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1,4-Bis(3-pyridylmethylenamino-methyl)benzene

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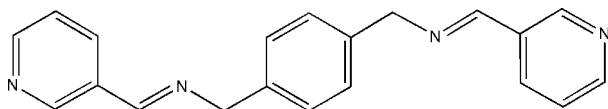
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 Key indicators: single-crystal X-ray study; $T = 291$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.048; wR factor = 0.101; data-to-parameter ratio = 15.2.

The title compound, $\text{C}_{20}\text{H}_{18}\text{N}_4$, is a flexible 3,3'-bipyridyl-type ligand with a long spacer group between the two pyridyl functions. The molecule crystallizes around an inversion center, with one half-molecule in the asymmetric unit and a dihedral angle of $71.85(8)^\circ$ between the pyridine ring and the central benzene ring.

Related literature

For background information on bipyridyl-type Schiff base ligands, see: Cho *et al.* (2006); Haga *et al.* (1985); Mahmoudi *et al.* (2007); Wang *et al.* (2008). Haga *et al.* (1985) describe the synthesis of the title compound.



Experimental

Crystal data

$\text{C}_{20}\text{H}_{18}\text{N}_4$	$V = 842.5(3) \text{ \AA}^3$
$M_r = 314.38$	$Z = 2$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 6.0990(11) \text{ \AA}$	$\mu = 0.08 \text{ mm}^{-1}$
$b = 14.589(3) \text{ \AA}$	$T = 291(2) \text{ K}$
$c = 9.9481(18) \text{ \AA}$	$0.24 \times 0.22 \times 0.20 \text{ mm}$
$\beta = 107.851(3)^\circ$	

Data collection

Bruker SMART APEX CCD diffractometer	6535 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 2000)	1661 independent reflections
$T_{\min} = 0.98$, $T_{\max} = 0.98$	1085 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.045$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$	109 parameters
$wR(F^2) = 0.101$	H-atom parameters constrained
$S = 1.01$	$\Delta\rho_{\text{max}} = 0.13 \text{ e \AA}^{-3}$
1661 reflections	$\Delta\rho_{\text{min}} = -0.12 \text{ e \AA}^{-3}$

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2166).

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supporting information

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1,4-Bis(3-pyridylmethyleneaminomethyl)benzene

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S1. Comment

Bipyridyl-type bidentate Schiff base ligands have been utilized intensively to assemble various coordination polymers with interesting topologies and fascinating structural diversities (Cho *et al.*, 2006; Mahmoudi *et al.*, 2007; Wang *et al.*, 2008). We report here the crystal structure of the title compound.

A perspective view of the title compound, including the atomic numbering scheme, is shown in Fig. 1. The title compound crystallizes around a crystallographic center with half a molecule in the asymmetric unit. The bond lengths and angles are within normal ranges. The terminal pyridyl groups are coplanar, and they form a dihedral angle of 71.85 (8)° with the central benzene ring. The molecular structure is stabilized by an intramolecular C9—H9···N2 bond (Table 1), but no classical intermolecular interactions have been found in the crystal packing of the title compound.

S2. Experimental

The title compound was synthesized and purified according to the method described by Haga *et al.* (1985), by the condensation reaction of pyridine-3-carboxaldehyde and 1,4-benzenedimethanamine (yield 83%). Colorless block single crystals (m.p. 397–397.2 K) suitable for X-ray analysis were obtained by slow evaporation of a methanol solution at room temperature. Analysis calculated for C₂₀H₁₈N₄: C 76.41, H 5.77, N 17.82%; found: C 76.53, H 5.74, N 17.75%. IR (KBr pellet, cm⁻¹): 3436 (*b*), 3060 (*m*), 3048 (*m*), 2942 (*m*), 2903 (*m*), 2849 (*m*), 1640 (*s*), 1586 (*s*), 1565 (*s*), 1469 (*s*), 1434 (*s*), 1359 (*m*), 1324 (*m*), 1150 (*w*), 1015 (*m*), 990 (*m*), 848 (*s*), 777 (*s*), 739 (*m*), 617 (*w*), 571 (*m*), 506 (*m*), 403 (*w*).

