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Crystal structure of (*E*)-4-methoxy-2-{[(5-methylpyridin-2-yl)imino]methyl}phenol

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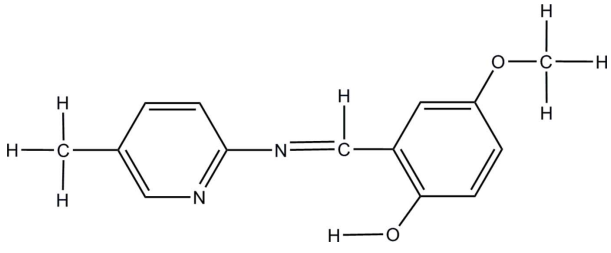
The molecule of the title Schiff base compound, $C_{14}H_{14}N_2O_2$, displays an *E* conformation with respect the imine C=N double bond. The molecule is approximately planar, with the dihedral angle formed by the planes of the pyridine and benzene rings being 5.72 (6)°. There is an intramolecular hydrogen bond involving the phenolic H and imine N atoms.

Keywords: crystal structure; Schiff base; *N*-heterocycle; intramolecular hydrogen bond.

CCDC reference: 1048553

1. Related literature

For the structure of related *N*-heterocyclic Schiff base compounds, see: Sahebalzamani *et al.* (2011); Rawat & Singh (2015); Thakar *et al.* (2015); Salam *et al.* (2011).



2. Experimental

2.1. Crystal data

$C_{14}H_{14}N_2O_2$ M_r = 242.27

Monoclinic $P2_1/n$
a = 12.7082 (12) Å
b = 4.7446 (4) Å
c = 21.124 (2) Å
β = 105.525 (2)°
V = 1227.21 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 294 K
0.41 × 0.35 × 0.12 mm

2.2. Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (SABABS; Bruker, 2014)
T_{min} = 0.030, T_{max} = 0.262
10950 measured reflections
2848 independent reflections
1910 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.151
S = 1.07
2848 reflections
170 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.17 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*
O2—H1*O*2···N2	0.95 (2)	1.76 (3)	2.6276 (19)	150 (2)

Data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINT* (Bruker, 2014); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2015); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: RZ5166).

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supporting information

Acta Cryst. (2015). E**71**, o819 [https://doi.org/10.1107/S2056989015018113]

Crystal structure of (*E*)-4-methoxy-2-{[(5-methylpyridin-2-yl)imino]methyl}-phenol

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S1. Comment

The molecule of the title compound (Fig. 1) has an *E* configuration about the imine C═N double bond, as indicated by the value of 179.11(12° of the C5-N2-C6-C7 torsion angle. The molecule is is almost planar, with a dihedral angle between the pyridine and benzene rings of 5.72 (6)°. The molecular conformation is stabilized by an intramolecular O—H···N hydrogen bond (Table 1) occurring between the phenolic hydrogen and imine nitrogen atoms. In the crystal, packing is stabilized only by van der Waals interactions.

S2. Experimental

2-Hydroxy-5-methoxybenzaldehyde (5 mmol, 0.761 g) and 2-amino-5-methylpyridin (5 mmol, 0.541 g) were dissolved in ethanol in separate beakers, then the amine solution was added drop wise with stirring to the aldehyde in the round bottomed flask. Acetic acid was added and the solution was refluxed for 4 h under Ar atmosphere (Fig. 3). The solid product obtained was dried under reduced pressure overnight, then recrystallized with dichloromethane, diethyl ether and excess n-hexane, filtered, washed again with diethyl ether/n-hexane (1:3 *v*/*v*) and dried out over 24 h under reduced pressure in a desiccator. Purple single crystals of the title compound were grown on evaporation of an ethanol solution. M. p.: 380-381 K. Yield: 85%. Anal. calc. for $C_{14}H_{14}N_2O_2$ (FW: 242.28 g/mol): C, 69.43; H, 5.77; N, 11.55%. Found: C, 69.93; H, 5.69; N, 11.54%. IR (KBr pellets μ_{max}/cm^{-1}): 3427 *ν*(N—H), 2952 and 1384 *ν*(CH_3), 1618 *ν*(C═N), 1497 *ν*(C═C, ar.), 1210 *ν*(C—O), 1035 *ν*(C—N).

