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2-Benzyliminomethyl-6-bromo-4-chlorophenol

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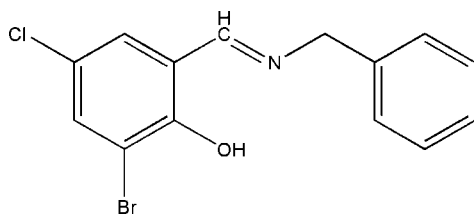
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.011$ Å; R factor = 0.068; wR factor = 0.177; data-to-parameter ratio = 14.3.

The title molecule, $\text{C}_{14}\text{H}_{11}\text{BrClNO}$, adopts a *trans* configuration with respect to the $\text{C}=\text{N}$ double bond. The dihedral angle between the two aromatic rings is $70.4(5)^\circ$. An intramolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bond is observed between the hydroxyl and imine groups.

Related literature

For related literature, see: Ali *et al.* (2002); Cukurovali *et al.* (2002); Tarafder *et al.* (2002). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$\text{C}_{14}\text{H}_{11}\text{BrClNO}$
 $M_r = 324.60$
Monoclinic, $P2_1/c$
 $a = 4.3334(8)$ Å

$b = 12.8976(14)$ Å
 $c = 23.892(2)$ Å
 $\beta = 92.992(1)^\circ$
 $V = 1333.5(3)$ Å³

$Z = 4$
Mo $K\alpha$ radiation
 $\mu = 3.27$ mm⁻¹

$T = 298(2)$ K
 $0.40 \times 0.37 \times 0.13$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.355$, $T_{\max} = 0.676$

6753 measured reflections
2325 independent reflections
1699 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.068$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.067$
 $wR(F^2) = 0.176$
 $S = 1.06$
2325 reflections

163 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 1.47$ e Å⁻³
 $\Delta\rho_{\min} = -0.76$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--------------------------------------|-------|-------------|-------------|---------------|
| $\text{O1}-\text{H1}\cdots\text{N1}$ | 0.82 | 1.86 | 2.590 (7) | 147 |

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2649).

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supplementary materials

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Comment

Schiff base compounds have been of great interest for many years. These compounds play an important role in the development of coordination chemistry related to catalysis and enzymatic reactions, magnetism and molecular architectures. As an extension of the work on the structural characterization of Schiff base compounds, the crystal structure of the title compound is reported here.

Bond lengths in the title molecule (Fig. 1) have normal values (Allen *et al.*, 1987). The C1=N1 bond length of 1.267 (9) Å conforms to the value for a double bond. The dihedral angle between the two aromatic rings is 70.4 (5)°. As expected, the molecule adopts a trans configuration about the C=N bond [C8—N1—C1—C2 = -178.6 (6)°]. An intramolecular O—H···N hydrogen bond is observed between hydroxyl and imine groups (Table 1).

Experimental

3-Bromine-5-chlorosalicylaldehyde (0.1 mmol, 23.55 mg) and 1-benzylamine (0.1 mmol, 10.7 mg) were added to methanol (10 ml). The mixture was stirred for 30 min at room temperature to give a clear brown solution. After allowing the resulting solution to stand in air for 7 d, yellow block-shaped crystals of the title compound were formed on slow evaporation of the solvent. The crystals were collected, washed with methanol and dried in a vacuum desiccator using anhydrous CaCl₂ (yield 54%). Analysis found: C 51.76, H 4.0%; calculated for C₁₄H₁₁BrClNO: C 51.77, H 3.39%.

Refinement

All H atoms were placed in geometrically idealized positions [O—H = 0.82 Å and C—H = 0.93–0.97 Å] and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ and $1.5U_{\text{eq}}(\text{O})$. The highest unassigned peak in the difference map is located 0.85 and 1.05 Å from atoms C11 and C6, respectively.

