Crystal structure of 2-bromo-4-chloro-6-[(4-bromo-phenylimino)methyl]phenol, $C_{13}H_8Br_2ClNO$

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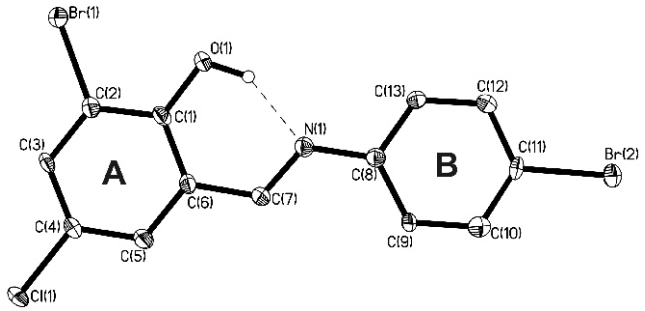

Abstract

$C_{13}H_8Br_2ClNO$, triclinic, $P\bar{1}$ (no. 2), a = 7.941(5) Å, b = 8.486(6) Å, c = 11.024(8) Å, α = 86.83(3)°, β = 75.39(3)°, γ = 64.50(2)°, V = 647.6 Å^3, Z = 2, $R_{gt}(F)$ = 0.057, $wR_{ref}(F^2)$ = 0.138, T = 173 K.

Source of material

3-Bromo-5-chlorosalicylaldehyde (23.5 mg, 0.1 mmol) dissolved in methanol (10 mL) was added to 4-bromoaniline (17.2 mg, 0.1 mmol) in methanol (10 mL). The reaction mixture was heated to 313 K for 2 h and then cooled to room temperature followed by concentrating the resulting mixture to give an orange product. The product was filtered off, washed with cold methanol, and finally dried in vacuum (yield 75.7 %).

Experimental details

All H atoms on carbon atoms were generated geometrically and refined as riding atoms with d(C—H) = 0.95 Å, d(O—H) = 0.93 Å and U_{iso}(H) = 1.2 U_{iso}(C) or 1.5 U_{iso}(O).

Discussion

Schiff bases are new important organic ligands forming metal complexes and they have in general been reported to possess antimicrobial [1,2] and antitumour activities [3-5].

The title 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 distance d(C7=N1) = 1.297(7) Å reveals the typical C=N Schiff base bond and d(C8—N1) = 1.407(7) Å a typical single bond C—N. The molecule adopts a *trans* configuration about the C=N. The six-membered ring (C1,C6,C7,O1, H1O,N1) is formed by the intramolecular hydrogen bond O1–H1···N1. The ring stabilization by intramolecular hydrogen bond and conjugation effect make the whole molecule nearly coplanar with a dihedral angle of 5.33(4)° between ring A(C1-C6) and ring B(C8-C13) of Schiff base ligand. Two 2-bromo-4-chloro-6-[(4-bromo-phenylimino)methyl]phenol molecules arrange in an anti-parallel fashion due to the aromatic π–π stacking interactions existing between them, which link two molecules into a dimer. The rings A and B of two neighbouring molecules at (x,y,z) and (–x+2,–y+1,–z+1) stack with the interplanar distances being 3.19 Å and 3.22 Å, respectively, and both inter-centroid distances are 3.635 Å. All the ring-centroid and interplanar distances lie in the normal range of 3.3-3.8 Å, indicative of π–π stacking interactions [7]. The dimers are further linked by C12–H12···O1 and C12–H12···Br1 from same molecule at (–x+2,–y,–z+1) to form a 2D network structure. The hydrogen bonds and π–π stacking interactions play very important roles in the formation, stability and crystallization of the title compound.

Table 1. Data collection and handling.

