Crystal structure of 2-[(2-bromophenylimino)methyl]-4-chlorophenol, $C_{13}H_9BrClNO$

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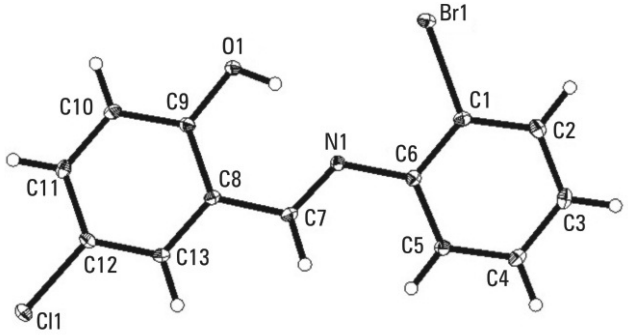

Abstract

$C_{13}H_9BrClNO$, orthorhombic, *Pbca* (no. 61), a = 7.102(1) Å, b = 13.082(2) Å, c = 25.333(5) Å, V = 2353.6 Å^3, Z = 8, $R_{gt}(F)$ = 0.025, $wR_{ref}(F^2)$ = 0.058, T = 103 K.

Source of material

5-Chlorosalicylaldehyde (0.5 mmol) and an equimolar quantity of 2-bromoaniline were dissolved in methanol (50 mL). The reaction mixture was stirred at room temperature for 30 min and allowed to stand in air. Yellow crystals were formed after 5 days. They were filtered and washed with small amounts of cooled methanol, and finally recrystallized from methanol in good yield (76.8 %).

Experimental details

H(1O) atom was located from Fourier difference map and refined. All other H atoms were generated geometrically and allowed to ride on their parent atoms with d(C—H) = 0.95 Å and d(O—H) = 0.83(1) Å, and U_{iso}(H) = 1.2 U_{eq}(C) and 1.5 U_{eq}(O).

Discussion

Schiff bases are important organic ligands forming metal complexes. They have demonstrated significant biological activities, e. g., antimicrobial [1], antitumour and antiviral activities [2-4]. The title crystal structure is built up only by the Schiff base molecules, within which all bond lengths and bond angles are in normal ranges [5]. The distance d(C7—N1) = 1.287(2) Å reveals the typical C=N Schiff base bond and the molecule adopts a *trans* configuration about the C=N. The whole molecule is nearly coplanar with the dihedral angle between two phenyl rings of 9.59°, which is interpreted by conjugation effect and the stabilization of rings from intramolecular hydrogen bond. In the structure, the bromine atom at the phenyl ring is slightly twisted out of the ring with the torsion angle Br1–C1–C2–C3 = –179.9(1)° and the hydroxyl group nearly oriented out of the phenyl ring, as indicated by the torsion angle C13–C8–C9–O1 = 179.6(2)°, which is due to the π-conjugate effect of atoms possessing unshared electron pairs and phenyl rings. Discrete molecules are held together by intermolecular hydrogen bonds C13···H13–O1 ($-x+\frac{3}{2},\frac{1}{2}+y,z$), which connect the molecules to form a zig-zag chain along [010]. The zig-zag chains are interlinked to form a two dimensional network through C···H–Cl ($x-\frac{1}{2},-y+\frac{3}{2},-z+1$) hydrogen bonding and π–π interactions with center-to-center distances of 3.656 Å and interplane distances of 3.313 Å [6].

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.27 × 0.29 × 0.32 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	37.00 cm^{-1}
Diffractometer, scan mode:	AFC10/Saturn724+, φ/ω
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	17077, 2703
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2373
$N(param)_{refined}$:	158
Programs:	SHELXS-97, SHELXL-97 [7]

