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# 1,4-Bis(3-pyridylmethyleneamino-methyl)benzene

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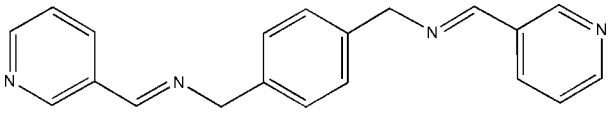
Key indicators: single-crystal X-ray study; $T$ = 291 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.048; $wR$ factor = 0.101; data-to-parameter ratio = 15.2.

The title compound, $C_{20}H_{18}N_4$, 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)° 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*

$C_{20}H_{18}N_4$
$M_r$ = 314.38
Monoclinic, $P2_1/c$
$a$ = 6.0990 (11) Å
$b$ = 14.589 (3) Å
$c$ = 9.9481 (18) Å
$\beta$ = 107.851 (3)°
$V$ = 842.5 (3) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 291 (2) K
0.24 × 0.22 × 0.20 mm

*Data collection*

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

*Refinement*

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

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).

## References

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# supplementary materials

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# 1,4-Bis(3-pyridylmethyleneaminomethyl)benzene

**M.-Y. He, C. Li, H. Xu, Z.-J. Hu and Q. Chen**

## 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.

## Experimental

The title compound was synthesized and purified according to the method described by 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 calclated for $C_{20}H_{18}N_4$: 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*).

## Refinement

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

## Figures

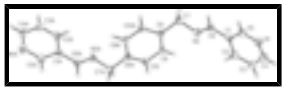


Fig. 1. The molecular structure of the title compound (thermal ellipsoids are shown at 30% probability levels).

## 1,4-Bis(3-pyridylmethyleneaminomethyl)benzene

*Crystal data*

| | |
|---|---|
| $C_{20}H_{18}N_4$ | $F_{000}$ = 332 |
| $M_r$ = 314.38 | $D_x$ = 1.239 Mg $m^{-3}$ |

| | |
|---|---|
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation<br>$\lambda = 0.71073$ Å |
| Hall symbol: -P 2ybc | Cell parameters from 2797 reflections |
| $a = 6.0990$ (11) Å | $\theta = 2.6–27.2°$ |
| $b = 14.589$ (3) Å | $\mu = 0.08$ mm$^{-1}$ |
| $c = 9.9481$ (18) Å | $T = 291$ (2) K |
| $\beta = 107.851$ (3)° | Block, colorless |
| $V = 842.5$ (3) Å$^3$ | 0.24 × 0.22 × 0.20 mm |
| $Z = 2$ | |

*Data collection*

| | |
|---|---|
| Bruker SMART APEX CCD diffractometer | 1661 independent reflections |
| Radiation source: sealed tube | 1085 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int} = 0.045$ |
| $T = 291(2)$ K | $\theta_{max} = 26.0°$ |
| $\varphi$ and $\omega$ scans | $\theta_{min} = 2.6°$ |
| Absorption correction: multi-scan (SADABS; Bruker, 2000) | $h = -7\rightarrow7$ |
| $T_{min} = 0.98$, $T_{max} = 0.98$ | $k = -17\rightarrow17$ |
| 6535 measured reflections | $l = -11\rightarrow12$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.048$ | H-atom parameters constrained |
| $wR(F^2) = 0.101$ | $w = 1/[\sigma^2(F_o^2) + (0.04P)^2]$<br>where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.01$ | $(\Delta/\sigma)_{max} < 0.001$ |
| 1661 reflections | $\Delta\rho_{max} = 0.13$ e Å$^{-3}$ |
| 109 parameters | $\Delta\rho_{min} = -0.12$ 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$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| 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 (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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 |

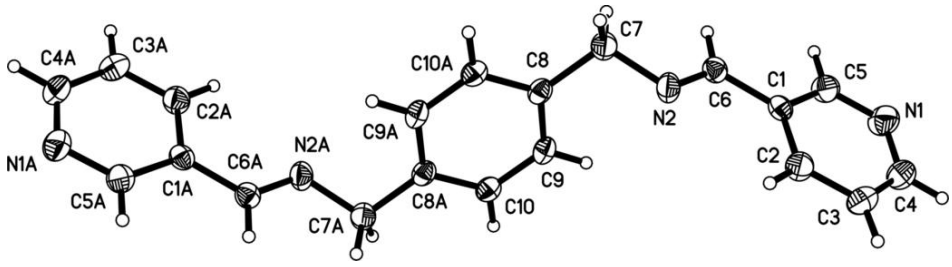
| | | | |
|---|---|---|---|
| C3—C4 | 1.372 (3) | C8—C10$^{i}$ | 1.391 (2) |
| C3—H3 | 0.9300 | C8—C9 | 1.396 (2) |
| C4—N1 | 1.325 (3) | C9—C10 | 1.378 (2) |
| C4—H4 | 0.9300 | C9—H9 | 0.9300 |
| C5—N1 | 1.338 (2) | C10—C8$^{i}$ | 1.391 (2) |
| C5—H5 | 0.9300 | C10—H10 | 0.9300 |
| C6—N2 | 1.253 (2) | | |
| 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 codes: (i) $-x+1, -y+1, -z+1$.

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C9—H9···N2 | 0.93 | 2.55 | 2.858 (2) | 100 |

**Fig. 1**


C4A
C3A
C2A
N1A
C5A
C1A
C6A
N2A
C7A
C10A
C9A
C8
C8A
C9
C10
C7
N2
C6
C1
C5
N1
C2
C3
C4