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N-[(*E*)-4-Pyridylmethylene]-4-[(*E*)-4-(4-pyridylmethyleneamino)benzyl]aniline tetrahydrate

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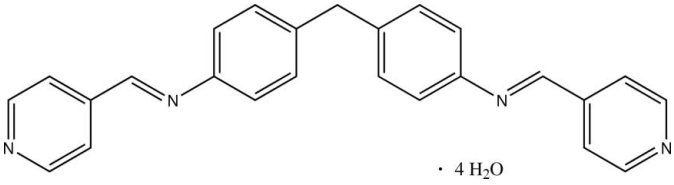
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.058; wR factor = 0.158; data-to-parameter ratio = 14.2.

The title compound, $C_{25}H_{20}N_4\cdot 4H_2O$, crystallizes with the organic molecule lying on a twofold rotation axis through the methylene bridge C atom; there are also two water molecules in the asymmetric unit. The crystal structure is stabilized by C—H···O, O—H···O and O—H···N hydrogen bonds, linking the water molecules to each other and to the pyridine N atom.

Related literature

For related literature, see: Jursic *et al.* (2002); Coucouvanis *et al.* (1993); Hodnett & Mooney (1970); Li *et al.* (2005); Yam & Pui (2002).



Experimental

Crystal data

$C_{25}H_{20}N_4\cdot 4H_2O$
M_r = 448.51
Monoclinic, $C2/c$
a = 17.7743 (14) Å
b = 4.7309 (4) Å
c = 28.478 (2) Å
β = 99.69 (1)°
V = 2360.5 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 294 (2) K
0.40 × 0.30 × 0.30 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
7552 measured reflections
2302 independent reflections
1850 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.158
S = 1.08
2302 reflections
162 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O1	0.93	2.52	3.443 (3)	175
O1—H1C···O2	0.91 (4)	1.90 (4)	2.810 (3)	178 (4)
O2—H2B···N2^{i}	0.87 (5)	1.94 (5)	2.782 (3)	163 (4)
O2—H2A···O1^{ii}	0.88 (5)	1.93 (5)	2.765 (3)	160 (4)
O1—H1D···O2^{iii}	0.91 (4)	1.88 (4)	2.799 (3)	178 (4)

Symmetry codes: (i) $-x+\frac{1}{2}, -y-\frac{1}{2}, -z+1$; (ii) $-x+1, -y+1, -z+1$; (iii) $-x+1, -y, -z+1$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2000); 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: SF2014).

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supporting information

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N-[(*E*)-4-Pyridylmethylene]-4-[(*E*)-4-(4-pyridylmethyleneamino)benzyl]aniline tetrahydrate

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S1. Comment

Schiff bases have received much attention due to their versatile properties and utilities such as anticancer properties (Hodnett & Mooney, 1970), supramolecule chemistry and molecular recognition (Coucouvanis *et al.*, 1993; Yam & Pui, 2002) and catalysis in organic reaction through their metal complexes(Li *et al.*, 2005). In this paper we report the crystal structure of the title schiff base, (I)(Fig. 1), I crystallizes with the main molecule lying on a twofold rotation axis through atom C1 and two independent water molecules in general positions. The phenyl ring C2/C3/C4/C5/C6/C7 and pyridine ring C9/C10/C11/N2/C12/C13 are *trans* with respect to C?N bond, and the dihedral angle between them is 7.21 (3)°. In the crystal structure, molecules are connected by water molecules *via* C—H···O, O—H···O, O—H···N hydrogen bonds. (Fig. 2 and Table 1). The molecular wings are approximately perpendicular to each other, and molecules pack by tucking their *exo* surfaces into the *endo* surface of the adjacent molecule along b - forming good Π stacking. The water molecules create an infinite puckered ladder along b which links to the terminal N atoms of the organic amide.

S2. Experimental

The title compound was synthesized using a method analogous to the literature procedure of Jursic *et al.* (2002), Crystals appropriate for data collection were obtained by slow evaporation from a methanol-chloroform solution (1:20 V/V)of (I).

S3. Refinement

The water probably derives from the methanol solvent used for recrystallization. The H atoms were constrained to an ideal geometry and constrained to ride on their parent atoms as follows: methylene H with d(C—H)=0.97 Å and U_{iso}(H) = 1.2U_{eq}(C); aromatic H with d(C—H)=0.93 Å and U_{iso}(H) = 1.2U_{eq}(C).