S3. Refinement

The phenolic hydrogen atom was located in a difference Fourier map and refined freely. All other H atoms were calculated geometrically and refined using a riding model, with C—H = 0.93 Å, and with U_{iso}(H) = 1.2 U_{eq}(C) or 1.5 U_{eq}(C) for methyl H atoms. A rotating model was used for all methyl group. The H atoms of the methyl carbon attached to the pyridine ring are disordered over two sets of sites with an occupancy ratio of 0.61 (2):0.39 (2).

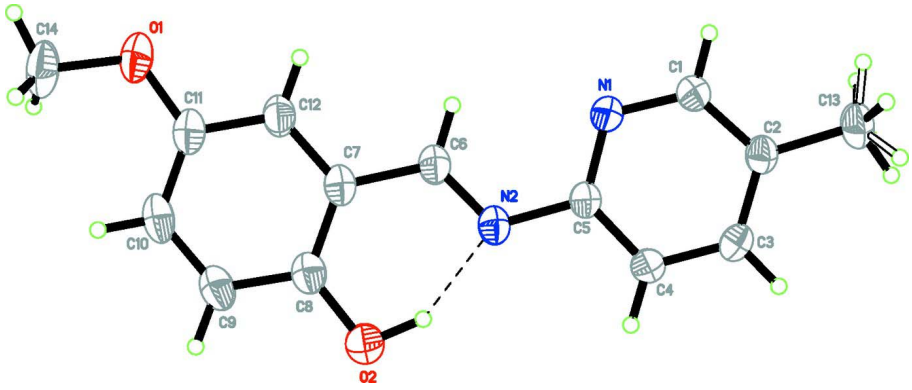

Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level. The dashed line indicates an intramolecular hydrogen bond.

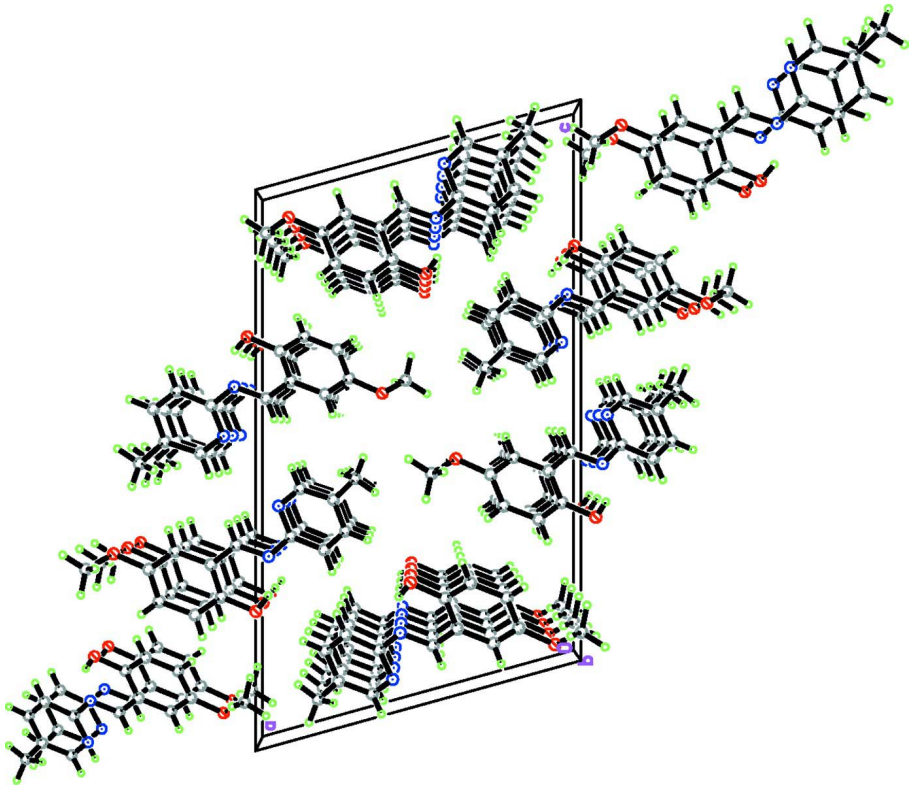

Figure 2

Packing of the title compound compound viewed down the *b* axis.

