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6-Methoxy-2-(2-pyridylmethylimino-methyl)phenol

Zhong-Hai Ni* and Hai-Long Wang

School of Chemistry and Chemical Technology, Shandong University, Jinan 250100, People's Republic of China

Correspondence e-mail: nizhh@sdu.edu.cn

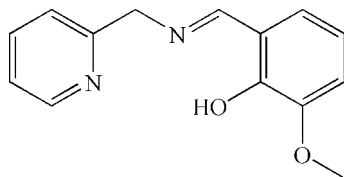
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Key indicators: single-crystal X-ray study; $T = 273$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.043; wR factor = 0.134; data-to-parameter ratio = 17.5.

A new tetradentate unsymmetrical Schiff base, $\text{C}_{14}\text{H}_{14}\text{N}_2\text{O}_2$, has been synthesized and structurally characterized. There exists a strong $\text{O} \cdots \text{H}-\text{N}$ intramolecular hydrogen bond in the structure.

Related literature

For related literature, see: Boskovic *et al.* (2003); Kannappan *et al.* (2005); Koizumi *et al.* (2005); Oshio *et al.* (2005); Zhang *et al.* (2003).



Experimental

Crystal data

 $\text{C}_{14}\text{H}_{14}\text{N}_2\text{O}_2$ $M_r = 242.27$ Monoclinic, $P2_1/c$ $a = 9.0899$ (2) Å $b = 5.66260$ (10) Å $c = 24.2440$ (5) Å $\beta = 92.626$ (2)° $V = 1246.59$ (4) Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 0.09$ mm⁻¹ $T = 273$ (2) K $0.2 \times 0.1 \times 0.08$ mm

Data collection

Bruker APEX II CCD area-detector diffractometer
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
 $T_{\min} = 0.989$, $T_{\max} = 0.993$

9173 measured reflections
2859 independent reflections
1920 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.043$ $wR(F^2) = 0.134$ $S = 0.99$

2859 reflections

163 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.13$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.20$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{O1}-\text{H1} \cdots \text{N2}$	0.82	1.84	2.571 (3)	148

Data collection: APEX2 (Bruker, 2004); cell refinement: SAINT-Plus (Bruker, 2001); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: XP (Sheldrick, 1998); software used to prepare material for publication: SHELXL97.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2277).

References

- Boskovic, C., Bircher, R., Tregenna-Piggott, P. L. W., Gudel, H. U., Paulsen, C., Wernsdorfer, W., Barra, A. L., Khatsko, E., Neels, A. & Stoeckli-Evans, H. (2003). *J. Am. Chem. Soc.* **125**, 14046–14058.
- Bruker (2001). *SAINTE-Plus*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (2004). *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Kannappan, R., Tanase, S., Mutikainen, I., Turpeinen, U. & Reedijk, J. (2005). *Inorg. Chim. Acta*, **358**, 383–388.
- Koizumi, S., Nihei, M., Nakano, M. & Oshio, H. (2005). *Inorg. Chem.* **44**, 1208–1210.
- Oshio, H., Nihei, M., Koizumi, S., Shiga, T., Nojiri, H., Nakano, M., Shirakawa, N. & Akatsu, M. (2005). *J. Am. Chem. Soc.* **127**, 4568–4569.
- Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
- Sheldrick, G. M. (1998). *XP*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (2003). *SADABS*. Version 2.10. University of Göttingen, Germany.
- Zhang, Y., Khoo, L. E. & Ng, S. W. (2003). *Acta Cryst.* **E59**, o1496–o1497.

supplementary materials

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6-Methoxy-2-(2-pyridylmethyliminomethyl)phenol

Z.-H. Ni and H.-L. Wang

Comment

Recently, Schiff base ligands, especially the relative flexible unsymmetrical tridentate Schiff base ligands and their hydrogenated derivatives have been employed to assembly alkoxo- or phenoxo-bridged clusters and polymers with beautiful molecular structures and interesting magnetic properties in the field of coordination chemistry. (Koizumi *et al.*, 2005; Boskovic *et al.*, 2003; Oshioh *et al.*, 2005).

Herein, we report the synthesis and crystal structure of a new unsymmetrical Schiff base 6-Methoxy-2-(2-pyridylmethyliminomethyl)phenol, (I), which possesses a O₂N₂ donor set affording a potentially tetradentate ligand.

