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## Structure Reports

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

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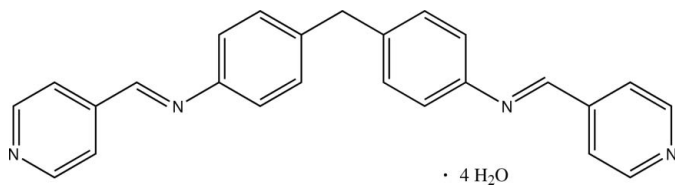
Received 21 November 2007; accepted 25 November 2007

Key indicators: single-crystal X-ray study;  $T = 294$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.058;  $wR$  factor = 0.158; data-to-parameter ratio = 14.2.

The title compound,  $\text{C}_{25}\text{H}_{20}\text{N}_4 \cdot 4\text{H}_2\text{O}$ , 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  $\text{C}-\text{H} \cdots \text{O}$ ,  $\text{O}-\text{H} \cdots \text{O}$  and  $\text{O}-\text{H} \cdots \text{N}$  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

$\text{C}_{25}\text{H}_{20}\text{N}_4 \cdot 4\text{H}_2\text{O}$   
 $M_r = 448.51$   
 Monoclinic,  $C2/c$   
 $a = 17.7743$  (14) Å  
 $b = 4.7309$  (4) Å

$c = 28.478$  (2) Å  
 $\beta = 99.69$  (1)°  
 $V = 2360.5$  (3) Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation

$\mu = 0.09$  mm<sup>-1</sup>  
 $T = 294$  (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  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.022$

### 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  
 $\Delta\rho_{\text{max}} = 0.20$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.18$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{C4}-\text{H4} \cdots \text{O1}$	0.93	2.52	3.443 (3)	175
$\text{O1}-\text{H1C} \cdots \text{O2}$	0.91 (4)	1.90 (4)	2.810 (3)	178 (4)
$\text{O2}-\text{H2B} \cdots \text{N2}^{\text{i}}$	0.87 (5)	1.94 (5)	2.782 (3)	163 (4)
$\text{O2}-\text{H2A} \cdots \text{O1}^{\text{ii}}$	0.88 (5)	1.93 (5)	2.765 (3)	160 (4)
$\text{O1}-\text{H1D} \cdots \text{O2}^{\text{iii}}$	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: *SAINTE* (Bruker, 2001); data reduction: *SAINTE*; 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*.

We are grateful to the Central China Normal University for financial support

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SF2014).

## References

- Bruker (2001). *SAINTE* (Version 6.45) and *SMART* (Version 5.628). Bruker AXS Inc., Madison, Wisconsin, USA.
- Coucouvanis, D., Jonasdottir, S. G., Jonasdottir, D., Kim, C. G. & Kampf, J. W. (1993). *Inorg. Chem.* **32**, 2987–2988.
- Hodnett, E. M. & Mooney, P. D. (1970). *J. Med. Chem.* **13**, 786.
- Jursic, B. S., Douelle, F., Bowdy, K. & Stevens, E. D. (2002). *Tetrahedron Lett.* **43**, 5361–5365.
- Li, Z.-K., Liang, L., Yang, L., Chen, H. & Zhou, X. G. (2005). *J. Mol. Catal. A Chem.* **235**, 108–113.
- Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
- Sheldrick, G. M. (2000). *SHELXTL*. Version 6.10. Bruker AXS Inc., Madison, Wisconsin, USA.
- Yam, V. W. W. & Pui, Y. L. (2002). *New J. Chem.* **26**, 536–542.

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**supplementary materials**

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

S.-L. Hu, Y.-T. Li and L.-P. Cao

### 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 (Coucouvani *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  $\Pi$  stacking. The water molecules create an infinite puckered ladder along *b* which links to the terminal N atoms of the organic amide.

### 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).

### 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  $d(\text{C—H})=0.97$  Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ ; aromatic H with  $d(\text{C—H})=0.93$  Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

### Figures

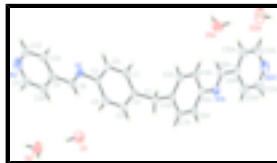


Fig. 1. The molecular structure showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as circles of arbitrary radii.