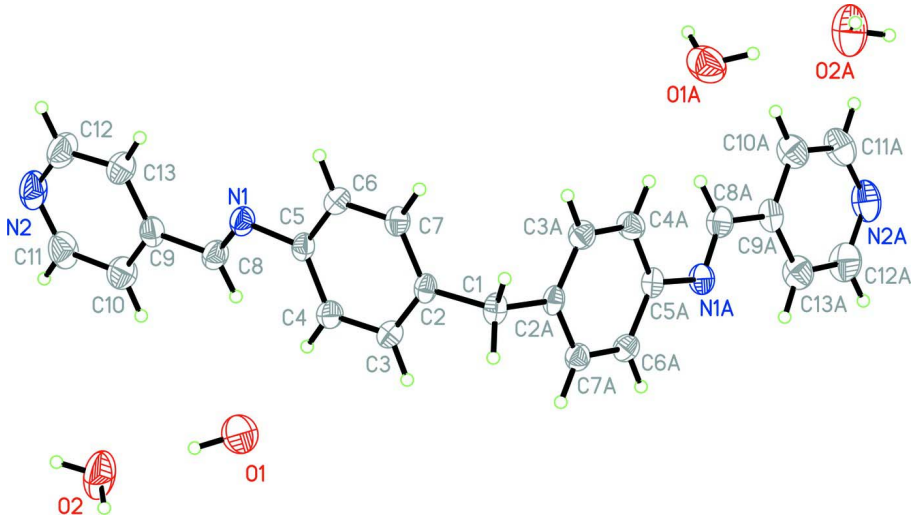

Figure 1

The molecular structure showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms atoms shown as circles of arbitrary radii.

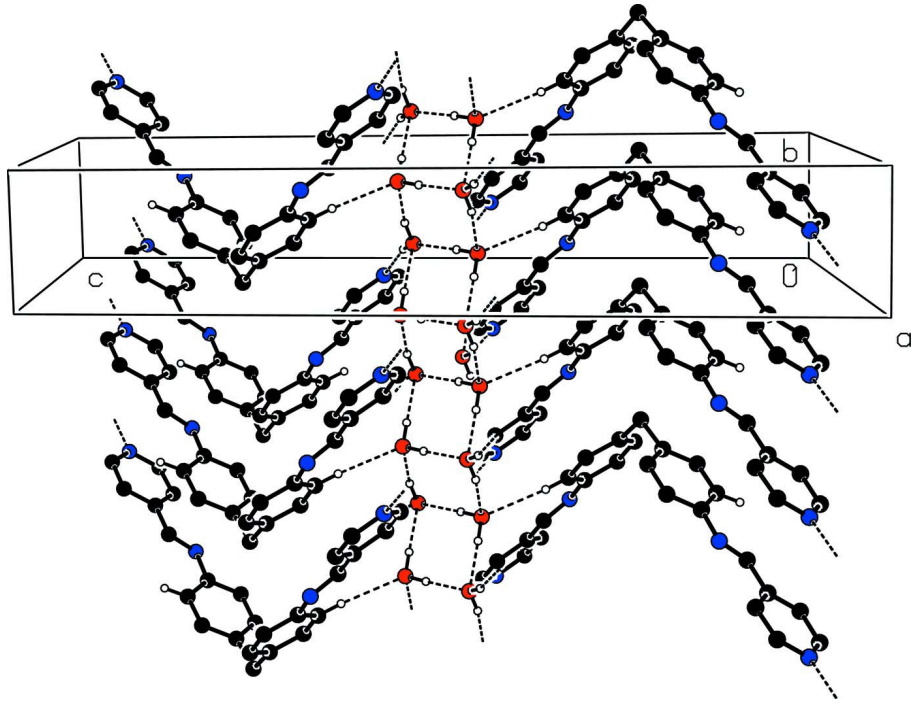

Figure 2

The molecular packing of (I), with hydrogen bonds shown as dashed lines

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Crystal data

$C_{25}H_{20}N_4 \cdot 4H_2O$
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Monoclinic, *C*2/*c*
Hall symbol: -C 2/c
$a = 17.7743\ (14)$ Å
$b = 4.7309\ (4)$ Å

c = 28.478 (2) Å
β = 99.69 (1)°
V = 2360.5 (3) Å^3
Z = 4
F(000) = 952
D_x = 1.262 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å

Cell parameters from 2376 reflections
θ = 2.3–27.4°
μ = 0.09 mm^{-1}
T = 294 K
Block, yellow
0.40 × 0.30 × 0.30 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
7552 measured reflections
2302 independent reflections

1850 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 26.0°, θ_{min} = 2.3°
h = −13→21
k = −5→5
l = −35→35

