane-1,2-diamine.
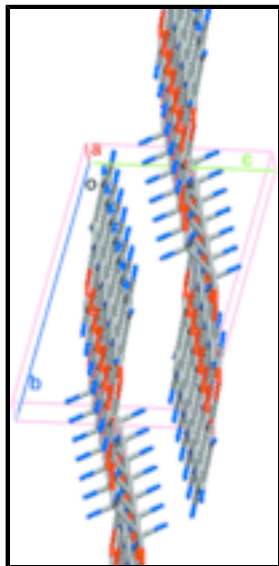

Fig. 3. Pairs of (C–)H⋯N interactions lead to a layer structure along the *a* axis.

N,N'-Bis[(*E*)-quinoxalin-2-ylmethylidene]ethane-1,2-diamine

Crystal data

$C_{20}H_{16}N_6$	$Z = 1$
$M_r = 340.39$	$F_{000} = 178$
Triclinic, $P\bar{1}$	$D_x = 1.345$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 6.888$ (2) Å	Cell parameters from 1465 reflections
$b = 7.381$ (3) Å	$\theta = 2.3–25.0°$
$c = 9.638$ (4) Å	$\mu = 0.09$ mm^{-1}
$\alpha = 101.674$ (6)°	$T = 298$ (2) K
$\beta = 96.233$ (6)°	Plate, yellow
$\gamma = 116.046$ (5)°	$0.40 \times 0.24 \times 0.18$ mm
$V = 420.1$ (3) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	1465 independent reflections
Radiation source: fine-focus sealed tube	1239 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.025$
$T = 298(2)$ K	$\theta_{max} = 25.0°$
φ and ω scans	$\theta_{min} = 2.2°$

Absorption correction: multi-scan (SADABS; Sheldrick, 2001)	$h = -8 \rightarrow 8$	
$T_{min} = 0.967$, $T_{max} = 0.995$	$k = -8 \rightarrow 8$	
3956 measured reflections	$l = -11 \rightarrow 11$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.071$	H-atom parameters constrained
$wR(F^2) = 0.164$	$w = 1/[\sigma^2(F_o^2) + (0.0542P)^2 + 0.1572P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.27$	$(\Delta/\sigma)_{max} < 0.001$
1465 reflections	$\Delta\rho_{max} = 0.13$ e Å^{-3}
118 parameters	$\Delta\rho_{min} = -0.21$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > \sigma(F^2)$ 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	0.4604 (3)	0.2446 (3)	0.5782 (2)	0.0439 (6)
N2	0.0673 (3)	0.2571 (3)	0.4735 (2)	0.0405 (6)
N3	0.0679 (3)	0.3587 (3)	0.8476 (2)	0.0425 (6)
C1	0.5203 (4)	0.1943 (4)	0.3355 (3)	0.0482 (7)
H1	0.6488	0.1881	0.3682	0.058*
C2	0.4561 (5)	0.1772 (4)	0.1921 (3)	0.0518 (8)
H2	0.5425	0.1608	0.1280	0.062*
C3	0.2624 (5)	0.1841 (4)	0.1406 (3)	0.0522 (8)
H3	0.2216	0.1735	0.0429	0.063*
C4	0.1338 (4)	0.2062 (4)	0.2329 (3)	0.0473 (7)
H4	0.0032	0.2070	0.1975	0.057*
C5	0.1963 (4)	0.2279 (4)	0.3820 (3)	0.0374 (6)
C6	0.3918 (4)	0.2210 (4)	0.4334 (3)	0.0381 (6)
C7	0.3349 (4)	0.2733 (4)	0.6617 (3)	0.0429 (7)

