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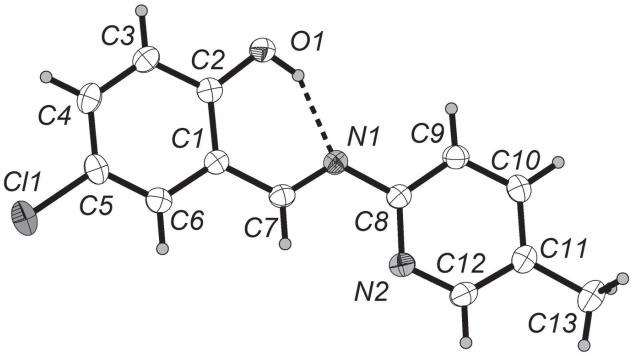
The crystal structure of (*E*)-4-chloro-2-(((5-methylpyridin-2-yl)imino)methyl)phenol, $C_{13}H_{11}ClN_2O$



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Abstract

$C_{13}H_{11}ClN_2O$, monoclinic, $P2_1/n$ (no. 14), $a = 13.997(5)$ Å, $b = 4.6734(15)$ Å, $c = 18.019(7)$ Å, $\beta = 102.714(5)°$, $V = 1149.7(7)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0481$, $wR_{ref}(F^2) = 0.1141$, $T = 153(2)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The ethanol solution (10 mL) of 5-chlorosalicylaldehyde (0.2 mmol, 31.32 mg) was added dropwise to the ethanol solution (10 mL) of 2-amino-5-methylpyridine (0.2 mmol, 21.63 mg,) over a period of 30 min with stirring. The stirring was continued for 1 h to give a clear orange solution, allowed to evaporate slowly in air at room temperature. After 7 days, orange prism-shaped crystals of the title compound were formed at the bottom of the vessel. The crystals were isolated, washed with ethanol and dried at room temperature (yield 54%).

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Table 1: Data collection and handling.

Crystal:	Prism, orange
Size:	0.58 × 0.13 × 0.13 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.32 mm^{-1}
Diffractometer, scan mode:	AFC10/Saturn724+, φ and ω-scans
θ_{max}, Completeness:	28°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	8971, 2775, 0.027
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2484
$N(param)_{refined}$:	159
Programs:	Rigaku programs [1], SHELX [2]

Experimental details

All hydrogen atoms were identified in difference Fourier syntheses and placed in geometrically idealized positions. The U_{iso} values of the hydrogen atoms of methyl groups were set to $1.5U_{eq}$(C) and the U_{iso} values of all other hydrogen atoms were set to $1.2U_{eq}$(C).

Discussion

The synthesis of Schiff bases and its metal complexes have drawn much attention due to their extensive applications. It was demonstrated that the complexes play an important role as antimicrobial [3], antioxidative [4], antibiotic [5] and anticancer [6] reagents. All the properties have a particularly close connection to its structural features. Previously, we have synthesized a series of Schiff bases and its metal complexes in order to explore its extensive applications [7, 8]. To study the structure-activity relationship we have undertaken the synthesis and single crystal structure determination of the title compound (*cf*. the figure).

The title crystal structure is built up by only one crystallographically independent Schiff base molecule. There is a *E* configuration at the C=N bond. All the bond lengths and bond angles within the Schiff base agree with the values reported [9]. The C(8)—N(1), C(7)—N(1), C(8)—N(2) and C(12)—N(2) bonds are significantly shorter than a normal single C—N bond (1.47 Å) [10] and longer than a C=N bond (1.28 Å) [11], which may be caused by the significant

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.88851(3)	−0.25239(11)	0.60279(3)	0.03357(15)
O1	0.77266(9)	0.3044(3)	0.30495(7)	0.0254(3)
N1	0.64689(10)	0.6072(3)	0.35980(8)	0.0204(3)
N2	0.53197(10)	0.8812(3)	0.40995(8)	0.0239(3)
C1	0.74993(12)	0.2552(3)	0.43334(9)	0.0199(3)
C2	0.79527(12)	0.1782(4)	0.37350(10)	0.0209(3)
C3	0.86697(12)	−0.0349(4)	0.38516(10)	0.0243(4)
H3	0.8965	−0.0896	0.3446	0.029*
C4	0.89540(13)	−0.1669(4)	0.45517(11)	0.0259(4)
H4	0.9446	−0.3105	0.4630	0.031*
C5	0.85125(12)	−0.0875(4)	0.51402(10)	0.0242(4)
C6	0.77900(12)	0.1176(4)	0.50359(10)	0.0226(4)
H6	0.7488	0.1658	0.5442	0.027*
C7	0.67370(12)	0.4718(4)	0.42309(9)	0.0209(3)
H7	0.6431	0.5142	0.4639	0.025*
C8	0.57167(12)	0.8149(4)	0.35096(9)	0.0197(3)
C9	0.54224(12)	0.9427(4)	0.28009(10)	0.0235(4)
H9	0.5731	0.8946	0.2398	0.028*
C10	0.46717(12)	1.1415(4)	0.26925(10)	0.0242(4)
H10	0.4457	1.2302	0.2210	0.029*
C11	0.42317(12)	1.2114(4)	0.32894(10)	0.0228(4)
C12	0.46019(12)	1.0762(4)	0.39815(10)	0.0252(4)
H12	0.4325	1.1259	0.4400	0.030*
C13	0.34098(13)	1.4245(4)	0.31998(11)	0.0312(4)
H13A	0.3630	1.6093	0.3042	0.037*
H13B	0.3213	1.4469	0.3686	0.037*
H13C	0.2851	1.3562	0.2813	0.037*
H1O	0.7287(18)	0.434(6)	0.3055(14)	0.053(7)*

electron delocalization in the pyridine system. The pyridinyl moiety is planar (maximum deviation = 0.0065 Å) and the chloro phenyl adopts a planar formation (maximum deviation = 0.0058 Å). The two rings are nearly coplanar with a dihedral angle of 2.49°, which is interpreted as conjugation effect and stablization of the ring by an intramolecular OH···N hydrogen bond, well known for such molecules [9]. The adjacent molecular unit are parallel to each other in one direction. one set of parallelmolecular connected with the other ones. CH···O play important roles in the formation, stability and crystallization of the title compound.

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