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.158
S = 1.08
2302 reflections
162 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0751P)^2 + 1.2009P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.5000	0.9902 (5)	0.2500	0.0468 (6)	
H1A	0.4695	1.1104	0.2266	0.056*	0.50
H1B	0.5305	1.1104	0.2734	0.056*	0.50
C2	0.44833 (9)	0.8053 (3)	0.27406 (6)	0.0400 (4)	
C3	0.47436 (10)	0.6805 (4)	0.31775 (6)	0.0493 (5)	
H3	0.5231	0.7234	0.3336	0.059*	
C4	0.42989 (11)	0.4944 (4)	0.33839 (7)	0.0504 (5)	
H4	0.4487	0.4148	0.3679	0.060*	
C5	0.35682 (10)	0.4251 (4)	0.31521 (6)	0.0419 (4)	
C6	0.32993 (10)	0.5543 (4)	0.27215 (6)	0.0456 (4)	

H6	0.2808	0.5141	0.2565	0.055*
C7	0.37470 (10)	0.7423 (4)	0.25190 (6)	0.0449 (4)
H7	0.3551	0.8276	0.2230	0.054*
C8	0.32973 (11)	0.0807 (4)	0.36783 (7)	0.0511 (5)
H8	0.3811	0.0887	0.3814	0.061*
C9	0.27972 (11)	−0.1144 (4)	0.38837 (6)	0.0492 (5)
C10	0.30718 (14)	−0.2665 (5)	0.42886 (8)	0.0658 (6)
H10	0.3578	−0.2467	0.4435	0.079*
C11	0.25914 (16)	−0.4484 (5)	0.44754 (9)	0.0739 (7)
H11	0.2788	−0.5490	0.4749	0.089*
C12	0.16076 (14)	−0.3398 (6)	0.38962 (10)	0.0770 (7)
H12	0.1098	−0.3633	0.3759	0.092*
C13	0.20413 (12)	−0.1544 (5)	0.36816 (8)	0.0642 (6)
H13	0.1830	−0.0579	0.3406	0.077*
N1	0.30633 (9)	0.2361 (3)	0.33305 (5)	0.0481 (4)
N2	0.18689 (13)	−0.4874 (4)	0.42866 (8)	0.0748 (6)
O1	0.49404 (13)	0.2296 (5)	0.45062 (7)	0.0944 (7)
H1C	0.472 (2)	0.249 (8)	0.4769 (14)	0.142*
H1D	0.521 (2)	0.065 (9)	0.4559 (14)	0.142*
O2	0.42372 (14)	0.2747 (5)	0.53161 (9)	0.1114 (8)
H2A	0.442 (3)	0.440 (11)	0.5419 (15)	0.167*
H2B	0.386 (3)	0.219 (10)	0.5448 (16)	0.167*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0515 (15)	0.0357 (13)	0.0585 (15)	0.000	0.0250 (12)	0.000
C2	0.0424 (9)	0.0319 (8)	0.0501 (10)	0.0024 (7)	0.0206 (7)	−0.0036 (7)
C3	0.0425 (10)	0.0537 (11)	0.0523 (10)	−0.0080 (8)	0.0102 (8)	−0.0007 (8)
C4	0.0506 (11)	0.0545 (11)	0.0463 (10)	−0.0080 (9)	0.0091 (8)	0.0062 (8)
C5	0.0434 (10)	0.0382 (9)	0.0481 (10)	−0.0037 (7)	0.0190 (7)	−0.0058 (7)
C6	0.0384 (9)	0.0460 (10)	0.0528 (10)	−0.0032 (8)	0.0089 (7)	−0.0003 (8)
C7	0.0458 (10)	0.0424 (10)	0.0487 (10)	0.0049 (8)	0.0140 (8)	0.0042 (7)
C8	0.0493 (11)	0.0475 (10)	0.0589 (11)	−0.0087 (8)	0.0159 (9)	−0.0010 (9)
C9	0.0597 (12)	0.0397 (10)	0.0530 (11)	−0.0085 (8)	0.0237 (9)	−0.0064 (8)
C10	0.0730 (14)	0.0611 (13)	0.0651 (13)	−0.0125 (11)	0.0167 (11)	0.0066 (10)
C11	0.0983 (19)	0.0629 (14)	0.0663 (14)	−0.0161 (13)	0.0303 (13)	0.0112 (11)
C12	0.0674 (14)	0.0750 (16)	0.0933 (18)	−0.0255 (12)	0.0271 (13)	0.0000 (14)
C13	0.0630 (13)	0.0626 (13)	0.0695 (14)	−0.0158 (11)	0.0179 (10)	0.0047 (10)
N1	0.0490 (9)	0.0456 (9)	0.0527 (9)	−0.0083 (7)	0.0173 (7)	0.0003 (7)
N2	0.0899 (15)	0.0609 (12)	0.0839 (14)	−0.0222 (11)	0.0440 (12)	−0.0001 (10)
O1	0.1031 (15)	0.1013 (15)	0.0787 (12)	−0.0129 (12)	0.0153 (10)	0.0224 (11)
O2	0.1177 (18)	0.0957 (16)	0.1373 (19)	−0.0295 (13)	0.0691 (15)	0.0062 (13)

