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N,*N*′-Bis(4-pyridylmethylene)benzene-1,4-diamine monohydrate

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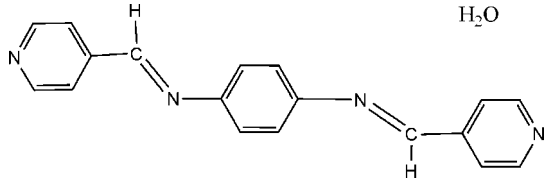
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.066; wR factor = 0.230; data-to-parameter ratio = 13.1.

In the title compound, $C_{18}H_{14}N_4\cdot H_2O$, the central benzene ring makes dihedral angles of 43.80 (14) and 24.94 (16)° with the two pyridine rings. The water molecule bridges benzene-1,4-diamine molecules *via* O—H···N hydrogen bonds, resulting in the formation of linear chains extending along the [20$\bar{1}$] direction.

Related literature

For general background, see: Yang *et al.* (2000); Mondal *et al.* (2001); Tarafder *et al.* (2002).



Experimental

Crystal data

$C_{18}H_{16}N_4O$
M_r = 304.35
Triclinic, $P\bar{1}$
a = 8.4780 (12) Å
b = 9.2472 (15) Å
c = 11.291 (2) Å
α = 109.914 (3)°
β = 94.884 (2)°
γ = 102.663 (3)°
V = 799.6 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 298 (2) K
0.52 × 0.34 × 0.18 mm

Data collection

Siemens SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.959, T_{max} = 0.985
4033 measured reflections
2718 independent reflections
1439 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.231
S = 1.01
2718 reflections
208 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1B···N4^{i}	0.85	2.20	3.005 (4)	158
O1—H1A···N3^{ii}	0.85	2.16	2.967 (4)	158

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); 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: CV2345).

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supplementary materials

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N,*N*'-Bis(4-pyridylmethylene)benzene-1,4-diamine monohydrate

Q. Wang, D.-Q. Wang and Y.-Y. Sun

Comment

Schiff bases have been intensively investigated recently owing to their strong coordination capability and diverse biological activities, such as antibacterial, antitumor activities *etc* (Yang *et al.*, 2000; Mondal *et al.*, 2001; Tarafder *et al.*, 2002). In this paper, we present a title compound, *N*,*N*'-Bis(4-pyridylmethylene)benzene-1,4-diamine monohydrate, synthesized by a condensation reaction of pyridine-4-carboxaldehyde with benzene-1,4-diamine in refluxing eathnol.

The molecular structure of the title compound is shown in Fig.1. This compound contains one Schiff base ligand (I) and one water molecule. The central benzene ring of (I) makes the dihedral angles of 43.80 (14) ° and 24.94 (16) °, respectively, with two outer pyridine rings. The relevant dihedral angle between the pyridine rings is 68.59(0.15) °.

The intermolecular O—H···N hydrogen bonds (Table 1) link the molecules of (I) and crystalline water molecules into linear chains extending in direction [20–1] (Fig. 2).

Experimental

Pyridine-4-carboxaldehyde (10 mmol, 1162 mg) in absolute ethanol (5 ml) was added dropwise to a absolute ethanol solution (15 ml) of benzene-1,4-diamine (5 mmol, 540.7 mg). The mixture was heated under reflux with stirring for 4 h and then filtered. The resulting clear pale yellow solution was diffused diethyl ether vapor at room temperature for two weeks, after which large yellow block-shaped crystals of the title complex suitable for X-ray diffraction analysis were obtained.

Refinement

All H-atoms were positioned geometrically and refined using a riding model, with C—H (methylene) 0.93, C—H (aromatic) 0.93, O—H 0.85 Å (water), and with U_{iso}(H) =1.2U_{eq}(C, O).

Figures

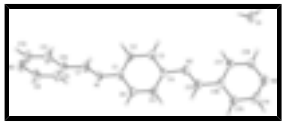

Fig. 1. The structure of the title complex, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

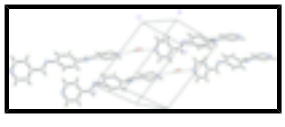

Fig. 2. A portion of the crystal packing of the title complex showing hydrogen bonds as dashed lines.