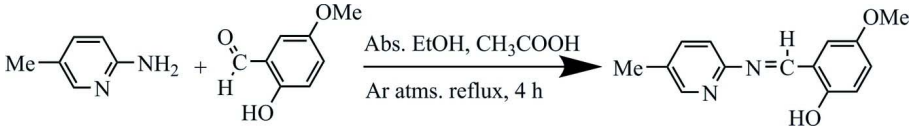

Figure 3

Synthesis of the title compound.

(I)

Crystal data

$C_{14}H_{14}N_2O_2$
$M_r = 242.27$
Monoclinic, $P2_1/n$
$a = 12.7082$ (12) Å
$b = 4.7446$ (4) Å
$c = 21.124$ (2) Å
$\beta = 105.525$ (2)°
$V = 1227.21$ (19) Å^3
$Z = 4$
$F(000) = 512$
$D_x = 1.311$ Mg m^{-3}
Melting point: 380 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$\theta = 1.7$–27.6°
$\mu = 0.09$ mm^{-1}
$T = 294$ K
Block, purple
0.41 × 0.35 × 0.12 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SABABS; Bruker, 2014)
$T_{min} = 0.030$, $T_{max} = 0.262$
10950 measured reflections
2848 independent reflections
1910 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 27.6°$, $\theta_{min} = 1.7°$
$h = -16\rightarrow14$
$k = -6\rightarrow6$
$l = -27\rightarrow27$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.151$
$S = 1.07$
2848 reflections
170 parameters
0 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0755P)^2 + 0.121P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.17$ 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.11027 (10)	0.5504 (3)	0.07471 (7)	0.0866 (5)	
O2	0.52254 (10)	0.4026 (3)	0.24106 (6)	0.0762 (4)	
N1	0.57565 (9)	−0.2914 (3)	0.07413 (6)	0.0527 (3)	
N2	0.55236 (9)	0.0280 (3)	0.15589 (6)	0.0495 (3)	
C1	0.63754 (12)	−0.4827 (3)	0.05470 (8)	0.0543 (4)	
H1A	0.6101	−0.5632	0.0134	0.065*	
C2	0.73934 (11)	−0.5698 (3)	0.09138 (7)	0.0476 (4)	
C3	0.77704 (12)	−0.4503 (4)	0.15259 (8)	0.0558 (4)	
H3A	0.8446	−0.5033	0.1797	0.067*	
C4	0.71504 (12)	−0.2527 (4)	0.17389 (7)	0.0548 (4)	
H4A	0.7403	−0.1707	0.2152	0.066*	

