Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Bromo-4-chloro-6-[(*E*)-*o*-tolylimino-methyl]phenol

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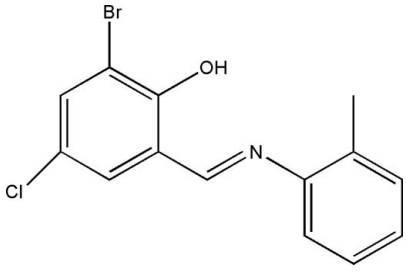
Received 20 September 2010; accepted 22 September 2010

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.032; wR factor = 0.057; data-to-parameter ratio = 13.9.

The title compound, $C_{14}H_{11}BrClNO$, is a Schiff base compound derived from the condensation of 3-bromo-5-chlorosalicylaldehyde and *o*-toluidine in methanol. The aromatic rings make a dihedral angle of 38.3 (1)°. The molecular conformation is stabilized by an intramolecular O—H···N hydrogen bond, generating an S(6) ring.

Related literature

For Schiff bases, see: Ali *et al.* (2002). For related structures, see: Li & Zhang (2005, 2006); Li *et al.* (2006).



Experimental

Crystal data

$C_{14}H_{11}BrClNO$
M_r = 324.60
Orthorhombic, $P2_12_12_1$
a = 7.5388 (9) Å
b = 12.2452 (11) Å
c = 14.2440 (16) Å
V = 1314.9 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.32 mm^{-1}
T = 298 K
0.40 × 0.38 × 0.33 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)
T_{min} = 0.351, T_{max} = 0.408
5422 measured reflections
2284 independent reflections
1815 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.057
S = 1.09
2284 reflections
164 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}
Absolute structure: Flack (1983), 938 Friedel pairs
Flack parameter: 0.006 (10)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.82	1.86	2.588 (3)	147

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); 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*.

This research was supported by a research grant (No. 09JS068) from the Phytochemistry Key Laboratory of Shaanxi Province.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5360).

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supporting information

Acta Cryst. (2010). E**66**, o2689 [doi:10.1107/S1600536810037931]

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S1. Comment

The roles of Schiff base compounds in biological processes have become a topic of study in recent years. Schiff base compounds have demonstrated significant biological activity and new examples are being tested for their antitumor, antimicrobial, and antiviral activity (Ali *et al.*, 2002). In the past years, we have prepared a series of Schiff base cmpounds, and investigated their structure and properties (Li *et al.*, 2006; Li & Zhang, 2006; Li & Zhang, 2005).

In the title compound (Fig. 1), all the bond lengths and angles are within normal value and are comparable to those observed in a similar Schiff base compound (Li & Zhang, 2005). The two aromatic rings are linked by a C ═N bond and enclose a dihedral angle of 38.3 (1) °. As expected, the molecule adopts a *trans* configuration about the C1 ═N1 bond. The molecular conformation is stabilized by an intramolecular O—H–N hydrogen bond (Table 1).

S2. Experimental

3-bromo-5-Chlorosalicylaldehyde (0.1 mmol, 23.5 mg) and *o*-toluidine (0.1 mmol, 10.7 mg) dissolved in MeOH (10 ml). The solution was stirred for half an hour and then filtered. Crystals of the title compound suitable for single-crystal X-ray analysis were recrystallized from methanol after one weeks at room temperature. The yellow block precipitate was filtered, washed with cold MeOH, and dried *in vacuo* for 48 h (yield 70%).