Figures

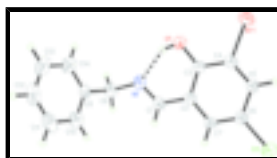


Fig. 1. The molecular structure of the title compound, showing 30% probability ellipsoids. The dashed line represents a hydrogen bond.

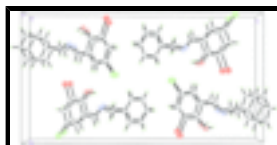


Fig. 2. The crystal packing of the title compound, viewed down the *b* axis.

2-Benzyliminomethyl-6-bromo-4-chlorophenol

Crystal data

| | |
|--------------------------------|---|
| $C_{14}H_{11}BrClNO$ | $F_{000} = 648$ |
| $M_r = 324.60$ | $D_x = 1.617 \text{ Mg m}^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation |
| Hall symbol: -P 2ybc | $\lambda = 0.71073 \text{ \AA}$ |
| $a = 4.3334 (8) \text{ \AA}$ | Cell parameters from 2314 reflections |
| $b = 12.8976 (14) \text{ \AA}$ | $\theta = 3.0\text{--}23.6^\circ$ |
| $c = 23.892 (2) \text{ \AA}$ | $\mu = 3.27 \text{ mm}^{-1}$ |
| $\beta = 92.992 (1)^\circ$ | $T = 298 (2) \text{ K}$ |
| $V = 1333.5 (3) \text{ \AA}^3$ | Block, yellow |
| $Z = 4$ | $0.40 \times 0.37 \times 0.13 \text{ mm}$ |

Data collection

| | |
|---|--|
| Bruker SMART CCD area-detector diffractometer | 2325 independent reflections |
| Radiation source: fine-focus sealed tube | 1699 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{\text{int}} = 0.068$ |
| $T = 298(2) \text{ K}$ | $\theta_{\text{max}} = 25.0^\circ$ |
| φ and ω scans | $\theta_{\text{min}} = 1.7^\circ$ |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1996) | $h = -5 \rightarrow 5$ |
| $T_{\text{min}} = 0.355$, $T_{\text{max}} = 0.676$ | $k = -14 \rightarrow 15$ |
| 6753 measured reflections | $l = -28 \rightarrow 19$ |

Refinement

| | |
|--|--|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.067$ | H-atom parameters constrained |
| $wR(F^2) = 0.176$ | $w = 1/[\sigma^2(F_o^2) + (0.0638P)^2 + 4.7838P]$ |
| $S = 1.06$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 2325 reflections | $(\Delta/\sigma)_{\text{max}} = 0.001$ |
| 163 parameters | $\Delta\rho_{\text{max}} = 1.47 \text{ e \AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | $\Delta\rho_{\text{min}} = -0.76 \text{ e \AA}^{-3}$ |
| | Extinction correction: none |