Crystal:	orange chunk, size 0.15 × 0.20 × 0.24 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	64.49 cm^{-1}
Diffractometer, scan mode:	AFC10/Saturn724+, φ/ω
$2\theta_{max}$:	58.26°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6807, 3357
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1561
$N(param)_{refined}$:	163
Programs:	SHELXS-97, SHELXL-97 [8]

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

Atom	Site	x	y	z	U_{iso}
H(1O)	2*i*	0.8248	0.2837	0.4183	0.038
H(3)	2*i*	0.4153	0.7259	0.1541	0.033
H(5)	2*i*	0.5025	0.8578	0.4734	0.040
H(7)	2*i*	0.6920	0.6373	0.5948	0.035
H(9)	2*i*	0.8399	0.5792	0.7377	0.033
H(10)	2*i*	0.9869	0.4717	0.9015	0.044
H(12)	2*i*	1.1795	–0.0237	0.7630	0.041
H(13)	2*i*	1.0427	0.0834	0.5947	0.035

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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)	2*i*	0.6275(1)	0.34370(9)	0.11921(7)	0.0523(6)	0.0279(4)	0.0353(5)	–0.0026(4)	–0.0244(4)	–0.0023(3)
Br(2)	2*i*	1.2038(1)	0.1331(1)	0.98498(7)	0.0563(6)	0.0320(4)	0.0360(5)	–0.0092(4)	–0.0291(4)	0.0069(3)
Cl(1)	2*i*	0.2968(3)	1.0357(2)	0.2957(2)	0.053(1)	0.0169(9)	0.048(1)	–0.0054(8)	–0.027(1)	0.0068(8)
O(1)	2*i*	0.7969(7)	0.2987(6)	0.3399(4)	0.041(3)	0.020(2)	0.033(3)	–0.005(2)	–0.021(3)	0.003(2)
N(1)	2*i*	0.8485(8)	0.3908(7)	0.5420(5)	0.026(3)	0.023(3)	0.028(3)	–0.013(3)	–0.011(3)	0.005(3)
C(1)	2*i*	0.682(1)	0.4660(8)	0.3331(6)	0.021(4)	0.018(3)	0.034(4)	–0.006(3)	–0.013(3)	0.006(3)
C(2)	2*i*	0.590(1)	0.5209(9)	0.2337(6)	0.033(5)	0.026(4)	0.027(4)	–0.009(3)	–0.013(4)	0.004(3)
C(3)	2*i*	0.474(1)	0.6934(8)	0.2224(6)	0.034(5)	0.018(3)	0.030(4)	–0.006(3)	–0.020(4)	0.010(3)
C(4)	2*i*	0.442(1)	0.8187(9)	0.3102(7)	0.054(6)	0.021(4)	0.033(4)	–0.012(4)	–0.022(4)	0.009(3)
C(5)	2*i*	0.526(1)	0.7709(9)	0.4125(7)	0.042(5)	0.019(4)	0.045(5)	–0.010(3)	–0.026(4)	0.006(3)
C(6)	2*i*	0.644(1)	0.5962(8)	0.4243(6)	0.029(4)	0.022(3)	0.026(4)	–0.011(3)	–0.013(3)	0.007(3)
C(7)	2*i*	0.727(1)	0.5496(8)	0.5313(6)	0.044(5)	0.018(3)	0.028(4)	–0.013(3)	–0.016(4)	0.006(3)
C(8)	2*i*	0.926(1)	0.3411(8)	0.6470(6)	0.027(4)	0.021(3)	0.026(4)	–0.012(3)	–0.008(3)	0.002(3)
C(9)	2*i*	0.910(1)	0.4566(8)	0.7417(6)	0.042(5)	0.020(3)	0.019(4)	–0.006(3)	–0.017(4)	0.004(3)
C(10)	2*i*	0.995(1)	0.3931(9)	0.8398(7)	0.047(5)	0.026(4)	0.035(4)	–0.012(4)	–0.013(4)	–0.001(3)
C(11)	2*i*	1.091(1)	0.2172(9)	0.8476(6)	0.028(4)	0.033(4)	0.019(4)	–0.013(3)	–0.014(3)	0.006(3)
C(12)	2*i*	1.111(1)	0.0986(9)	0.7570(6)	0.047(5)	0.018(3)	0.037(4)	–0.008(3)	–0.020(4)	0.002(3)
C(13)	2*i*	1.029(1)	0.1633(8)	0.6577(6)	0.041(5)	0.017(3)	0.036(4)	–0.013(3)	–0.020(4)	0.003(3)

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