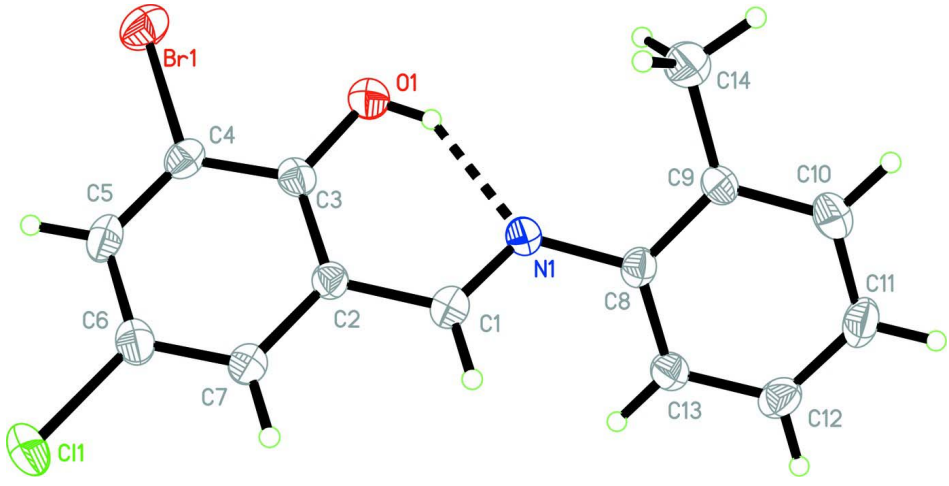

Figure 1

The molecular structure of the title compound, showing 50% probability displacement ellipsoids. The dashed line indicates a hydrogen bond.

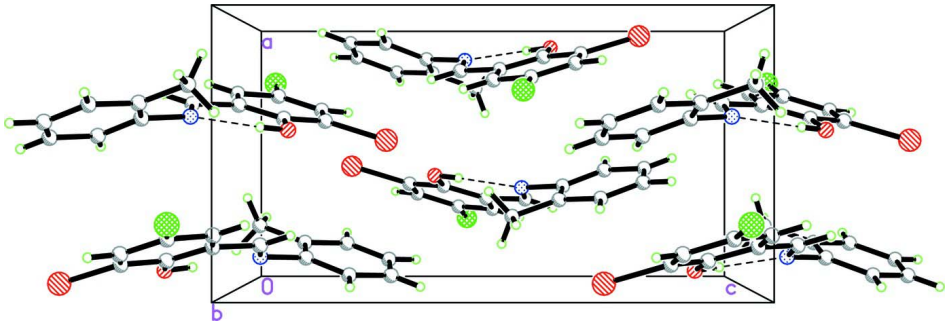

Figure 2

The crystal packing of the title compound, viewed along the *b* axis.

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$C_{14}H_{11}BrClNO$
$M_r = 324.60$
Orthorhombic, $P2_12_12_1$
$a = 7.5388\ (9)$ Å
$b = 12.2452\ (11)$ Å
$c = 14.2440\ (16)$ Å
$V = 1314.9\ (2)$ Å^3
$Z = 4$
$F(000) = 648$
$D_x = 1.640$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2013 reflections
$\theta = 2.2$–$23.7°$
$\mu = 3.32$ mm^{-1}
$T = 298$ K
Block, yellow
0.40 × 0.38 × 0.33 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)
$T_{min} = 0.351$, $T_{max} = 0.408$
5422 measured reflections
2284 independent reflections
1815 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.2°$
$h = -8\rightarrow8$
$k = -14\rightarrow14$
$l = -16\rightarrow8$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.057$
$S = 1.09$
2284 reflections
164 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0039P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.33$ e Å^{-3}
Absolute structure: Flack (1983), 938 Friedel pairs
Absolute structure parameter: 0.006 (10)