The structure of (I) is shown in Fig. 1. The imide bond length of 1.278 (3) Å for N2—C7 is slightly longer than that of 4-Bromo-2-(2-pyridylmethyliminomethyl)phenol (1.269 (4) Å) (Zhang *et al.*, 2003). The C—N bond distances of 1.338 (2) Å for C1—N1 and 1.330 (2) Å for C5—N1 are slightly shorter than those of C—C bond lengths in the same pyridine ring. It is noteworthy that there exists a relative strong intramolecular bond in the title compound with hydrogen bond length 2.573 (3) Å for N2—O1 and bond angle 145.75 (3)° for N2—H1—O1, which is similar to those of its derivative 4-Bromo-2-(2-pyridylmethyliminomethyl)phenol (Zhang *et al.*, 2003).

The molecules in (I) are connected through relatively weak π - π interactions between the two adjacent pyridine rings and between benzene rings and imine groups yielding a one-dimensional supramolecular structure along the *b* axis (Fig. 2).

Experimental

6-Methoxy-2-(2-pyridylmethyliminomethyl)phenol was prepared according to the method reported in the literature (Kannappan *et al.*, 2005). 2-(2-aminomethyl)-pyridine (2.16 g, 0.02 mol) was added to a stirred ethanol solution of O-vanillin (3.04 g, 0.02 mol (10 ml)). The reaction mixture was stirred about 3 h and then the mixture was stand at room temperature for about two days. to yield yellow crystals suitable for X-ray diffraction analysis. Yield: 60%.

Refinement

H atoms bound to C and O atoms were visible in difference maps and were placed using the HFIX commands in *SHELXL97*. All H atoms were allowed for as riding atoms (C—H 0.97 Å, O—H 0.86 Å) with the constraint $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{methyl carrier})$, $1.5U_{\text{eq}}(\text{O})$ and $1.2U_{\text{eq}}(\text{carrier})$ for all other H atoms.

Figures

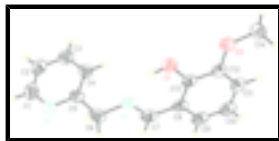


Fig. 1. A view of (I) with the unique atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

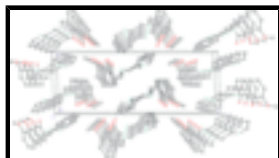


Fig. 2. The one-dimensional supramolecular structure in (I).

6-Methoxy-2-(2-pyridylmethyliminomethyl)phenol

Crystal data

$C_{14}H_{14}N_2O_2$

$M_r = 242.27$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 9.0899$ (2) Å

$b = 5.66260$ (10) Å

$c = 24.2440$ (5) Å

$\beta = 92.626$ (2)°

$V = 1246.59$ (4) Å³

$Z = 4$

$F_{000} = 512$

$D_x = 1.291$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 2859 reflections

$\theta = 2.2$ – 27.5 °

$\mu = 0.09$ mm⁻¹

$T = 273$ (2) K

Strip, yellow

$0.2 \times 0.1 \times 0.08$ mm

Data collection

Bruker APEX II CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 273$ (2) K

φ and ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 2003)

$T_{\min} = 0.989$, $T_{\max} = 0.993$

9173 measured reflections

2859 independent reflections

1920 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.020$

$\theta_{\text{max}} = 27.5$ °

$\theta_{\text{min}} = 2.2$ °

$h = -10 \rightarrow 11$

$k = -7 \rightarrow 7$

$l = -31 \rightarrow 31$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.043$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$wR(F^2) = 0.134$	$w = 1/[\sigma^2(F_o^2) + (0.0707P)^2 + 0.1506P]$
$S = 0.99$	where $P = (F_o^2 + 2F_c^2)/3$
2859 reflections	$(\Delta/\sigma)_{\max} < 0.001$
163 parameters	$\Delta\rho_{\max} = 0.13 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\min} = -0.19 \text{ e } \text{\AA}^{-3}$
	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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.17097 (10)	0.9235 (2)	0.15683 (4)	0.0665 (3)
H1	0.1843	0.8212	0.1337	0.100*
C13	0.29151 (14)	1.0610 (3)	0.16245 (5)	0.0549 (4)
O2	0.16576 (11)	1.2706 (2)	0.22814 (4)	0.0723 (3)
C8	0.41579 (15)	1.0222 (3)	0.13125 (6)	0.0602 (4)
N2	0.30337 (15)	0.7011 (3)	0.08109 (5)	0.0684 (4)
C12	0.29218 (14)	1.2481 (3)	0.20015 (6)	0.0589 (4)
C7	0.41632 (17)	0.8307 (3)	0.09163 (6)	0.0688 (4)
H7	0.5019	0.8009	0.0732	0.083*
C9	0.53800 (16)	1.1743 (4)	0.13884 (7)	0.0735 (5)
H9	0.6205	1.1511	0.1182	0.088*
C11	0.41353 (16)	1.3924 (3)	0.20683 (6)	0.0679 (4)
H11	0.4132	1.5160	0.2321	0.081*
C5	0.22332 (15)	0.5451 (3)	-0.01100 (6)	0.0586 (4)
N1	0.22922 (16)	0.3658 (2)	-0.04625 (6)	0.0763 (4)
C6	0.3129 (2)	0.5075 (3)	0.04198 (6)	0.0771 (5)
H6A	0.2799	0.3637	0.0593	0.093*
H6B	0.4152	0.4857	0.0334	0.093*
C14	0.1541 (2)	1.4681 (4)	0.26385 (7)	0.0784 (5)
H14A	0.0610	1.4632	0.2810	0.118*
H14B	0.1608	1.6112	0.2429	0.118*
H14C	0.2324	1.4634	0.2918	0.118*
C4	0.14319 (18)	0.7454 (3)	-0.02253 (7)	0.0697 (4)
H4	0.1425	0.8690	0.0027	0.084*
C2	0.06709 (19)	0.5785 (4)	-0.10834 (7)	0.0825 (5)