C5	0.61512 (10)	−0.1780 (3)	0.13323 (7)	0.0445 (3)	
C6	0.45685 (12)	0.0882 (3)	0.12034 (8)	0.0521 (4)	
H6A	0.4309	−0.0023	0.0801	0.063*	
C7	0.38731 (11)	0.2931 (3)	0.14055 (7)	0.0487 (4)	
C8	0.42234 (12)	0.4410 (3)	0.19963 (7)	0.0536 (4)	
C9	0.35180 (14)	0.6348 (4)	0.21589 (8)	0.0630 (5)	
H9A	0.3750	0.7380	0.2545	0.076*	
C10	0.24841 (14)	0.6764 (3)	0.17591 (9)	0.0621 (4)	
H10A	0.2021	0.8053	0.1880	0.074*	
C11	0.21273 (13)	0.5282 (4)	0.11786 (9)	0.0593 (4)	
C12	0.28284 (12)	0.3399 (4)	0.10047 (8)	0.0583 (4)	
H12A	0.2596	0.2422	0.0610	0.070*	
C13	0.80422 (14)	−0.7829 (4)	0.06515 (9)	0.0651 (5)	
H13A	0.8653	−0.8443	0.0999	0.098*	0.61 (2)
H13B	0.8302	−0.6989	0.0308	0.098*	0.61 (2)
H13C	0.7587	−0.9416	0.0479	0.098*	0.61 (2)
H13D	0.8763	−0.7110	0.0692	0.098*	0.39 (2)
H13E	0.7689	−0.8191	0.0197	0.098*	0.39 (2)
H13F	0.8090	−0.9547	0.0897	0.098*	0.39 (2)
C14	0.03899 (15)	0.7589 (5)	0.08715 (13)	0.0898 (7)	
H14A	−0.0274	0.7588	0.0523	0.135*	
H14B	0.0732	0.9404	0.0895	0.135*	
H14C	0.0227	0.7192	0.1281	0.135*	
H1O2	0.5562 (19)	0.261 (5)	0.2213 (13)	0.120 (9)*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0597 (7)	0.0913 (10)	0.1072 (10)	0.0322 (7)	0.0198 (7)	−0.0079 (8)
O2	0.0739 (8)	0.0858 (10)	0.0632 (7)	0.0210 (7)	0.0084 (6)	−0.0106 (7)
N1	0.0450 (6)	0.0544 (7)	0.0554 (7)	0.0084 (6)	0.0076 (5)	−0.0070 (6)
N2	0.0510 (7)	0.0454 (7)	0.0547 (7)	0.0090 (5)	0.0186 (5)	0.0028 (6)
C1	0.0491 (8)	0.0565 (9)	0.0558 (8)	0.0042 (7)	0.0113 (6)	−0.0122 (7)
C2	0.0453 (7)	0.0406 (7)	0.0595 (8)	0.0022 (6)	0.0182 (6)	0.0003 (7)
C3	0.0449 (7)	0.0614 (10)	0.0567 (8)	0.0126 (7)	0.0057 (6)	0.0021 (8)
C4	0.0527 (8)	0.0620 (10)	0.0461 (7)	0.0114 (7)	0.0067 (6)	−0.0049 (7)
C5	0.0448 (7)	0.0409 (7)	0.0494 (7)	0.0047 (6)	0.0151 (6)	0.0012 (6)
C6	0.0520 (8)	0.0489 (8)	0.0578 (8)	0.0094 (7)	0.0187 (7)	0.0000 (7)
C7	0.0524 (8)	0.0419 (7)	0.0576 (8)	0.0073 (6)	0.0247 (7)	0.0058 (7)
C8	0.0619 (9)	0.0489 (8)	0.0553 (8)	0.0079 (7)	0.0248 (7)	0.0076 (7)
C9	0.0803 (11)	0.0557 (9)	0.0618 (9)	0.0118 (9)	0.0342 (9)	−0.0001 (8)
C10	0.0730 (10)	0.0500 (9)	0.0774 (11)	0.0155 (8)	0.0448 (9)	0.0074 (8)
C11	0.0554 (9)	0.0545 (9)	0.0749 (10)	0.0134 (7)	0.0293 (8)	0.0078 (8)
C12	0.0557 (8)	0.0558 (9)	0.0663 (9)	0.0131 (8)	0.0213 (7)	−0.0019 (8)
C13	0.0633 (9)	0.0536 (9)	0.0842 (11)	0.0102 (8)	0.0297 (9)	−0.0057 (9)
C14	0.0614 (10)	0.0771 (13)	0.1362 (19)	0.0267 (10)	0.0357 (11)	0.0035 (13)

Geometric parameters (Å, º)