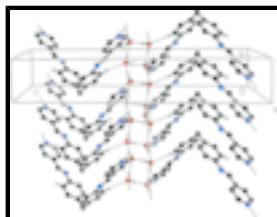


Fig. 2. The molecular packing of (I), with hydrogen bonds shown as dashed lines

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

### Crystal data

C<sub>25</sub>H<sub>20</sub>N<sub>4</sub>·4H<sub>2</sub>O<sub>1</sub>

*M<sub>r</sub>* = 448.51

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<sup>3</sup>

*Z* = 4

*F*<sub>000</sub> = 952

*D<sub>x</sub>* = 1.262 Mg m<sup>-3</sup>

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 2376 reflections

θ = 2.3–27.4°

μ = 0.09 mm<sup>-1</sup>

*T* = 294 (2) K

Block, yellow

0.40 × 0.30 × 0.30 mm

### Data collection

Bruker SMART CCD area-detector  
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

*T* = 294(2) K

φ and ω scans

Absorption correction: none

7552 measured reflections

2302 independent reflections

1850 reflections with *I* > 2σ(*I*)

*R*<sub>int</sub> = 0.022

θ<sub>max</sub> = 26.0°

θ<sub>min</sub> = 2.3°

*h* = -13→21

*k* = -5→5

*l* = -35→35

### Refinement

Refinement on *F*<sup>2</sup>

Least-squares matrix: full

*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.058

*wR* (*F*<sup>2</sup>) = 0.158

*S* = 1.08

2302 reflections

162 parameters

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<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3

(Δ/σ)<sub>max</sub> < 0.001

Δρ<sub>max</sub> = 0.20 e Å<sup>-3</sup>

Δρ<sub>min</sub> = -0.18 e Å<sup>-3</sup>

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  $F^2 > 2\sigma(F^2)$  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 ( $\text{\AA}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$	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 ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0515 (15)	0.0357 (13)	0.0585 (15)	0.000	0.0250 (12)	0.000

## supplementary materials

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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, °)

C1—C2 <sup>i</sup>	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 <sup>i</sup> —C1—C2	109.37 (19)	C2—C7—H7	119.5
C2 <sup>i</sup> —C1—H1A	109.8	N1—C8—C9	122.89 (18)
C2—C1—H1A	109.8	N1—C8—H8	118.6
C2 <sup>i</sup> —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 <sup>i</sup> —C1—C2—C3	79.47 (15)	N1—C8—C9—C13	5.0 (3)
C2 <sup>i</sup> —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 codes: (i)  $-x+1, y, -z+1/2$ .

*Hydrogen-bond geometry* ( $\text{\AA}, ^\circ$ )

<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>
C4—H4 $\cdots$ O1	0.93	2.52	3.443 (3)	175
O1—H1C $\cdots$ O2	0.91 (4)	1.90 (4)	2.810 (3)	178 (4)
O2—H2B $\cdots$ N2 <sup>ii</sup>	0.87 (5)	1.94 (5)	2.782 (3)	163 (4)
O2—H2A $\cdots$ O1 <sup>iii</sup>	0.88 (5)	1.93 (5)	2.765 (3)	160 (4)
O1—H1D $\cdots$ O2 <sup>iv</sup>	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)  $-x+1, -y, -z+1$ .

Fig. 1

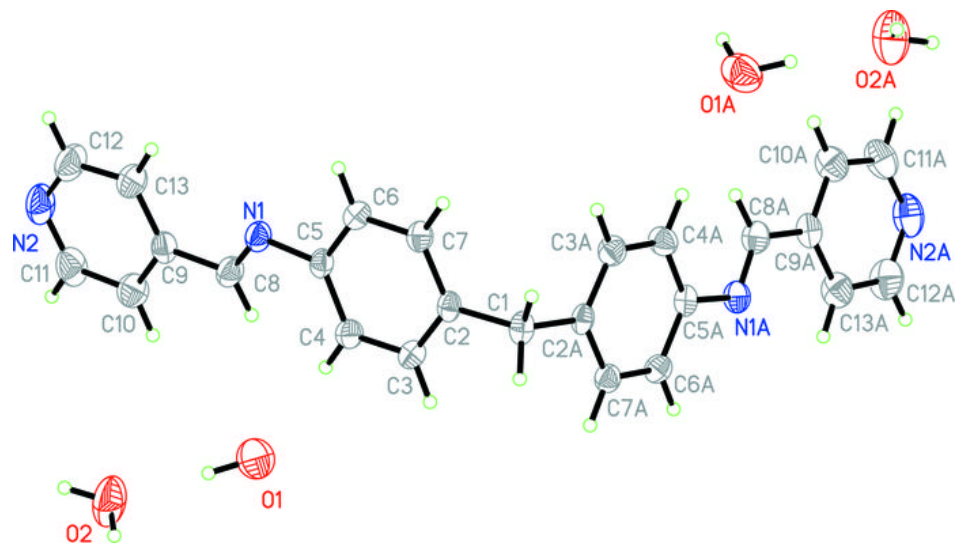




Fig. 2