N,*N*'-Bis(4-pyridylmethylene)benzene-1,4-diamine monohydrate

Crystal data

$C_{18}H_{16}N_4O$	$Z = 2$
$M_r = 304.35$	$F_{000} = 320$
Triclinic, $P\overline{1}$	$D_x = 1.264$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 8.4780$ (12) Å	Cell parameters from 1119 reflections
$b = 9.2472$ (15) Å	$\theta = 2.4$–$27.0°$
$c = 11.291$ (2) Å	$\mu = 0.08$ mm^{-1}
$\alpha = 109.914$ (3)°	$T = 298$ (2) K
$\beta = 94.884$ (2)°	Block, yellow
$\gamma = 102.663$ (3)°	0.52 × 0.34 × 0.18 mm
$V = 799.6$ (2) Å^3	

Data collection

Siemens SMART CCD area-detector diffractometer	2718 independent reflections
Radiation source: fine-focus sealed tube	1439 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.038$
$T = 298(2)$ K	$\theta_{max} = 25.0°$
φ and ω scans	$\theta_{min} = 2.0°$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -4 \rightarrow 10$
$T_{min} = 0.959$, $T_{max} = 0.985$	$k = -10 \rightarrow 9$
4033 measured reflections	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.066$	H-atom parameters constrained
$wR(F^2) = 0.231$	$w = 1/[\sigma^2(F_o^2) + (0.1265P)^2 + 0.0065P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} < 0.001$
2718 reflections	$\Delta\rho_{max} = 0.19$ e Å^{-3}
208 parameters	$\Delta\rho_{min} = -0.24$ 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 $F^2 > 2\text{sigma}(F^2)$ 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	−0.1455 (3)	0.2126 (3)	1.0021 (2)	0.0530 (7)
N2	0.4300 (3)	0.2611 (3)	0.7853 (2)	0.0576 (8)
N3	−0.5784 (4)	0.2933 (4)	1.2834 (3)	0.0779 (10)
N4	0.8932 (3)	0.1819 (4)	0.5373 (3)	0.0672 (9)
O1	0.8862 (3)	0.6807 (3)	0.6144 (2)	0.0868 (9)
H1A	0.7989	0.7064	0.6338	0.104*
H1B	0.9292	0.7337	0.5714	0.104*
C1	0.0031 (4)	0.2242 (4)	0.9535 (3)	0.0490 (8)
C2	0.1255 (4)	0.3655 (4)	0.9904 (3)	0.0545 (9)
H2	0.1141	0.4557	1.0545	0.065*
C3	0.2647 (4)	0.3757 (4)	0.9339 (3)	0.0570 (9)
H3	0.3436	0.4725	0.9577	0.068*
C4	0.2851 (4)	0.2389 (4)	0.8409 (3)	0.0519 (8)
C5	0.1640 (4)	0.0977 (4)	0.8052 (3)	0.0610 (9)
H5	0.1769	0.0060	0.7437	0.073*
C6	0.0243 (4)	0.0905 (4)	0.8594 (3)	0.0578 (9)
H6	−0.0569	−0.0053	0.8325	0.069*
C7	−0.1438 (4)	0.2861 (4)	1.1190 (3)	0.0575 (9)
H7	−0.0436	0.3433	1.1726	0.069*
C8	−0.5868 (5)	0.2162 (5)	1.1575 (4)	0.0745 (11)
H8	−0.6894	0.1632	1.1064	0.089*
C9	−0.4478 (4)	0.2128 (4)	1.1010 (3)	0.0621 (10)
H9	−0.4587	0.1614	1.0131	0.075*
C10	−0.2960 (4)	0.2842 (4)	1.1736 (3)	0.0541 (9)
C11	−0.2877 (5)	0.3637 (5)	1.3026 (4)	0.0874 (14)
H11	−0.1865	0.4173	1.3558	0.105*
C12	−0.4301 (6)	0.3629 (5)	1.3517 (4)	0.0894 (14)
H12	−0.4213	0.4152	1.4394	0.107*
C13	0.4910 (4)	0.1473 (4)	0.7382 (3)	0.0587 (9)
H13	0.4452	0.0505	0.7457	0.070*
C14	0.8149 (5)	0.0460 (5)	0.5445 (4)	0.0709 (11)
H14	0.8495	−0.0444	0.5036	0.085*
C15	0.6844 (4)	0.0294 (4)	0.6091 (3)	0.0622 (9)