O1—C11	1.379 (2)	C7—C12	1.388 (2)
O1—C14	1.413 (2)	C7—C8	1.397 (2)
O2—C8	1.3505 (19)	C8—C9	1.390 (2)
O2—H1O2	0.95 (3)	C9—C10	1.373 (2)
N1—C5	1.3282 (18)	C9—H9A	0.9300
N1—C1	1.3356 (18)	C10—C11	1.381 (2)
N2—C6	1.2771 (18)	C10—H10A	0.9300
N2—C5	1.4231 (17)	C11—C12	1.379 (2)
C1—C2	1.381 (2)	C12—H12A	0.9300
C1—H1A	0.9300	C13—H13A	0.9600
C2—C3	1.375 (2)	C13—H13B	0.9600
C2—C13	1.501 (2)	C13—H13C	0.9600
C3—C4	1.376 (2)	C13—H13D	0.9600
C3—H3A	0.9300	C13—H13E	0.9600
C4—C5	1.3754 (19)	C13—H13F	0.9600
C4—H4A	0.9300	C14—H14A	0.9600
C6—C7	1.4526 (19)	C14—H14B	0.9600
C6—H6A	0.9300	C14—H14C	0.9600
C11—O1—C14	118.00 (16)	C10—C9—H9A	119.4
C8—O2—H1O2	105.5 (15)	C8—C9—H9A	119.4
C5—N1—C1	117.31 (12)	C9—C10—C11	120.38 (14)
C6—N2—C5	119.06 (13)	C9—C10—H10A	119.8
N1—C1—C2	124.82 (14)	C11—C10—H10A	119.8
N1—C1—H1A	117.6	C12—C11—O1	115.88 (16)
C2—C1—H1A	117.6	C12—C11—C10	118.98 (16)
C3—C2—C1	116.25 (13)	O1—C11—C10	125.14 (14)
C3—C2—C13	122.49 (14)	C11—C12—C7	121.46 (16)
C1—C2—C13	121.26 (14)	C11—C12—H12A	119.3
C2—C3—C4	120.18 (13)	C7—C12—H12A	119.3
C2—C3—H3A	119.9	C2—C13—H13A	109.5
C4—C3—H3A	119.9	C2—C13—H13B	109.5
C5—C4—C3	119.02 (14)	H13A—C13—H13B	109.5
C5—C4—H4A	120.5	C2—C13—H13C	109.5
C3—C4—H4A	120.5	H13A—C13—H13C	109.5
N1—C5—C4	122.41 (13)	H13B—C13—H13C	109.5
N1—C5—N2	119.32 (12)	C2—C13—H13D	109.5
C4—C5—N2	118.27 (13)	C2—C13—H13E	109.5
N2—C6—C7	122.28 (14)	H13D—C13—H13E	109.5
N2—C6—H6A	118.9	C2—C13—H13F	109.5
C7—C6—H6A	118.9	H13D—C13—H13F	109.5
C12—C7—C8	119.25 (13)	H13E—C13—H13F	109.5
C12—C7—C6	119.06 (14)	O1—C14—H14A	109.5
C8—C7—C6	121.69 (14)	O1—C14—H14B	109.5
O2—C8—C9	119.18 (15)	H14A—C14—H14B	109.5
O2—C8—C7	122.08 (13)	O1—C14—H14C	109.5

C9—C8—C7	118.74 (15)	H14A—C14—H14C	109.5
C10—C9—C8	121.17 (16)	H14B—C14—H14C	109.5
C5—N1—C1—C2	−0.3 (2)	C12—C7—C8—O2	179.28 (14)
N1—C1—C2—C3	1.0 (2)	C6—C7—C8—O2	0.2 (2)
N1—C1—C2—C13	−178.93 (14)	C12—C7—C8—C9	−1.1 (2)
C1—C2—C3—C4	−0.9 (2)	C6—C7—C8—C9	179.78 (13)
C13—C2—C3—C4	178.97 (14)	O2—C8—C9—C10	−178.66 (14)
C2—C3—C4—C5	0.3 (2)	C7—C8—C9—C10	1.7 (2)
C1—N1—C5—C4	−0.5 (2)	C8—C9—C10—C11	−0.9 (2)
C1—N1—C5—N2	179.64 (12)	C14—O1—C11—C12	−174.24 (16)
C3—C4—C5—N1	0.5 (2)	C14—O1—C11—C10	6.3 (3)
C3—C4—C5—N2	−179.64 (13)	C9—C10—C11—C12	−0.6 (2)
C6—N2—C5—N1	3.8 (2)	C9—C10—C11—O1	178.92 (15)
C6—N2—C5—C4	−176.11 (13)	O1—C11—C12—C7	−178.37 (14)
C5—N2—C6—C7	179.11 (12)	C10—C11—C12—C7	1.2 (3)
N2—C6—C7—C12	−177.58 (14)	C8—C7—C12—C11	−0.3 (2)
N2—C6—C7—C8	1.5 (2)	C6—C7—C12—C11	178.81 (14)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H1*O2*···N2	0.95 (2)	1.76 (3)	2.6276 (19)	150 (2)