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.04915 (6)	0.84654 (3)	−0.29615 (2)	0.06176 (15)
Cl1	0.26999 (17)	1.17472 (7)	−0.05785 (7)	0.0692 (3)
N1	0.1302 (3)	0.6835 (2)	0.05868 (17)	0.0362 (7)
O1	0.0835 (3)	0.71558 (18)	−0.11917 (15)	0.0500 (7)
H1	0.0929	0.6804	−0.0704	0.075*
C1	0.1777 (5)	0.7832 (3)	0.0667 (2)	0.0388 (9)
H1A	0.2136	0.8093	0.1250	0.047*
C2	0.1770 (4)	0.8568 (3)	−0.0137 (2)	0.0339 (8)
C3	0.1263 (4)	0.8203 (3)	−0.1027 (2)	0.0376 (9)
C4	0.1196 (5)	0.8952 (3)	−0.1764 (2)	0.0400 (9)
C5	0.1624 (5)	1.0029 (3)	−0.1627 (2)	0.0450 (10)
H5	0.1554	1.0522	−0.2122	0.054*
C6	0.2160 (5)	1.0382 (3)	−0.0746 (2)	0.0447 (10)
C7	0.2229 (5)	0.9665 (3)	−0.0010 (2)	0.0408 (9)
H7	0.2583	0.9909	0.0579	0.049*
C8	0.1220 (5)	0.6153 (3)	0.1403 (2)	0.0364 (9)
C9	0.1705 (5)	0.5059 (3)	0.1305 (2)	0.0360 (9)
C10	0.1639 (5)	0.4404 (3)	0.2096 (3)	0.0477 (9)
H10	0.1993	0.3678	0.2053	0.057*
C11	0.1059 (5)	0.4805 (3)	0.2947 (3)	0.0516 (11)
H11	0.1014	0.4348	0.3467	0.062*
C12	0.0553 (5)	0.5866 (3)	0.3027 (2)	0.0478 (9)
H12	0.0163	0.6133	0.3602	0.057*
C13	0.0618 (5)	0.6549 (3)	0.22552 (19)	0.0415 (8)
H13	0.0259	0.7273	0.2309	0.050*
C14	0.2313 (6)	0.4607 (3)	0.0376 (2)	0.0600 (12)
H14A	0.2589	0.3845	0.0445	0.090*
H14B	0.3352	0.4993	0.0171	0.090*
H14C	0.1387	0.4692	−0.0081	0.090*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0701 (3)	0.0765 (3)	0.03869 (18)	0.0032 (3)	−0.0065 (2)	0.0004 (2)
Cl1	0.1052 (9)	0.0331 (5)	0.0692 (6)	−0.0064 (6)	−0.0041 (6)	0.0075 (5)
N1	0.0390 (18)	0.0323 (16)	0.0372 (15)	−0.0012 (14)	0.0006 (13)	0.0021 (14)

O1	0.064 (2)	0.0434 (14)	0.0423 (13)	−0.0024 (13)	−0.0035 (13)	−0.0011 (11)
C1	0.034 (2)	0.045 (2)	0.038 (2)	0.0001 (18)	0.0003 (18)	0.0037 (18)
C2	0.035 (2)	0.033 (2)	0.0335 (17)	0.0014 (19)	0.0021 (15)	0.0014 (18)
C3	0.033 (2)	0.034 (2)	0.046 (2)	0.0048 (17)	0.0093 (16)	−0.0010 (18)
C4	0.036 (2)	0.050 (2)	0.034 (2)	0.0074 (18)	0.0019 (16)	0.0027 (17)
C5	0.045 (3)	0.050 (2)	0.040 (2)	0.009 (2)	0.0088 (19)	0.0144 (19)
C6	0.045 (3)	0.038 (2)	0.050 (2)	0.001 (2)	0.0066 (19)	0.006 (2)
C7	0.045 (3)	0.041 (2)	0.0354 (19)	−0.002 (2)	0.0011 (17)	0.0011 (18)
C8	0.038 (2)	0.036 (2)	0.0354 (19)	−0.0042 (16)	−0.0018 (16)	0.0049 (16)
C9	0.039 (2)	0.030 (2)	0.0396 (19)	−0.0054 (18)	−0.0004 (18)	0.0001 (17)
C10	0.052 (3)	0.033 (2)	0.058 (2)	−0.0044 (18)	−0.001 (2)	0.004 (2)
C11	0.063 (3)	0.051 (3)	0.041 (2)	−0.010 (2)	−0.006 (2)	0.017 (2)
C12	0.054 (3)	0.053 (2)	0.0363 (19)	−0.012 (2)	0.005 (2)	0.0002 (19)
C13	0.049 (2)	0.0355 (19)	0.0402 (18)	0.002 (2)	0.0055 (17)	−0.0007 (17)
C14	0.075 (3)	0.050 (2)	0.055 (2)	0.000 (3)	0.015 (2)	0.000 (2)

Geometric parameters (Å, º)