H7	0.3764	0.2897	0.7604	0.051*
C8	0.1382 (4)	0.2813 (4)	0.6116 (3)	0.0371 (6)
C9	0.0064 (4)	0.3217 (4)	0.7124 (3)	0.0419 (6)
H9	−0.1251	0.3198	0.6754	0.050*
C10	−0.0707 (4)	0.3998 (4)	0.9395 (3)	0.0438 (7)
H10A	−0.1415	0.2835	0.9804	0.053*
H10B	−0.1857	0.4133	0.8820	0.053*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0419 (12)	0.0551 (14)	0.0405 (12)	0.0272 (11)	0.0095 (10)	0.0150 (10)
N2	0.0409 (12)	0.0465 (13)	0.0350 (12)	0.0231 (11)	0.0064 (9)	0.0084 (10)
N3	0.0463 (13)	0.0504 (13)	0.0342 (12)	0.0257 (11)	0.0108 (9)	0.0113 (10)
C1	0.0440 (15)	0.0514 (17)	0.0495 (16)	0.0233 (14)	0.0136 (13)	0.0113 (13)
C2	0.0565 (18)	0.0512 (17)	0.0433 (16)	0.0210 (14)	0.0221 (13)	0.0093 (13)
C3	0.0596 (18)	0.0568 (18)	0.0332 (14)	0.0228 (15)	0.0095 (13)	0.0101 (13)
C4	0.0497 (16)	0.0554 (18)	0.0345 (14)	0.0243 (14)	0.0039 (12)	0.0122 (12)
C5	0.0375 (14)	0.0327 (13)	0.0385 (14)	0.0138 (11)	0.0077 (11)	0.0098 (11)
C6	0.0380 (13)	0.0377 (14)	0.0372 (13)	0.0173 (12)	0.0074 (11)	0.0096 (11)
C7	0.0448 (15)	0.0524 (16)	0.0313 (13)	0.0233 (13)	0.0052 (11)	0.0127 (12)
C8	0.0368 (13)	0.0367 (14)	0.0357 (14)	0.0173 (11)	0.0061 (11)	0.0071 (11)
C9	0.0414 (15)	0.0457 (16)	0.0406 (15)	0.0229 (13)	0.0076 (12)	0.0113 (12)
C10	0.0443 (15)	0.0528 (17)	0.0368 (14)	0.0235 (13)	0.0139 (11)	0.0139 (12)

Geometric parameters (Å, °)

N1—C7	1.298 (3)	C3—H3	0.9300
N1—C6	1.373 (3)	C4—C5	1.410 (3)
N2—C8	1.315 (3)	C4—H4	0.9300
N2—C5	1.369 (3)	C5—C6	1.409 (3)
N3—C9	1.260 (3)	C7—C8	1.418 (3)
N3—C10	1.455 (3)	C7—H7	0.9300
C1—C2	1.367 (4)	C8—C9	1.472 (3)
C1—C6	1.404 (3)	C9—H9	0.9300
C1—H1	0.9300	C10—C10^{i}	1.512 (5)
C2—C3	1.398 (4)	C10—H10A	0.9700
C2—H2	0.9300	C10—H10B	0.9700
C3—C4	1.357 (4)		
C7—N1—C6	115.8 (2)	N1—C6—C1	119.7 (2)
C8—N2—C5	116.2 (2)	N1—C6—C5	120.9 (2)
C9—N3—C10	117.9 (2)	C1—C6—C5	119.4 (2)
C2—C1—C6	119.9 (3)	N1—C7—C8	124.0 (2)
C2—C1—H1	120.1	N1—C7—H7	118.0
C6—C1—H1	120.1	C8—C7—H7	118.0
C1—C2—C3	121.0 (3)	N2—C8—C7	121.6 (2)
C1—C2—H2	119.5	N2—C8—C9	116.9 (2)
C3—C2—H2	119.5	C7—C8—C9	121.5 (2)

C4—C3—C2	120.1 (3)	N3—C9—C8	121.5 (2)
C4—C3—H3	120.0	N3—C9—H9	119.3
C2—C3—H3	120.0	C8—C9—H9	119.3
C3—C4—C5	120.7 (3)	N3—C10—C10	109.5 (3)
C3—C4—H4	119.7	N3—C10—H10A	109.8
C5—C4—H4	119.7	C10—C10—H10A	109.8
N2—C5—C6	121.6 (2)	N3—C10—H10B	109.8
N2—C5—C4	119.4 (2)	C10—C10—H10B	109.8
C6—C5—C4	119.0 (2)	H10A—C10—H10B	108.2

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

Hydrogen-bond geometry (Å, °)

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
C1—H1⋯N2[ii]	0.93	2.73	3.647 (4)	168
C9—H9⋯N1[iii]	0.93	2.67	3.593 (3)	169

Symmetry codes: (ii) $x+1, y, z$; (iii) $x-1, y, z$.

Fig. 1

Fig. 2

Fig. 3