S3. Refinement

H atoms were assigned to calculated positions, with C—H = 0.97 (methylene) and 0.93 Å (aromatic), and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$.

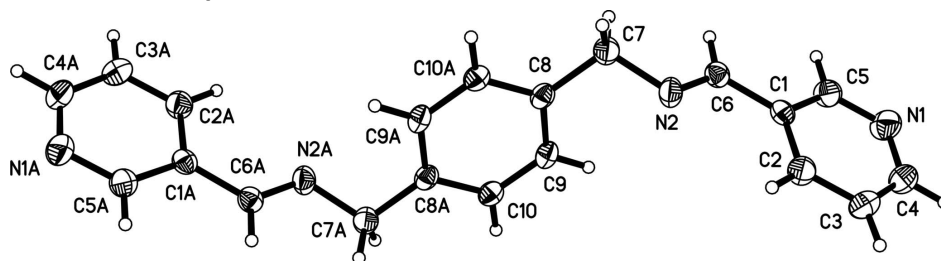


Figure 1

The molecular structure of the title compound (thermal ellipsoids are shown at 30% probability levels).

1,4-Bis(3-pyridylmethyleneaminomethyl)benzene*Crystal data*C₂₀H₁₈N₄ $M_r = 314.38$ Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

 $a = 6.0990$ (11) Å $b = 14.589$ (3) Å $c = 9.9481$ (18) Å $\beta = 107.851$ (3)° $V = 842.5$ (3) Å³ $Z = 2$ $F(000) = 332$ $D_x = 1.239$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2797 reflections

 $\theta = 2.6$ – 27.2 ° $\mu = 0.08$ mm⁻¹ $T = 291$ K

Block, colorless

 $0.24 \times 0.22 \times 0.20$ mm*Data collection*

Bruker SMART APEX CCD

diffractometer

Radiation source: sealed tube

Graphite monochromator

 φ and ω scans

Absorption correction: multi-scan

(SADABS; Bruker, 2000)

 $T_{\min} = 0.98$, $T_{\max} = 0.98$

6535 measured reflections

1661 independent reflections

1085 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.045$ $\theta_{\max} = 26.0$ °, $\theta_{\min} = 2.6$ ° $h = -7 \rightarrow 7$ $k = -17 \rightarrow 17$ $l = -11 \rightarrow 12$ *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.048$ $wR(F^2) = 0.101$ $S = 1.01$

1661 reflections

109 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

 $w = 1/[\sigma^2(F_o^2) + (0.04P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.13$ e Å⁻³ $\Delta\rho_{\min} = -0.12$ e Å⁻³*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 (Å²)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.0338 (3)	0.59733 (9)	0.90988 (16)	0.0402 (4)
C2	0.2284 (3)	0.63315 (11)	1.00966 (18)	0.0512 (4)
H2	0.3749	0.6142	1.0117	0.061*
C3	0.2005 (3)	0.69698 (11)	1.1050 (2)	0.0592 (5)