supplementary materials

H2	0.0134	0.5838	-0.1419	0.099*
C10	0.53675 (17)	1.3544 (4)	0.17593 (7)	0.0761 (5)
H10	0.6184	1.4525	0.1807	0.091*
C3	0.0637 (2)	0.7607 (4)	-0.07208 (8)	0.0817 (5)
H3	0.0081	0.8948	-0.0806	0.098*
C1	0.1515 (2)	0.3875 (4)	-0.09402 (8)	0.0862 (5)
H1A	0.1553	0.2641	-0.1192	0.103*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0514 (5)	0.0836 (7)	0.0651 (6)	-0.0019 (5)	0.0084 (4)	-0.0028 (5)
C13	0.0409 (6)	0.0741 (9)	0.0495 (7)	0.0037 (6)	0.0002 (5)	0.0160 (6)
O2	0.0535 (6)	0.0903 (8)	0.0740 (7)	-0.0043 (5)	0.0140 (5)	-0.0109 (6)
C8	0.0467 (7)	0.0854 (10)	0.0486 (7)	0.0129 (7)	0.0027 (5)	0.0182 (7)
N2	0.0651 (8)	0.0878 (9)	0.0523 (7)	0.0203 (7)	0.0023 (5)	0.0062 (6)
C12	0.0470 (7)	0.0755 (10)	0.0542 (7)	0.0035 (7)	0.0031 (6)	0.0125 (7)
C7	0.0535 (8)	0.1018 (12)	0.0516 (7)	0.0252 (9)	0.0070 (6)	0.0189 (8)
C9	0.0438 (7)	0.1100 (14)	0.0671 (9)	0.0060 (8)	0.0085 (6)	0.0268 (10)
C11	0.0566 (8)	0.0792 (11)	0.0676 (9)	-0.0033 (7)	-0.0010 (6)	0.0125 (8)
C5	0.0590 (8)	0.0606 (9)	0.0571 (8)	0.0078 (7)	0.0124 (6)	0.0051 (6)
N1	0.0852 (9)	0.0679 (9)	0.0763 (9)	0.0097 (7)	0.0103 (7)	-0.0070 (7)
C6	0.0834 (11)	0.0843 (11)	0.0636 (9)	0.0316 (9)	0.0042 (8)	0.0065 (8)
C14	0.0738 (10)	0.0926 (13)	0.0695 (10)	0.0045 (9)	0.0108 (8)	-0.0056 (9)
C4	0.0752 (10)	0.0659 (10)	0.0676 (9)	0.0148 (8)	-0.0018 (7)	-0.0001 (8)
C2	0.0691 (10)	0.1114 (15)	0.0660 (10)	-0.0136 (10)	-0.0057 (8)	0.0048 (10)
C10	0.0532 (8)	0.0937 (13)	0.0812 (11)	-0.0099 (8)	0.0015 (7)	0.0178 (10)
C3	0.0782 (11)	0.0851 (12)	0.0803 (11)	0.0114 (9)	-0.0125 (9)	0.0118 (10)
C1	0.0923 (13)	0.0908 (13)	0.0759 (11)	-0.0093 (11)	0.0079 (10)	-0.0207 (10)

Geometric parameters (\AA , $^\circ$)