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

Atom	Site	x	y	z	U_{iso}
H(2)	8*c*	0.4433	0.3929	0.7562	0.022
H(3)	8*c*	0.3809	0.5630	0.7786	0.023
H(4)	8*c*	0.4355	0.6928	0.7171	0.022
H(5)	8*c*	0.5506	0.6533	0.6335	0.019
H(7)	8*c*	0.6872	0.6044	0.5669	0.016
H(10)	8*c*	0.8131	0.2981	0.4320	0.021
H(11)	8*c*	0.9015	0.4228	0.3712	0.019
H(13)	8*c*	0.8005	0.6447	0.4788	0.016
H(1O)	8*c*	0.667(4)	0.349(2)	0.5543(7)	0.051(9)

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}
Br(1)	8*c*	0.58264(3)	0.28403(1)	0.665137(8)	0.0291(1)	0.0118(1)	0.0208(1)	0.00094(8)	0.00576(8)	0.00281(7)
Cl(1)	8*c*	0.92737(7)	0.63601(4)	0.37369(2)	0.0255(2)	0.0172(2)	0.0158(2)	–0.0012(2)	0.0037(2)	0.0043(2)
O(1)	8*c*	0.6965(2)	0.3216(1)	0.52598(5)	0.0377(9)	0.0114(6)	0.0160(7)	–0.0033(6)	0.0066(6)	–0.0005(5)

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Table 3. Continued.

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	8*c*	0.6302(2)	0.4687(1)	0.59330(6)	0.0158(7)	0.0132(7)	0.0104(7)	0.0002(6)	–0.0015(6)	–0.0004(6)
C(1)	8*c*	0.5344(3)	0.4231(1)	0.68163(7)	0.0139(9)	0.0122(9)	0.0171(9)	–0.0003(7)	–0.0014(7)	–0.0008(7)
C(2)	8*c*	0.4652(3)	0.4459(2)	0.73136(8)	0.018(1)	0.021(1)	0.0164(9)	–0.0014(8)	0.0016(7)	0.0025(7)
C(3)	8*c*	0.4283(3)	0.5466(2)	0.74459(8)	0.0185(9)	0.025(1)	0.0143(9)	0.0004(8)	0.0022(8)	–0.0038(8)
C(4)	8*c*	0.4606(3)	0.6236(2)	0.70803(8)	0.020(1)	0.0164(9)	0.0186(9)	0.0019(7)	–0.0009(8)	–0.0045(7)
C(5)	8*c*	0.5294(3)	0.5999(2)	0.65825(7)	0.0196(9)	0.0134(9)	0.0152(9)	–0.0003(7)	–0.0014(7)	0.0003(7)
C(6)	8*c*	0.5680(2)	0.4989(1)	0.64387(7)	0.0121(9)	0.0147(9)	0.0122(8)	–0.0004(7)	–0.0026(7)	–0.0008(7)
C(7)	8*c*	0.6851(2)	0.5337(1)	0.55844(7)	0.0148(8)	0.0106(8)	0.0149(8)	0.0013(7)	–0.0027(7)	–0.0019(7)
C(8)	8*c*	0.7440(2)	0.5005(1)	0.50640(7)	0.0132(8)	0.0114(8)	0.0130(8)	0.0009(7)	–0.0024(7)	–0.0009(7)
C(9)	8*c*	0.7484(3)	0.3961(1)	0.49214(7)	0.0187(9)	0.0126(8)	0.0146(8)	–0.0021(7)	0.0005(7)	0.0007(7)
C(10)	8*c*	0.8087(3)	0.3683(1)	0.44164(7)	0.024(1)	0.0117(9)	0.0174(9)	–0.0021(7)	0.0007(8)	–0.0034(7)
C(11)	8*c*	0.8619(3)	0.4421(1)	0.40562(7)	0.0177(9)	0.018(1)	0.0124(9)	0.0004(7)	–0.0002(7)	–0.0034(7)
C(12)	8*c*	0.8573(2)	0.5443(1)	0.41977(7)	0.0134(8)	0.0142(9)	0.0140(9)	–0.0014(7)	–0.0012(7)	0.0032(7)
C(13)	8*c*	0.8009(2)	0.5743(1)	0.46951(7)	0.0140(9)	0.0109(8)	0.0160(9)	0.0006(7)	–0.0016(7)	0.0002(7)

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