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|-------------|--------------|--------------|----------------------------------|
| Br1 | 1.1330 (2) | 0.05445 (6) | 0.34840 (4) | 0.0688 (3) |
| Cl1 | 1.1495 (6) | 0.47661 (15) | 0.37758 (10) | 0.0761 (6) |
| N1 | 0.4056 (13) | 0.2399 (4) | 0.1816 (2) | 0.0495 (14) |
| O1 | 0.7218 (12) | 0.1150 (3) | 0.2472 (2) | 0.0588 (13) |
| H1 | 0.6061 | 0.1323 | 0.2205 | 0.088* |
| C1 | 0.4902 (15) | 0.3140 (6) | 0.2137 (3) | 0.0465 (16) |
| H1A | 0.4104 | 0.3798 | 0.2062 | 0.056* |
| C2 | 0.7087 (14) | 0.3002 (5) | 0.2620 (3) | 0.0409 (14) |
| C3 | 0.8091 (15) | 0.1982 (5) | 0.2763 (3) | 0.0419 (15) |
| C4 | 1.0094 (15) | 0.1881 (5) | 0.3238 (3) | 0.0442 (15) |
| C5 | 1.1108 (15) | 0.2704 (5) | 0.3547 (3) | 0.0425 (15) |
| H5 | 1.2421 | 0.2609 | 0.3864 | 0.051* |
| C6 | 1.0135 (16) | 0.3711 (5) | 0.3381 (3) | 0.0476 (16) |
| C7 | 0.8123 (16) | 0.3849 (5) | 0.2934 (3) | 0.0481 (16) |
| H7 | 0.7434 | 0.4511 | 0.2839 | 0.058* |
| C8 | 0.1902 (16) | 0.2605 (6) | 0.1331 (3) | 0.0562 (19) |
| H8A | 0.0875 | 0.3264 | 0.1380 | 0.067* |
| H8B | 0.0339 | 0.2066 | 0.1302 | 0.067* |
| C9 | 0.3671 (15) | 0.2630 (6) | 0.0799 (3) | 0.0480 (17) |
| C10 | 0.4684 (18) | 0.3549 (7) | 0.0600 (3) | 0.066 (2) |
| H10 | 0.4266 | 0.4163 | 0.0786 | 0.080* |
| C11 | 0.635 (2) | 0.3571 (9) | 0.0117 (4) | 0.082 (3) |
| H11 | 0.7064 | 0.4196 | -0.0021 | 0.099* |
| C12 | 0.691 (2) | 0.2664 (10) | -0.0149 (4) | 0.086 (3) |
| H12 | 0.8006 | 0.2675 | -0.0473 | 0.103* |
| C13 | 0.594 (2) | 0.1762 (9) | 0.0043 (4) | 0.084 (3) |
| H13 | 0.6386 | 0.1154 | -0.0146 | 0.101* |
| C14 | 0.4270 (18) | 0.1718 (7) | 0.0517 (4) | 0.069 (2) |
| H14 | 0.3557 | 0.1087 | 0.0646 | 0.083* |