H15	0.6332	−0.0700	0.6099	0.075*
C16	0.6313 (4)	0.1596 (4)	0.6715 (3)	0.0526 (8)
C17	0.7126 (4)	0.3042 (4)	0.6648 (3)	0.0648 (10)
H17	0.6804	0.3963	0.7050	0.078*
C18	0.8414 (4)	0.3094 (4)	0.5978 (4)	0.0712 (11)
H18	0.8949	0.4069	0.5948	0.085*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0433 (15)	0.0684 (17)	0.0521 (16)	0.0176 (13)	0.0251 (12)	0.0226 (13)
N2	0.0442 (16)	0.0711 (18)	0.0602 (16)	0.0175 (14)	0.0248 (13)	0.0225 (14)
N3	0.070 (2)	0.105 (3)	0.093 (2)	0.049 (2)	0.052 (2)	0.055 (2)
N4	0.0625 (19)	0.080 (2)	0.0762 (19)	0.0290 (17)	0.0417 (16)	0.0374 (16)
O1	0.084 (2)	0.0951 (19)	0.109 (2)	0.0440 (16)	0.0549 (17)	0.0503 (16)
C1	0.0373 (17)	0.070 (2)	0.0441 (16)	0.0202 (16)	0.0166 (13)	0.0201 (15)
C2	0.0465 (19)	0.065 (2)	0.0546 (18)	0.0261 (16)	0.0204 (15)	0.0153 (16)
C3	0.0466 (19)	0.063 (2)	0.065 (2)	0.0182 (16)	0.0202 (16)	0.0231 (17)
C4	0.0421 (18)	0.072 (2)	0.0497 (17)	0.0206 (17)	0.0217 (15)	0.0261 (16)
C5	0.060 (2)	0.074 (2)	0.0481 (18)	0.0259 (18)	0.0277 (16)	0.0118 (16)
C6	0.0480 (19)	0.061 (2)	0.0530 (18)	0.0031 (15)	0.0212 (15)	0.0116 (16)
C7	0.0429 (19)	0.078 (2)	0.0535 (19)	0.0172 (16)	0.0183 (15)	0.0236 (17)
C8	0.053 (2)	0.110 (3)	0.082 (3)	0.026 (2)	0.0283 (19)	0.055 (2)
C9	0.052 (2)	0.086 (2)	0.057 (2)	0.0187 (19)	0.0235 (17)	0.0347 (18)
C10	0.050 (2)	0.076 (2)	0.0494 (18)	0.0296 (17)	0.0230 (15)	0.0271 (16)
C11	0.059 (2)	0.125 (3)	0.061 (2)	0.020 (2)	0.0273 (19)	0.012 (2)
C12	0.073 (3)	0.118 (4)	0.072 (3)	0.031 (3)	0.043 (2)	0.020 (2)
C13	0.055 (2)	0.072 (2)	0.063 (2)	0.0245 (18)	0.0289 (17)	0.0326 (17)
C14	0.075 (3)	0.090 (3)	0.080 (2)	0.052 (2)	0.045 (2)	0.045 (2)
C15	0.057 (2)	0.066 (2)	0.083 (2)	0.0244 (17)	0.0347 (19)	0.0407 (18)
C16	0.0442 (18)	0.074 (2)	0.0482 (17)	0.0225 (17)	0.0178 (15)	0.0269 (16)
C17	0.057 (2)	0.065 (2)	0.068 (2)	0.0190 (17)	0.0289 (18)	0.0125 (17)
C18	0.069 (2)	0.064 (2)	0.086 (3)	0.0170 (19)	0.045 (2)	0.0277 (19)

Geometric parameters (Å, °)

N1—C7	1.260 (4)	C7—C10	1.476 (4)
N1—C1	1.413 (4)	C7—H7	0.9300
N2—C13	1.249 (4)	C8—C9	1.388 (4)
N2—C4	1.433 (4)	C8—H8	0.9300
N3—C12	1.316 (5)	C9—C10	1.357 (5)
N3—C8	1.342 (5)	C9—H9	0.9300
N4—C14	1.319 (5)	C10—C11	1.378 (5)
N4—C18	1.334 (4)	C11—C12	1.370 (5)
O1—H1A	0.8500	C11—H11	0.9300
O1—H1B	0.8500	C12—H12	0.9300
C1—C2	1.385 (4)	C13—C16	1.465 (4)
C1—C6	1.387 (4)	C13—H13	0.9300
C2—C3	1.390 (4)	C14—C15	1.385 (4)

