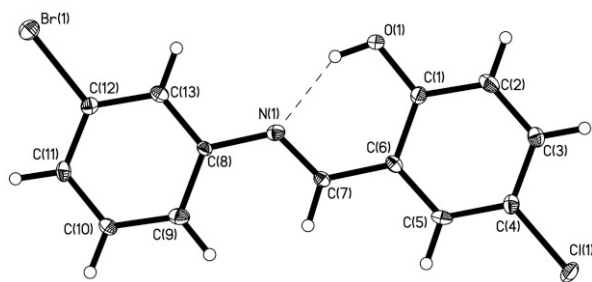


Crystal structure of 2-[(3-bromo-phenylimino)-methyl]-4-chloro-phenol, C₁₃H₉BrClNO

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Abstract

C₁₃H₉BrClNO, monoclinic, *Pc* (no. 7), $a = 10.929(4)$ Å, $b = 4.3884(16)$ Å, $c = 12.267(5)$ Å, $\beta = 95.920(7)^\circ$, $V = 585.2(4)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.039$, $wR_{\text{ref}}(F^2) = 0.075$, $T = 153(2)$ K

Table 1. Data collection and handling.

Crystal:	orange plates, size 0.51×0.19×0.03 mm
Wavelength:	Mo K_{α} radiation (0.71073 Å)
μ :	37.20 cm ⁻¹
Diffractometer, scan mode:	AFC10/Saturn724+, φ and ω
$2\theta_{\text{max}}$:	58.30°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	4688, 1572
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1949
$N(\text{param})_{\text{refined}}$:	155
Programs:	SHELXS-97 [7]

Source of material

5-chlorosalicylaldehyde (15.7 mg, 0.1 mmol) was dissolved into 15 mL methanol, then a solution of *m*-bromoaniline (17.2 mg, 0.1 mmol) in 15 mL methanol was added with vigorous stirring. The reaction mixture was refluxed for 1 h and cooled to room temperature, then the orange product appeared. The product was filtered off, washed with cold methanol, and finally dried to obtain the Schiff base 2-[(3-bromo-phenylimino)-methyl]-4-chloro-phenol (yield 67.3%). Single crystals of the title compound suitable for X-ray structure determination were obtained by slow evaporation of the filtrate within seven days at room temperature.

Experimental details

All H atoms bonded to carbon and oxygen atoms were generated geometrically and refined as riding atoms with $d(\text{C}-\text{H}) = 0.95$ Å, $d(\text{O}-\text{H}) = 0.84$ Å and $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{iso}}(\text{C})$ or $1.5 U_{\text{iso}}(\text{O})$.

Discussion

Schiff bases in general have been reported to possess biological properties [1–3]. Metal complexes with Schiff base ligands have played an important role in the development of coordination chemistry due to their preparative accessibility and structural va-

riety [4,5]. In this work, the new Schiff base 2-[(3-bromo-phenylimino)-methyl]-4-chloro-phenol was prepared and characterized by single-crystal X-ray diffraction.

The crystal structure is built up by only the Schiff base molecules, within which all bond lengths and bond angles are in normal ranges [6]. The C7=N1 distance (1.282(5) Å) reveals the typical C=N Schiff base bond and C8–N1 (1.419(5) Å) a single C–N bond. The molecule adopts a *trans* configuration at the C=N bond and the whole molecule is nearly coplanar with the dihedral angle between the two aromatic rings being $1.52(3)^\circ$, which is interpreted as conjugation effect and stabilization of the ring by an intramolecular O–H–N hydrogen bond.

The discrete molecules are held together as a three-dimensional network structure by the intermolecular C12–Br1–H1O ($x, -y+3, -\frac{1}{2}+z$) and Br1–Cl1 ($x-1, -y+1, -\frac{1}{2}+z$ and $x-1, -y+2, -\frac{1}{2}+z$) interactions with the distance of Br1–Br1 of 3.557 Å and 3.729 Å, respectively. The hydrogen bonds and halogen-halogen interactions play very important roles in the formation, stability and crystallization of the title compound.