supplementary materials

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| Br1 | 0.0795 (6) | 0.0510 (5) | 0.0758 (6) | 0.0131 (4) | 0.0027 (4) | 0.0117 (4) |
| Cl1 | 0.0979 (16) | 0.0525 (11) | 0.0767 (14) | -0.0047 (10) | -0.0060 (12) | -0.0098 (10) |
| N1 | 0.039 (3) | 0.066 (4) | 0.044 (3) | 0.000 (3) | 0.003 (3) | 0.003 (3) |
| O1 | 0.076 (3) | 0.047 (3) | 0.053 (3) | 0.000 (2) | 0.001 (2) | -0.002 (2) |
| C1 | 0.039 (4) | 0.056 (4) | 0.045 (4) | 0.002 (3) | 0.013 (3) | 0.009 (3) |
| C2 | 0.039 (3) | 0.046 (3) | 0.040 (4) | -0.001 (3) | 0.016 (3) | 0.001 (3) |
| C3 | 0.042 (4) | 0.042 (3) | 0.043 (4) | -0.001 (3) | 0.016 (3) | 0.001 (3) |
| C4 | 0.040 (4) | 0.049 (4) | 0.045 (4) | 0.004 (3) | 0.019 (3) | 0.008 (3) |
| C5 | 0.042 (4) | 0.047 (3) | 0.039 (4) | 0.002 (3) | 0.008 (3) | 0.001 (3) |
| C6 | 0.049 (4) | 0.048 (4) | 0.046 (4) | -0.001 (3) | 0.009 (3) | -0.003 (3) |
| C7 | 0.053 (4) | 0.042 (3) | 0.050 (4) | 0.005 (3) | 0.011 (3) | 0.000 (3) |
| C8 | 0.035 (4) | 0.078 (5) | 0.055 (5) | 0.000 (3) | -0.002 (3) | 0.001 (4) |
| C9 | 0.037 (4) | 0.068 (4) | 0.038 (4) | 0.002 (3) | -0.007 (3) | 0.002 (3) |
| C10 | 0.052 (5) | 0.082 (6) | 0.065 (5) | 0.000 (4) | -0.002 (4) | 0.007 (4) |
| C11 | 0.059 (5) | 0.115 (8) | 0.072 (6) | 0.000 (5) | -0.003 (5) | 0.028 (6) |
| C12 | 0.062 (6) | 0.144 (10) | 0.051 (6) | 0.013 (6) | 0.005 (4) | 0.006 (6) |
| C13 | 0.068 (6) | 0.113 (8) | 0.070 (6) | 0.022 (6) | -0.007 (5) | -0.028 (6) |
| C14 | 0.055 (5) | 0.081 (6) | 0.071 (6) | 0.004 (4) | -0.010 (4) | -0.010 (5) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|-----------|-----------|------------|------------|
| Br1—C4 | 1.890 (6) | C7—H7 | 0.93 |
| Cl1—C6 | 1.741 (7) | C8—C9 | 1.518 (10) |
| N1—C1 | 1.267 (9) | C8—H8A | 0.97 |
| N1—C8 | 1.473 (9) | C8—H8B | 0.97 |
| O1—C3 | 1.325 (8) | C9—C10 | 1.359 (10) |
| O1—H1 | 0.82 | C9—C14 | 1.386 (11) |
| C1—C2 | 1.465 (9) | C10—C11 | 1.392 (12) |
| C1—H1A | 0.93 | C10—H10 | 0.93 |
| C2—C7 | 1.387 (9) | C11—C12 | 1.360 (14) |
| C2—C3 | 1.422 (9) | C11—H11 | 0.93 |
| C3—C4 | 1.397 (9) | C12—C13 | 1.326 (14) |
| C4—C5 | 1.353 (9) | C12—H12 | 0.93 |
| C5—C6 | 1.416 (9) | C13—C14 | 1.377 (13) |
| C5—H5 | 0.93 | C13—H13 | 0.93 |
| C6—C7 | 1.355 (9) | C14—H14 | 0.93 |
| C1—N1—C8 | 119.5 (6) | N1—C8—H8A | 109.7 |
| C3—O1—H1 | 109.5 | C9—C8—H8A | 109.7 |
| N1—C1—C2 | 122.7 (6) | N1—C8—H8B | 109.7 |
| N1—C1—H1A | 118.7 | C9—C8—H8B | 109.7 |
| C2—C1—H1A | 118.7 | H8A—C8—H8B | 108.2 |
| C7—C2—C3 | 120.7 (6) | C10—C9—C14 | 119.8 (8) |
| C7—C2—C1 | 120.6 (6) | C10—C9—C8 | 119.9 (7) |
| C3—C2—C1 | 118.7 (6) | C14—C9—C8 | 120.3 (7) |

