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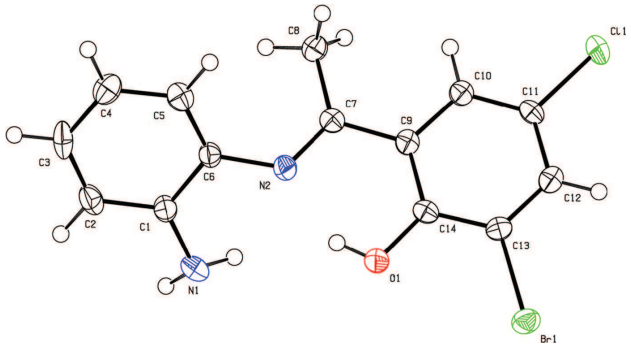
Crystal structure of (*E*)-2-(1-((2-aminophenyl)-imino)ethyl)-6-bromo-4-chlorophenol, $C_{14}H_{12}BrClN_2O$



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Abstract

$C_{14}H_{12}BrClN_2O$, monoclinic, $P2_1/c$ (no. 14), $a = 3.9454(10)$ Å, $b = 24.189(6)$ Å, $c = 13.728(3)$ Å, $\beta = 90.07°$, $V = 643.99(7)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0589$, $wR_{ref}(F^2) = 0.1584$, $T = 293(2)$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

1,2-Diaminobenzene (1 mmol, 0.108 g) and 1-(3-bromo-5-chloro-2-hydroxyphenyl)ethanone (1 mmol, 0.249 g) were added to the solution of ethanol (10 mL), then the mixture was refluxed 2 h. After filtering the precipitate was disolved in mixed solvents of dichloromethane ethyl acetate and evaporated slowly at room temperature to obtain colorless prismatic crystals.

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Table 1: Data collection and handling.

Crystal:	Colorless block
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	3.33 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω-scans
θ_{max}, completeness:	27.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	11411, 3001, 0.188
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2117
$N(param)_{refined}$:	175
Programs:	Bruker programs [1], SHELX [2], OLEX2 [3]

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

Atom	x	y	z	U_{iso}*/U_{eq}
Br1	−0.29045(12)	0.38586(2)	−0.39960(3)	0.0277(2)
Cl1	−0.7092(3)	0.44311(5)	−0.02995(9)	0.0332(3)
O1	−0.5796(9)	0.27468(14)	−0.3354(2)	0.0269(8)
H1	−0.636253	0.244189	−0.314952	0.040*
N1	−1.1860(11)	0.15480(19)	−0.3723(3)	0.0304(10)
H1B	−1.357243	0.139598	−0.401683	0.036*
H1A	−1.243616	0.187889	−0.354315	0.036*
N2	−0.8479(10)	0.20647(16)	−0.2209(3)	0.0223(8)
C1	−1.0962(12)	0.12366(19)	−0.2914(4)	0.0246(10)
C2	−1.1520(12)	0.0664(2)	−0.2866(4)	0.0292(11)
H2	−1.260622	0.048644	−0.337928	0.035*
C3	−1.0476(12)	0.0360(2)	−0.2062(4)	0.0318(12)
H3	−1.087639	−0.001907	−0.204233	0.038*
C4	−0.8861(12)	0.0613(2)	−0.1299(4)	0.0292(11)
H4	−0.816975	0.040701	−0.076220	0.035*
C5	−0.8262(12)	0.1177(2)	−0.1330(4)	0.0261(11)
H5	−0.714677	0.134561	−0.081267	0.031*
C6	−0.9287(11)	0.14928(19)	−0.2114(3)	0.0214(10)
C7	−0.8896(11)	0.24310(19)	−0.1532(3)	0.0202(9)
C8	−1.0634(12)	0.2315(2)	−0.0589(3)	0.0266(10)
H8B	−0.897795	0.228707	−0.007804	0.040*
H8A	−1.185853	0.197268	−0.063746	0.040*
H8C	−1.218439	0.260914	−0.044358	0.040*
C9	−0.7633(11)	0.29912(19)	−0.1738(3)	0.0197(9)
C10	−0.7896(11)	0.3403(2)	−0.1022(3)	0.0223(10)
H10	−0.890604	0.332307	−0.042624	0.027*
C11	−0.6659(11)	0.3925(2)	−0.1201(3)	0.0217(10)
C12	−0.5146(11)	0.4060(2)	−0.2078(3)	0.0222(10)
H12	−0.432676	0.441500	−0.218438	0.027*
C13	−0.4867(11)	0.3665(2)	−0.2787(3)	0.0207(9)
C14	−0.6074(11)	0.31188(19)	−0.2641(3)	0.0199(9)

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

Mono-imine with *o*-amino is a class of important tridentate ligands. Their complexes can be used for catalysis [4]. Mono-imine with *o*-amino is also a key intermediate for the synthesis of unsymmetrical tetradentate bis-Schiff base ligands [5].

In the crystal structure of the title compound, the dihedral angle between the two benzene rings (C1—C2—C3—C4—C5—C6 and C9—C10—C11—C12—C13—C14) is 48.25°. Bond lengths and angles are in the expected ranges and similar to a related structure [6]. There is an intramolecular O—H···N hydrogen bond (*cf*. the figure). The packing of crystal structure is *via* van der Waals forces.

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