H3	0.3278	0.7216	1.1728	0.071*
C4	-0.0183 (4)	0.72359 (11)	1.0983 (2)	0.0627 (5)
H4	-0.0346	0.7673	1.1625	0.075*
C5	-0.1780 (3)	0.62821 (11)	0.91503 (19)	0.0522 (4)
H5	-0.3091	0.6036	0.8506	0.063*
C6	0.0482 (3)	0.53005 (10)	0.80278 (16)	0.0418 (4)
H6	-0.0874	0.5064	0.7416	0.050*
C7	0.2348 (3)	0.43652 (11)	0.68220 (17)	0.0475 (4)
H7A	0.0771	0.4255	0.6247	0.057*
H7B	0.2981	0.3791	0.7264	0.057*
C8	0.3738 (3)	0.46922 (10)	0.58876 (15)	0.0390 (4)
C9	0.3934 (3)	0.56235 (10)	0.56217 (16)	0.0430 (4)
H9	0.3223	0.6050	0.6044	0.052*
C10	0.5155 (3)	0.59265 (10)	0.47492 (17)	0.0424 (4)
H10	0.5239	0.6551	0.4585	0.051*
N1	-0.2079 (3)	0.69096 (10)	1.00596 (19)	0.0667 (5)
N2	0.2369 (2)	0.50359 (9)	0.79117 (15)	0.0489 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0437 (9)	0.0402 (7)	0.0394 (9)	-0.0009 (6)	0.0165 (7)	0.0070 (6)
C2	0.0458 (10)	0.0571 (10)	0.0500 (10)	0.0002 (8)	0.0134 (8)	-0.0032 (8)
C3	0.0643 (12)	0.0516 (10)	0.0579 (12)	-0.0079 (8)	0.0134 (9)	-0.0118 (8)
C4	0.0786 (14)	0.0431 (9)	0.0738 (13)	-0.0012 (9)	0.0345 (11)	-0.0131 (9)
C5	0.0470 (9)	0.0501 (9)	0.0600 (11)	0.0011 (8)	0.0169 (8)	-0.0034 (8)
C6	0.0425 (9)	0.0458 (8)	0.0374 (9)	-0.0011 (6)	0.0127 (7)	0.0029 (6)
C7	0.0467 (9)	0.0508 (8)	0.0462 (9)	0.0005 (7)	0.0161 (7)	-0.0041 (7)
C8	0.0379 (8)	0.0423 (7)	0.0333 (9)	0.0013 (6)	0.0055 (6)	-0.0063 (6)
C9	0.0464 (9)	0.0423 (7)	0.0414 (9)	0.0065 (7)	0.0149 (7)	-0.0081 (7)
C10	0.0477 (9)	0.0346 (7)	0.0442 (9)	0.0017 (6)	0.0129 (7)	-0.0025 (6)
N1	0.0609 (10)	0.0550 (9)	0.0887 (13)	0.0041 (8)	0.0297 (9)	-0.0173 (8)
N2	0.0478 (8)	0.0624 (9)	0.0393 (8)	0.0002 (6)	0.0175 (6)	-0.0074 (6)

Geometric parameters (Å, °)

C1—C5	1.384 (2)	C6—H6	0.9300
C1—C2	1.395 (2)	C7—N2	1.458 (2)
C1—C6	1.471 (2)	C7—C8	1.514 (2)
C2—C3	1.376 (2)	C7—H7A	0.9700
C2—H2	0.9300	C7—H7B	0.9700
C3—C4	1.372 (3)	C8—C10 ⁱ	1.391 (2)
C3—H3	0.9300	C8—C9	1.396 (2)
C4—N1	1.325 (3)	C9—C10	1.378 (2)
C4—H4	0.9300	C9—H9	0.9300
C5—N1	1.338 (2)	C10—C8 ⁱ	1.391 (2)
C5—H5	0.9300	C10—H10	0.9300
C6—N2	1.253 (2)		

C5—C1—C2	116.88 (15)	N2—C7—C8	111.49 (13)
C5—C1—C6	120.51 (15)	N2—C7—H7A	109.3
C2—C1—C6	122.61 (14)	C8—C7—H7A	109.3
C3—C2—C1	119.09 (16)	N2—C7—H7B	109.3
C3—C2—H2	120.5	C8—C7—H7B	109.3
C1—C2—H2	120.5	H7A—C7—H7B	108.0
C4—C3—C2	118.78 (18)	C10 ⁱ —C8—C9	117.60 (13)
C4—C3—H3	120.6	C10 ⁱ —C8—C7	121.10 (13)
C2—C3—H3	120.6	C9—C8—C7	121.30 (12)
N1—C4—C3	124.15 (17)	C10—C9—C8	121.72 (13)
N1—C4—H4	117.9	C10—C9—H9	119.1
C3—C4—H4	117.9	C8—C9—H9	119.1
N1—C5—C1	124.74 (18)	C9—C10—C8 ⁱ	120.68 (13)
N1—C5—H5	117.6	C9—C10—H10	119.7
C1—C5—H5	117.6	C8 ⁱ —C10—H10	119.7
N2—C6—C1	122.21 (15)	C4—N1—C5	116.34 (16)
N2—C6—H6	118.9	C6—N2—C7	118.48 (14)
C1—C6—H6	118.9		

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

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C9—H9 \cdots N2	0.93	2.55	2.858 (2)	100