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(10)	2a	0.3252	0.5938	0.4340	0.030
H(2)	2a	0.5428	0.0998	0.3778	0.030
H(3)	2a	0.6978	-0.0813	0.5059	0.025
H(5)	2a	0.5541	0.3968	0.7434	0.024
H(7)	2a	0.3733	0.7127	0.7043	0.021
H(9)	2a	0.2661	1.0318	0.7618	0.028
H(10)	2a	0.1176	1.3827	0.8079	0.027
H(11)	2a	-0.0340	1.5574	0.6752	0.024
H(13)	2a	0.1121	1.0539	0.4461	0.022

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Br(1)	2a	-0.09539(5)	1.46589(9)	0.44113(5)	0.0225(2)	0.0349(3)	0.0277(3)	0.0038(4)	-0.00398(18)	-0.0008(5)
Cl(1)	2a	0.75168(10)	0.0015(3)	0.73278(10)	0.0185(7)	0.0351(8)	0.0269(7)	0.0059(5)	-0.0038(5)	0.0042(6)
O(1)	2a	0.3730(4)	0.4758(8)	0.4049(3)	0.026(2)	0.035(3)	0.015(2)	0.0108(17)	0.0011(16)	-0.0002(18)
N(1)	2a	0.2887(4)	0.7980(8)	0.5621(3)	0.025(2)	0.015(2)	0.016(2)	-0.0013(16)	-0.0004(18)	0.0028(17)
C(1)	2a	0.4582(6)	0.3727(13)	0.4810(5)	0.016(3)	0.016(3)	0.022(3)	0.001(2)	0.000(2)	-0.003(2)
C(2)	2a	0.5458(5)	0.1657(11)	0.4517(4)	0.037(3)	0.019(3)	0.021(3)	0.002(2)	0.009(3)	-0.005(2)
C(3)	2a	0.6370(4)	0.0540(11)	0.5277(4)	0.022(3)	0.018(2)	0.024(3)	0.002(2)	0.007(2)	0.002(2)
C(4)	2a	0.6382(4)	0.1428(11)	0.6364(4)	0.018(3)	0.021(3)	0.023(3)	0.003(2)	0.006(2)	0.004(2)
C(5)	2a	0.5514(4)	0.3395(10)	0.6685(4)	0.030(3)	0.019(2)	0.011(3)	-0.006(2)	0.001(2)	-0.001(2)
C(6)	2a	0.4593(4)	0.4571(10)	0.5930(4)	0.018(3)	0.016(3)	0.017(3)	-0.003(2)	0.007(2)	0.000(2)
C(7)	2a	0.3708(4)	0.6678(10)	0.6284(4)	0.020(3)	0.015(2)	0.018(3)	0.000(2)	0.000(2)	-0.006(2)
C(8)	2a	0.2025(4)	1.0079(9)	0.5976(4)	0.020(3)	0.013(2)	0.011(2)	0.0020(18)	0.0044(19)	0.0004(19)
C(9)	2a	0.2051(5)	1.1057(11)	0.7074(4)	0.030(3)	0.023(3)	0.017(3)	-0.004(2)	0.002(2)	0.002(2)
C(10)	2a	0.1164(5)	1.3131(11)	0.7345(4)	0.024(3)	0.027(3)	0.015(3)	0.004(2)	0.006(2)	-0.002(2)
C(11)	2a	0.0268(4)	1.4186(11)	0.6559(4)	0.013(3)	0.021(2)	0.027(3)	0.004(2)	0.006(2)	0.000(2)
C(12)	2a	0.0266(4)	1.3213(10)	0.5499(4)	0.011(3)	0.021(3)	0.020(3)	-0.0026(19)	0.000(2)	0.000(2)
C(13)	2a	0.1136(4)	1.1179(10)	0.5202(4)	0.020(3)	0.016(2)	0.019(3)	-0.005(2)	0.000(2)	-0.006(2)

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