Crystal structure of *N*-(3-hydroxyphenyl)-5-chloro-salicylideneimine, $C_{13}H_{10}ClNO_2$

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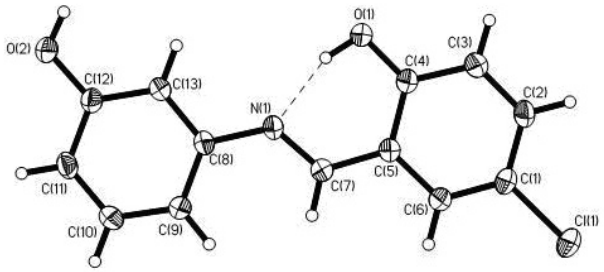

Abstract

$C_{13}H_{10}ClNO_2$, monoclinic, $P2_1/n$ (no. 14), a = 10.149(7) Å, b = 5.925(4) Å, c = 18.27(1) Å, β = 90.84(1)°, V = 1098.5 Å^3, Z = 4, $R_{gt}(F)$ = 0.046, $wR_{ref}(F^2)$ = 0.164, T = 293 K.

Source of material

To the solution of *m*-hydroxyaniline (1.09 g, 10 mmol) dissolved in 50 ml ethanol, was added 5-cholosalicylaldehyde (1.57 g, 10 mmol), and the mixture was refluxed for 0.5 h. Then the solution was evaporated slowly at room temperature to obtain orange red prismatic crystals suitable for X-ray structure determination.

Experimental details

The H atoms were positioned geometrically with d(C—H) = 0.93 - 0.98 Å and refined as riding with U_{iso}(H) = 1.2 U_{eq}(carrier) or 1.5 U_{eq}(methyl).

Discussion

Proton tautomerization plays an important role in many fields of chemistry and biochemistry. Schiff bases of salicylaldehyde with amines (anils) comprise a chemical system undergoing hydrogen-atom tautomerism between enol and keto forms and show the phenomena of solid state photochromism and thermochromism [1-3].

In the title compound, the bond length of C4—O1 (1.302(2) Å) is shorter than that of O2—C12 (1.354(2) Å) indicating hydrogen-atom tautomerism between O1–H1O–N1. Supposedly, there should be intramolecular proton transfer from the hydroxyl O atom to the imine N atom in the solid state of the compound [4]. The dihedral angle between the plane (N1–C7–C5–C4–O1–H1O) and the plane (C1–C2–C3–C4–C5–C6) is 2.7°, which shows the hydrogen bonded ring is almost coplanar with the adjacent ring.

Table 1. Data collection and handling.

Crystal:	orange red prism, size 0.15 × 0.20 × 0.26 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	3.34 cm^{-1}
Diffractometer, scan mode:	Bruker Apex CCD, ω
$2\theta_{max}$:	52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6200, 2150
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1926
$N(param)_{refined}$:	154
Programs:	SHELXTL [4]

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

Atom	Site	x	y	z	U_{iso}
H(2O)	4*e*	1.3094	0.6310	0.5320	0.087
H(1O)	4*e*	0.8406	0.6395	0.4018	0.076
H(7A)	4*e*	0.8707	1.1387	0.3401	0.047
H(6A)	4*e*	0.6557	1.1802	0.2844	0.049
H(13A)	4*e*	1.1126	0.6732	0.4681	0.046
H(9A)	4*e*	1.0603	1.2713	0.3677	0.053
H(11A)	4*e*	1.4066	1.1264	0.4656	0.053
H(2B)	4*e*	0.3842	0.6971	0.3050	0.055
H(3A)	4*e*	0.5298	0.4751	0.3684	0.059
H(10A)	4*e*	1.2744	1.3681	0.3997	0.057

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*e*	0.39363(5)	1.12856(9)	0.24194(2)	0.0435(4)	0.0619(4)	0.0587(4)	0.0077(2)	–0.0166(3)	0.0078(2)
N(1)	4*e*	0.9376(1)	0.8850(2)	0.39502(7)	0.0300(8)	0.0417(8)	0.0417(8)	–0.0043(6)	–0.0042(6)	0.0013(6)
O(2)	4*e*	1.3528(1)	0.7387(2)	0.51328(8)	0.0352(8)	0.060(1)	0.078(1)	–0.0068(7)	–0.0165(7)	0.0231(8)
O(1)	4*e*	0.7707(1)	0.5606(2)	0.40250(8)	0.0374(7)	0.0454(8)	0.0681(8)	–0.0060(6)	–0.0123(6)	0.0168(6)
C(7)	4*e*	0.8497(2)	0.9956(3)	0.35728(9)	0.0354(9)	0.036(1)	0.0455(9)	–0.0019(7)	–0.0027(7)	0.0035(7)

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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(6)	4*e*	0.6314(2)	1.0389(3)	0.30178(9)	0.0375(9)	0.041(1)	0.0424(9)	–0.0016(8)	–0.0041(7)	0.0057(7)
C(5)	4*e*	0.7233(2)	0.9067(3)	0.34134(8)	0.0325(9)	0.0379(9)	0.0383(8)	–0.0012(7)	–0.0017(7)	0.0004(7)
C(8)	4*e*	1.0656(2)	0.9614(3)	0.41402(8)	0.0290(8)	0.040(1)	0.0371(8)	–0.0039(7)	–0.0003(6)	–0.0016(6)
C(13)	4*e*	1.1450(2)	0.8136(3)	0.45434(9)	0.0337(9)	0.0379(9)	0.0432(8)	–0.0053(7)	–0.0019(7)	0.0034(7)
C(4)	4*e*	0.6871(2)	0.6877(3)	0.36675(9)	0.0343(9)	0.042(1)	0.0413(8)	–0.0015(7)	–0.0048(7)	0.0027(7)
C(12)	4*e*	1.2720(2)	0.8760(3)	0.47394(9)	0.0318(9)	0.044(1)	0.0423(9)	–0.0008(7)	–0.0028(7)	0.0021(7)
C(9)	4*e*	1.1133(2)	1.1709(3)	0.3939(1)	0.037(1)	0.041(1)	0.054(1)	–0.0034(8)	–0.0075(8)	0.0106(8)
C(11)	4*e*	1.3209(2)	1.0846(3)	0.4530(1)	0.0296(9)	0.051(1)	0.051(1)	–0.0101(8)	–0.0022(7)	0.0004(8)
C(2)	4*e*	0.4695(2)	0.7484(3)	0.3141(1)	0.0329(9)	0.054(1)	0.051(1)	–0.0066(8)	–0.0088(8)	0.0021(8)
C(1)	4*e*	0.5077(2)	0.9608(3)	0.28898(8)	0.0349(9)	0.048(1)	0.0375(8)	0.0046(8)	–0.0053(7)	–0.0009(7)
C(3)	4*e*	0.5568(2)	0.6160(3)	0.3520(1)	0.042(1)	0.045(1)	0.059(1)	–0.0093(8)	–0.0087(8)	0.0074(8)
C(10)	4*e*	1.2415(2)	1.2283(3)	0.4136(1)	0.041(1)	0.041(1)	0.061(1)	–0.0097(8)	–0.0011(8)	0.0076(8)

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