C2—H2	0.9300	C14—H14	0.9300
C3—C4	1.398 (4)	C15—C16	1.365 (4)
C3—H3	0.9300	C15—H15	0.9300
C4—C5	1.381 (5)	C16—C17	1.393 (5)
C5—C6	1.377 (4)	C17—C18	1.382 (4)
C5—H5	0.9300	C17—H17	0.9300
C6—H6	0.9300	C18—H18	0.9300
C7—N1—C1	119.8 (3)	C10—C9—H9	119.8
C13—N2—C4	120.5 (3)	C8—C9—H9	119.8
C12—N3—C8	116.3 (3)	C9—C10—C11	117.2 (3)
C14—N4—C18	116.4 (3)	C9—C10—C7	122.9 (3)
H1A—O1—H1B	108.9	C11—C10—C7	119.8 (3)
C2—C1—C6	118.1 (3)	C12—C11—C10	119.2 (4)
C2—C1—N1	123.0 (3)	C12—C11—H11	120.4
C6—C1—N1	118.8 (3)	C10—C11—H11	120.4
C1—C2—C3	121.6 (3)	N3—C12—C11	124.5 (4)
C1—C2—H2	119.2	N3—C12—H12	117.7
C3—C2—H2	119.2	C11—C12—H12	117.7
C2—C3—C4	119.4 (3)	N2—C13—C16	122.9 (3)
C2—C3—H3	120.3	N2—C13—H13	118.5
C4—C3—H3	120.3	C16—C13—H13	118.5
C5—C4—C3	119.0 (3)	N4—C14—C15	124.1 (3)
C5—C4—N2	125.3 (3)	N4—C14—H14	117.9
C3—C4—N2	115.6 (3)	C15—C14—H14	117.9
C6—C5—C4	121.0 (3)	C16—C15—C14	119.7 (3)
C6—C5—H5	119.5	C16—C15—H15	120.1
C4—C5—H5	119.5	C14—C15—H15	120.1
C5—C6—C1	120.9 (3)	C15—C16—C17	117.0 (3)
C5—C6—H6	119.5	C15—C16—C13	121.7 (3)
C1—C6—H6	119.5	C17—C16—C13	121.3 (3)
N1—C7—C10	122.0 (3)	C18—C17—C16	119.3 (3)
N1—C7—H7	119.0	C18—C17—H17	120.3
C10—C7—H7	119.0	C16—C17—H17	120.3
N3—C8—C9	122.3 (4)	N4—C18—C17	123.5 (3)
N3—C8—H8	118.8	N4—C18—H18	118.3
C9—C8—H8	118.8	C17—C18—H18	118.3
C10—C9—C8	120.3 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1B···N4^{i}	0.85	2.20	3.005 (4)	158
O1—H1A···N3^{ii}	0.85	2.16	2.967 (4)	158

Symmetry codes: (i) −*x*+2, −*y*+1, −*z*+1; (ii) −*x*, −*y*+1, −*z*+2.

Fig. 1

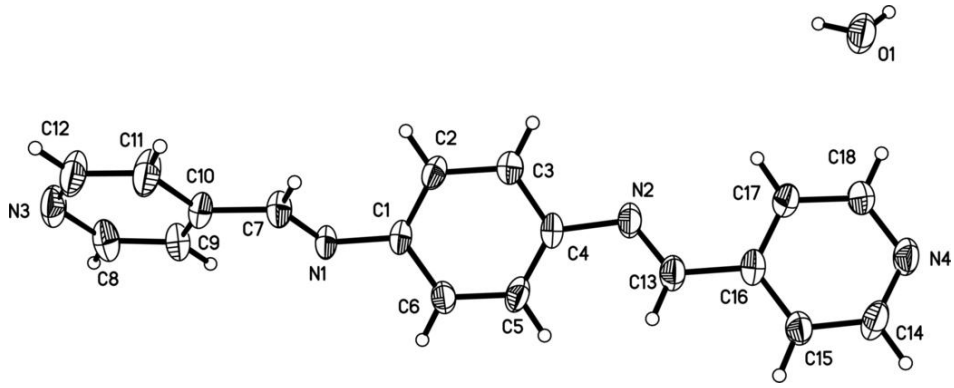
O1
C12
C11
C10
N3
C9
C8
C7
N1
C1
C2
C3
C4
N2
C13
C6
C5
C16
C17
C18
N4
C14
C15

Fig. 2