Crystal structure of 2,4-dichloro-6-[(6-methylpyridin-2-ylimino)methyl]phenol, $C_{13}H_{10}Cl_2N_2O$

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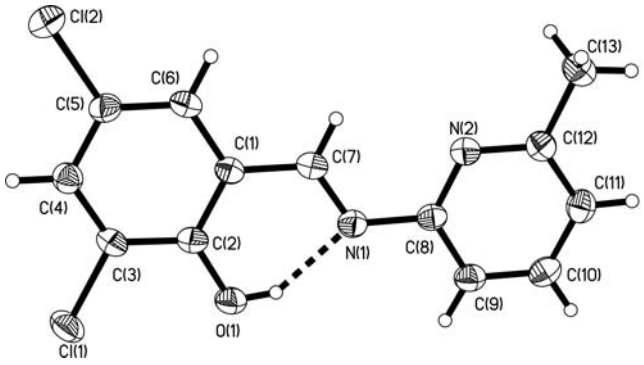

Abstract

$C_{13}H_{10}Cl_2N_2O$, monoclinic, *P*2/*c* (no. 13), $a = 14.603(3)$ Å, $b = 4.323(2)$ Å, $c = 20.445(3)$ Å, $\beta = 102.234(2)°$, $V = 1261.4$ Å^3, $Z = 4$, $R_{gf}(F) = 0.042$, $wR_{ref}(F^2) = 0.101$, $T = 298$ K.

Source of material

Reagents and solvents used were of commercially available quality. An ethanol solution (10 ml) of 2-amino-6-methylpyridine (0.1 mmol, 10.8 mg) was added to a stirred ethanol solution (10 ml) of 3,5-dichlorosalicylaldehyde (0.1 mmol, 19.1 mg). After stirring for about 30 min at room temperature, the clear yellow solution was left to stand still in air. Yellow block-shaped crystals of the compound were formed after slow evaporation of the solvent for a few days.

Experimental details

H atoms were placed in calculated positions and constrained to ride on their parent atoms with d(C—H) = 0.93 - 0.96 Å, d(O—H) = 0.82 Å, and U_{iso}(H) = 1.2 U_{eq}(C) and 1.5 U_{eq}(O1, C13).

Discussion

Schiff bases play an important role in biological [1-3] and coordination chemistry [4-6]. Recently, we have reported the crystal structures of a few Schiff bases and their complexes with metal atoms [7,8].

The molecule of the title compound is approximately planar, with a mean deviation from the least squares plane through all 18 non-hydrogen atoms of 0.058(2) Å. The dihedral angle between the C1-C6 benzene ring and the C8-C12/N2 pyridine ring is 5.3(2)°. There is an intramolecular O1–H1···N1 hydrogen bond, which supports planarity of the molecule. The bond lengths and angles are comparable to those found in the similar Schiff base compounds [9-11].

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.18 × 0.20 × 0.20 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	5.02 cm^{-1}
Diffractometer, scan mode:	Siemens P4, ω
$2\theta_{max}$:	54°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	9591, 2750
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1754
$N(param)_{refined}$:	165
Programs:	SHELXS-97, SHELXL-97 [12]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	4*g*	0.1119	0.6082	0.4668	0.085
H(4)	4*g*	0.2813	–0.0308	0.3255	0.058
H(6)	4*g*	0.4136	0.4232	0.4894	0.057
H(7)	4*g*	0.3277	0.7422	0.5538	0.052
H(9)	4*g*	0.0553	1.0466	0.5754	0.061
H(10)	4*g*	0.0573	1.3875	0.6637	0.067
H(11)	4*g*	0.2002	1.5446	0.7273	0.065
H(13A)	4*g*	0.4128	1.2250	0.7291	0.099
H(13B)	4*g*	0.3679	1.5239	0.7523	0.099
H(13C)	4*g*	0.4050	1.5270	0.6858	0.099

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cl(1)	4*g*	0.09187(4)	0.0994(2)	0.31694(3)	0.0483(4)	0.0947(5)	0.0521(3)	–0.0126(3)	–0.0016(3)	–0.0131(3)
Cl(2)	4*g*	0.46825(4)	0.0446(2)	0.39328(3)	0.0464(4)	0.0890(5)	0.0809(5)	0.0071(3)	0.0179(3)	–0.0143(4)
N(1)	4*g*	0.1946(1)	0.8035(4)	0.53578(8)	0.043(1)	0.045(1)	0.045(1)	0.0019(8)	0.0068(8)	0.0029(8)
N(2)	4*g*	0.2799(1)	1.1055(4)	0.62510(8)	0.042(1)	0.048(1)	0.045(1)	0.0016(9)	0.0076(8)	0.0025(9)
O(1)	4*g*	0.1047(1)	0.4870(4)	0.43531(7)	0.0367(8)	0.076(1)	0.055(1)	0.0017(8)	0.0046(7)	–0.0079(8)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	4*g*	0.2725(1)	0.4808(5)	0.4703(1)	0.038(1)	0.040(1)	0.040(1)	–0.004(1)	0.0052(8)	0.0042(9)
C(2)	4*g*	0.1881(1)	0.3935(5)	0.4264(1)	0.036(1)	0.048(1)	0.043(1)	–0.002(1)	0.0060(9)	0.007(1)
C(3)	4*g*	0.1942(2)	0.2024(5)	0.3722(1)	0.041(1)	0.052(1)	0.039(1)	–0.009(1)	0.0019(9)	0.003(1)
C(4)	4*g*	0.2790(2)	0.0959(5)	0.3619(1)	0.049(1)	0.053(2)	0.044(1)	–0.001(1)	0.010(1)	0.001(1)
C(5)	4*g*	0.3609(1)	0.1803(5)	0.4065(1)	0.038(1)	0.054(2)	0.053(1)	–0.001(1)	0.013(1)	0.002(1)
C(6)	4*g*	0.3582(2)	0.3692(5)	0.4599(1)	0.037(1)	0.052(2)	0.049(1)	–0.004(1)	0.0006(9)	0.000(1)
C(7)	4*g*	0.2714(2)	0.6893(5)	0.5253(1)	0.038(1)	0.047(1)	0.043(1)	–0.003(1)	0.0053(9)	0.007(1)
C(8)	4*g*	0.1966(1)	1.0139(5)	0.5895(1)	0.039(1)	0.042(1)	0.046(1)	0.002(1)	0.0082(9)	0.006(1)
C(9)	4*g*	0.1117(2)	1.1140(5)	0.6018(1)	0.041(1)	0.053(2)	0.058(1)	0.000(1)	0.008(1)	0.002(1)
C(10)	4*g*	0.1130(2)	1.3158(6)	0.6540(1)	0.047(1)	0.059(2)	0.063(2)	0.008(1)	0.019(1)	0.003(1)
C(11)	4*g*	0.1980(2)	1.4098(5)	0.6916(1)	0.062(2)	0.054(2)	0.050(1)	0.002(1)	0.018(1)	–0.003(1)
C(12)	4*g*	0.2805(2)	1.3029(5)	0.6761(1)	0.050(1)	0.047(1)	0.042(1)	–0.001(1)	0.009(1)	0.004(1)
C(13)	4*g*	0.3751(2)	1.4038(6)	0.7143(1)	0.056(2)	0.078(2)	0.060(2)	–0.002(1)	0.003(1)	–0.014(1)

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