| | | | |
|--------------|------------|-----------------|------------|
| O1—C3—C4 | 120.0 (6) | C9—C10—C11 | 119.9 (9) |
| O1—C3—C2 | 123.2 (6) | C9—C10—H10 | 120.0 |
| C4—C3—C2 | 116.9 (6) | C11—C10—H10 | 120.0 |
| C5—C4—C3 | 122.7 (6) | C12—C11—C10 | 118.9 (10) |
| C5—C4—Br1 | 117.8 (5) | C12—C11—H11 | 120.6 |
| C3—C4—Br1 | 119.4 (5) | C10—C11—H11 | 120.6 |
| C4—C5—C6 | 118.9 (6) | C13—C12—C11 | 121.7 (10) |
| C4—C5—H5 | 120.6 | C13—C12—H12 | 119.1 |
| C6—C5—H5 | 120.6 | C11—C12—H12 | 119.1 |
| C7—C6—C5 | 120.7 (6) | C12—C13—C14 | 120.6 (10) |
| C7—C6—C11 | 120.7 (5) | C12—C13—H13 | 119.7 |
| C5—C6—C11 | 118.6 (5) | C14—C13—H13 | 119.7 |
| C6—C7—C2 | 120.1 (6) | C13—C14—C9 | 119.0 (9) |
| C6—C7—H7 | 120.0 | C13—C14—H14 | 120.5 |
| C2—C7—H7 | 120.0 | C9—C14—H14 | 120.5 |
| N1—C8—C9 | 109.6 (5) | | |
| C8—N1—C1—C2 | -178.6 (6) | C5—C6—C7—C2 | -2.7 (10) |
| N1—C1—C2—C7 | 175.2 (6) | C11—C6—C7—C2 | 179.2 (5) |
| N1—C1—C2—C3 | -5.7 (9) | C3—C2—C7—C6 | 0.7 (10) |
| C7—C2—C3—O1 | -178.8 (6) | C1—C2—C7—C6 | 179.8 (6) |
| C1—C2—C3—O1 | 2.1 (9) | C1—N1—C8—C9 | 102.8 (7) |
| C7—C2—C3—C4 | 1.2 (9) | N1—C8—C9—C10 | -94.4 (8) |
| C1—C2—C3—C4 | -177.9 (5) | N1—C8—C9—C14 | 85.3 (8) |
| O1—C3—C4—C5 | 178.9 (6) | C14—C9—C10—C11 | -0.7 (11) |
| C2—C3—C4—C5 | -1.1 (9) | C8—C9—C10—C11 | 179.0 (6) |
| O1—C3—C4—Br1 | -3.9 (8) | C9—C10—C11—C12 | 0.4 (12) |
| C2—C3—C4—Br1 | 176.1 (4) | C10—C11—C12—C13 | -0.6 (14) |
| C3—C4—C5—C6 | -0.8 (9) | C11—C12—C13—C14 | 1.0 (14) |
| Br1—C4—C5—C6 | -178.1 (5) | C12—C13—C14—C9 | -1.2 (13) |
| C4—C5—C6—C7 | 2.7 (10) | C10—C9—C14—C13 | 1.1 (11) |
| C4—C5—C6—C11 | -179.1 (5) | C8—C9—C14—C13 | -178.6 (7) |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|-------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| O1—H1 \cdots N1 | 0.82 | 1.86 | 2.590 (7) | 147 |

Fig. 1

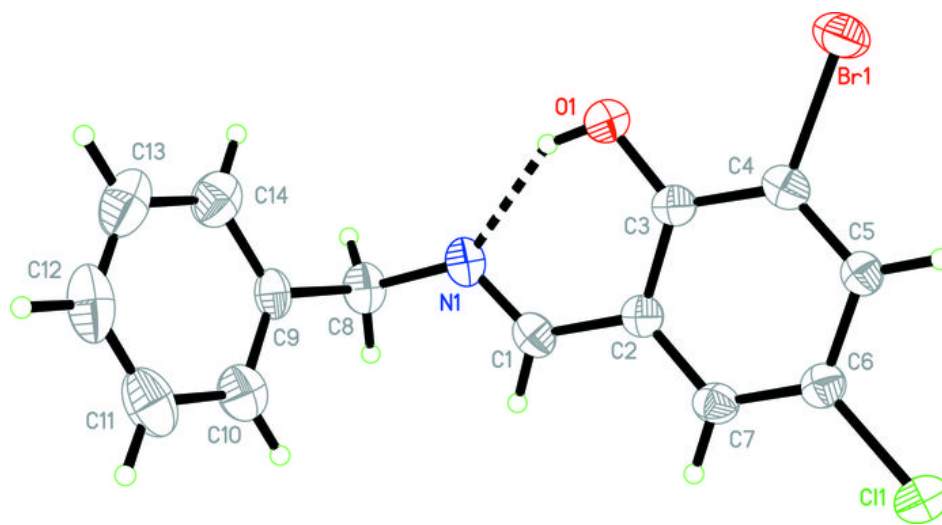


Fig. 2