O1—C13	1.3459 (17)	C5—C4	1.370 (2)
O1—H1	0.8200	C5—C6	1.504 (2)
C13—C12	1.399 (2)	N1—C1	1.334 (2)
C13—C8	1.4052 (19)	C6—H6A	0.9700
O2—C12	1.3669 (16)	C6—H6B	0.9700
O2—C14	1.421 (2)	C14—H14A	0.9600
C8—C9	1.411 (2)	C14—H14B	0.9600
C8—C7	1.449 (2)	C14—H14C	0.9600
N2—C7	1.278 (2)	C4—C3	1.376 (2)
N2—C6	1.454 (2)	C4—H4	0.9300
C12—C11	1.376 (2)	C2—C1	1.362 (3)
C7—H7	0.9300	C2—C3	1.357 (3)
C9—C10	1.360 (3)	C2—H2	0.9300
C9—H9	0.9300	C10—H10	0.9300
C11—C10	1.392 (2)	C3—H3	0.9300
C11—H11	0.9300	C1—H1A	0.9300
C5—N1	1.3300 (19)		

C13—O1—H1	109.5	N2—C6—H6A	108.8
O1—C13—C12	118.90 (11)	C5—C6—H6A	108.8
O1—C13—C8	121.74 (14)	N2—C6—H6B	108.8
C12—C13—C8	119.36 (13)	C5—C6—H6B	108.8
C12—O2—C14	117.61 (13)	H6A—C6—H6B	107.7
C13—C8—C9	118.82 (15)	O2—C14—H14A	109.5
C13—C8—C7	120.05 (14)	O2—C14—H14B	109.5
C9—C8—C7	121.13 (14)	H14A—C14—H14B	109.5
C7—N2—C6	119.44 (14)	O2—C14—H14C	109.5
O2—C12—C11	125.02 (14)	H14A—C14—H14C	109.5
O2—C12—C13	114.59 (12)	H14B—C14—H14C	109.5
C11—C12—C13	120.39 (13)	C5—C4—C3	118.90 (16)
N2—C7—C8	122.36 (13)	C5—C4—H4	120.6
N2—C7—H7	118.8	C3—C4—H4	120.6
C8—C7—H7	118.8	C1—C2—C3	117.91 (17)
C10—C9—C8	120.90 (14)	C1—C2—H2	121.0
C10—C9—H9	119.5	C3—C2—H2	121.0
C8—C9—H9	119.5	C9—C10—C11	120.19 (16)
C12—C11—C10	120.33 (16)	C9—C10—H10	119.9
C12—C11—H11	119.8	C11—C10—H10	119.9
C10—C11—H11	119.8	C2—C3—C4	119.42 (17)
N1—C5—C4	122.53 (14)	C2—C3—H3	120.3
N1—C5—C6	113.86 (13)	C4—C3—H3	120.3
C4—C5—C6	123.60 (14)	N1—C1—C2	124.35 (17)
C5—N1—C1	116.88 (15)	N1—C1—H1A	117.8
N2—C6—C5	113.82 (13)	C2—C1—H1A	117.8

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O1—H1...N2	0.82	?	2.573 (3)	146

Fig. 1

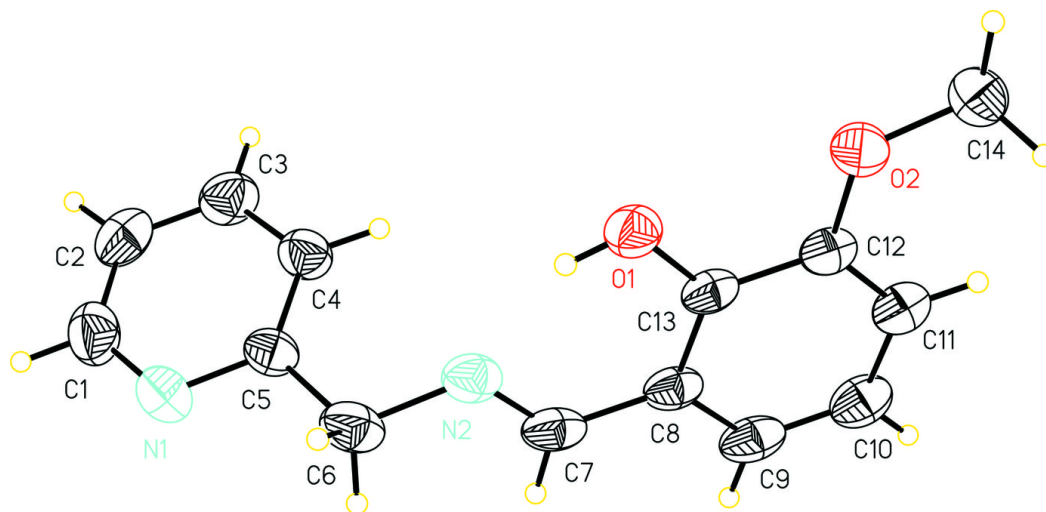


Fig. 2