Geometric parameters (Å, º)

C1—C2^{i}	1.513 (2)	C8—C9	1.469 (3)
C1—C2	1.513 (2)	C8—H8	0.9300

C1—H1A	0.9700	C9—C10	1.377 (3)
C1—H1B	0.9700	C9—C13	1.383 (3)
C2—C3	1.385 (3)	C10—C11	1.380 (3)
C2—C7	1.386 (3)	C10—H10	0.9300
C3—C4	1.379 (2)	C11—N2	1.319 (3)
C3—H3	0.9300	C11—H11	0.9300
C4—C5	1.393 (3)	C12—N2	1.329 (3)
C4—H4	0.9300	C12—C13	1.377 (3)
C5—C6	1.382 (2)	C12—H12	0.9300
C5—N1	1.420 (2)	C13—H13	0.9300
C6—C7	1.382 (2)	O1—H1C	0.91 (4)
C6—H6	0.9300	O1—H1D	0.91 (4)
C7—H7	0.9300	O2—H2A	0.88 (5)
C8—N1	1.247 (2)	O2—H2B	0.87 (5)

C2^{i}—C1—C2	109.37 (19)	C2—C7—H7	119.5
C2^{i}—C1—H1A	109.8	N1—C8—C9	122.89 (18)
C2—C1—H1A	109.8	N1—C8—H8	118.6
C2^{i}—C1—H1B	109.8	C9—C8—H8	118.6
C2—C1—H1B	109.8	C10—C9—C13	117.36 (18)
H1A—C1—H1B	108.2	C10—C9—C8	120.58 (19)
C3—C2—C7	117.66 (16)	C13—C9—C8	122.06 (18)
C3—C2—C1	121.12 (14)	C9—C10—C11	119.6 (2)
C7—C2—C1	121.11 (14)	C9—C10—H10	120.2
C4—C3—C2	121.77 (17)	C11—C10—H10	120.2
C4—C3—H3	119.1	N2—C11—C10	123.6 (2)
C2—C3—H3	119.1	N2—C11—H11	118.2
C3—C4—C5	120.24 (17)	C10—C11—H11	118.2
C3—C4—H4	119.9	N2—C12—C13	124.3 (2)
C5—C4—H4	119.9	N2—C12—H12	117.9
C6—C5—C4	118.18 (16)	C13—C12—H12	117.9
C6—C5—N1	116.81 (15)	C12—C13—C9	118.7 (2)
C4—C5—N1	124.99 (16)	C12—C13—H13	120.7
C5—C6—C7	121.16 (16)	C9—C13—H13	120.7
C5—C6—H6	119.4	C8—N1—C5	120.59 (16)
C7—C6—H6	119.4	C11—N2—C12	116.53 (19)
C6—C7—C2	120.95 (16)	H1C—O1—H1D	104 (3)
C6—C7—H7	119.5	H2A—O2—H2B	113 (4)

C2^{i}—C1—C2—C3	79.47 (15)	N1—C8—C9—C13	5.0 (3)
C2^{i}—C1—C2—C7	−96.86 (15)	C13—C9—C10—C11	−0.3 (3)
C7—C2—C3—C4	1.5 (3)	C8—C9—C10—C11	179.97 (19)
C1—C2—C3—C4	−174.93 (16)	C9—C10—C11—N2	0.1 (4)
C2—C3—C4—C5	0.5 (3)	N2—C12—C13—C9	−0.7 (4)
C3—C4—C5—C6	−2.0 (3)	C10—C9—C13—C12	0.6 (3)
C3—C4—C5—N1	179.84 (16)	C8—C9—C13—C12	−179.7 (2)
C4—C5—C6—C7	1.5 (3)	C9—C8—N1—C5	178.70 (15)
N1—C5—C6—C7	179.84 (15)	C6—C5—N1—C8	169.49 (17)

C5—C6—C7—C2	0.5 (3)	C4—C5—N1—C8	−12.3 (3)
C3—C2—C7—C6	−2.0 (2)	C10—C11—N2—C12	−0.1 (4)
C1—C2—C7—C6	174.46 (16)	C13—C12—N2—C11	0.5 (4)
N1—C8—C9—C10	−175.31 (19)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O1	0.93	2.52	3.443 (3)	175
O1—H1*C*···O2	0.91 (4)	1.90 (4)	2.810 (3)	178 (4)
O2—H2*B*···N2^{ii}	0.87 (5)	1.94 (5)	2.782 (3)	163 (4)
O2—H2*A*···O1^{iii}	0.88 (5)	1.93 (5)	2.765 (3)	160 (4)
O1—H1*D*···O2^{iv}	0.91 (4)	1.88 (4)	2.799 (3)	178 (4)

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