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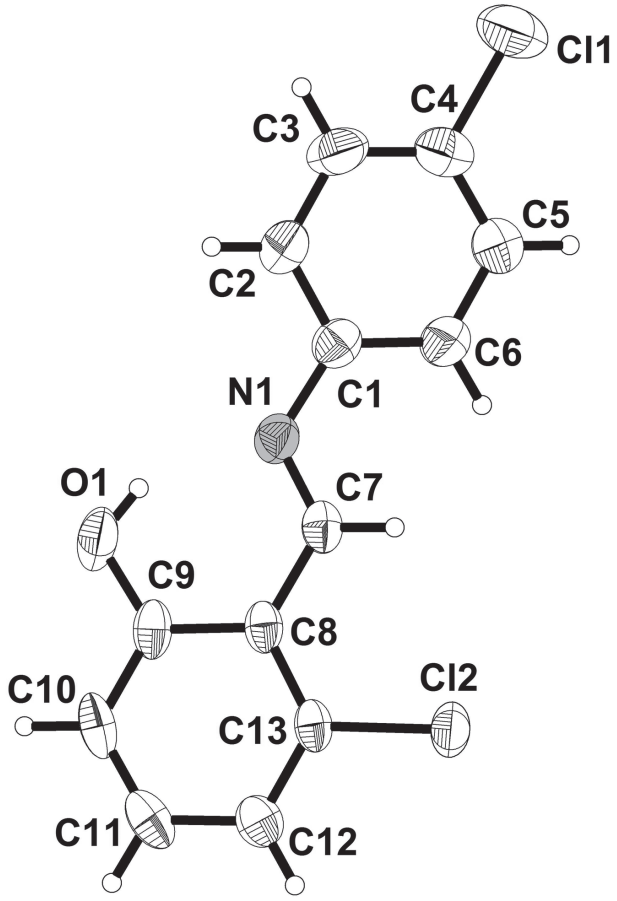
Crystal structure of (*E*)-3-chloro-2-(((4-chlorophenyl)imino)methyl)phenol, $C_{13}H_9Cl_2NO$



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Abstract

$C_{13}H_9Cl_2NO$, monoclinic, $P2_1/c$ (no. 14), $a = 21.039(6)$ Å, $b = 4.0172(12)$ Å, $c = 14.561(4)$ Å, $\beta = 97.849(5)°$, $V = 1219.1(6)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0378$, $wR_{ref}(F^2) = 0.1211$, $T = 293(2)$ K.

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The asymmetric unit of the title molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.20 × 0.15 × 0.12 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.51 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	5658, 2138, 0.028
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1724
$N(param)_{refined}$:	155
Programs:	Bruker [1], SHELX [2]

Source of material

A mixture of 4-chloroaniline (1 mmol, 127.6 mg), 2-chloro-6-hydroxybenzaldehyde (1 mmol, 156.6 mg) and a few drops of glacial acetic acid in anhydrous ethanol (20 mL) was kept under reflux conditions for 4 h. The solvent was removed under reduced pressure and the solid product was recrystallized from 15 mL of anhydrous tetrahydrofuran. After three days, yellow block crystals were obtained.

Experimental details

All H atoms were placed in idealized positions (C—H = 0.93 Å, O—H = 0.82 Å) and refined using a riding model approximation. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atom for oxygen H atoms and $1.2U_{eq}$ for the remaining H atoms.

Comment

Schiff-bases and their derivatives have received continuous attention and been investigated for many years due to their bioactivity, pharmacy and applications in coordination chemistry. Especial for the Schiff bases containing halogen atom are particular interest because of their higher biological activities [3–7]. In order to search for new Schiff bases

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.69103(10)	0.4769(6)	0.99961(16)	0.0444(5)
C2	0.63083(12)	0.5731(7)	0.95752(18)	0.0589(7)
H2	0.6213	0.5646	0.8933	0.071*
C3	0.58465(12)	0.6818(7)	1.0098(2)	0.0652(8)
H3	0.5443	0.7450	0.9809	0.078*
C4	0.59873(12)	0.6956(6)	1.10418(19)	0.0551(7)
C5	0.65817(13)	0.6081(7)	1.14736(18)	0.0644(7)
H5	0.6674	0.6226	1.2115	0.077*
C6	0.70437(12)	0.4985(7)	1.09604(17)	0.0584(7)
H6	0.7447	0.4386	1.1258	0.070*
C7	0.78474(10)	0.1915(5)	0.97360(15)	0.0392(5)
H7	0.7875	0.1106	1.0339	0.047*
C8	0.83479(10)	0.1059(5)	0.91790(13)	0.0354(5)
C9	0.83110(11)	0.2145(5)	0.82472(14)	0.0421(5)
C10	0.88006(13)	0.1395(6)	0.77278(15)	0.0500(6)
H10	0.8771	0.2115	0.7116	0.060*
C11	0.93244(12)	−0.0390(6)	0.81080(16)	0.0505(6)
H11	0.9647	−0.0867	0.7751	0.061*
C12	0.93838(11)	−0.1510(6)	0.90233(15)	0.0454(6)
H12	0.9742	−0.2712	0.9281	0.055*
C13	0.88961(10)	−0.0782(5)	0.95331(13)	0.0363(5)
Cl1	0.54094(4)	0.8360(2)	1.17083(6)	0.0811(3)
Cl2	0.89754(3)	−0.22329(15)	1.06773(4)	0.0481(2)
N1	0.73676(8)	0.3758(5)	0.94184(12)	0.0436(5)
O1	0.78048(9)	0.3933(5)	0.78446(10)	0.0592(5)
H1	0.7560	0.4300	0.8225	0.089*

containing halogen substituents, the title compound was synthesized and its crystal structure determined.

The asymmetric unit of the title compound consists of one formula unit (*cf*. the figure). The title compound has an *E* configuration. In the crystal structure of title compound, the C=N double bond length is 1.286(3) Å (C7=N1), exhibiting the double-double bond character. The dihedral angle formed by the two aromatic rings is 23.4°. All geometric parameters are similar to those in the parent structure [8]. In the crystal structure, the molecules are linked into two-dimensional networks by weak C—H···O and C—H···Cl hydrogen bonds. There also exists one intramolecular O—H···N hydrogen bond, which further consolidate the crystal packing.

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