Br1—C4	1.883 (3)	C7—H7	0.9300
Cl1—C6	1.737 (4)	C8—C13	1.383 (4)
N1—C1	1.277 (4)	C8—C9	1.395 (4)
N1—C8	1.433 (4)	C9—C10	1.385 (4)
O1—C3	1.343 (4)	C9—C14	1.506 (5)
O1—H1	0.8200	C10—C11	1.378 (5)
C1—C2	1.458 (4)	C10—H10	0.9300
C1—H1A	0.9300	C11—C12	1.359 (5)
C2—C7	1.398 (5)	C11—H11	0.9300
C2—C3	1.398 (4)	C12—C13	1.382 (4)
C3—C4	1.394 (4)	C12—H12	0.9300
C4—C5	1.373 (5)	C13—H13	0.9300
C5—C6	1.386 (4)	C14—H14A	0.9600
C5—H5	0.9300	C14—H14B	0.9600
C6—C7	1.368 (4)	C14—H14C	0.9600
C1—N1—C8	119.8 (3)	C13—C8—N1	121.5 (3)
C3—O1—H1	109.5	C9—C8—N1	117.8 (3)
N1—C1—C2	121.3 (3)	C10—C9—C8	117.7 (3)
N1—C1—H1A	119.3	C10—C9—C14	120.8 (3)
C2—C1—H1A	119.3	C8—C9—C14	121.4 (3)
C7—C2—C3	119.4 (3)	C11—C10—C9	121.3 (3)
C7—C2—C1	119.5 (3)	C11—C10—H10	119.3
C3—C2—C1	121.1 (3)	C9—C10—H10	119.3
O1—C3—C4	119.2 (3)	C12—C11—C10	120.2 (3)
O1—C3—C2	121.9 (3)	C12—C11—H11	119.9
C4—C3—C2	118.9 (3)	C10—C11—H11	119.9
C5—C4—C3	121.0 (3)	C11—C12—C13	120.1 (3)
C5—C4—Br1	120.0 (3)	C11—C12—H12	119.9
C3—C4—Br1	119.0 (3)	C13—C12—H12	119.9

C4—C5—C6	119.8 (3)	C12—C13—C8	119.8 (3)
C4—C5—H5	120.1	C12—C13—H13	120.1
C6—C5—H5	120.1	C8—C13—H13	120.1
C7—C6—C5	120.3 (3)	C9—C14—H14A	109.5
C7—C6—Cl1	120.2 (3)	C9—C14—H14B	109.5
C5—C6—Cl1	119.5 (3)	H14A—C14—H14B	109.5
C6—C7—C2	120.5 (3)	C9—C14—H14C	109.5
C6—C7—H7	119.7	H14A—C14—H14C	109.5
C2—C7—H7	119.7	H14B—C14—H14C	109.5
C13—C8—C9	120.7 (3)		

C8—N1—C1—C2	−176.1 (3)	Cl1—C6—C7—C2	179.0 (3)
N1—C1—C2—C7	177.3 (3)	C3—C2—C7—C6	0.9 (5)
N1—C1—C2—C3	−0.9 (5)	C1—C2—C7—C6	−177.3 (3)
C7—C2—C3—O1	179.0 (3)	C1—N1—C8—C13	38.4 (5)
C1—C2—C3—O1	−2.8 (5)	C1—N1—C8—C9	−144.0 (3)
C7—C2—C3—C4	−1.3 (5)	C13—C8—C9—C10	−2.8 (5)
C1—C2—C3—C4	176.9 (3)	N1—C8—C9—C10	179.5 (3)
O1—C3—C4—C5	−180.0 (3)	C13—C8—C9—C14	178.3 (3)
C2—C3—C4—C5	0.3 (5)	N1—C8—C9—C14	0.6 (5)
O1—C3—C4—Br1	0.4 (4)	C8—C9—C10—C11	2.1 (5)
C2—C3—C4—Br1	−179.4 (3)	C14—C9—C10—C11	−179.0 (3)
C3—C4—C5—C6	1.1 (5)	C9—C10—C11—C12	−0.7 (6)
Br1—C4—C5—C6	−179.3 (3)	C10—C11—C12—C13	0.0 (6)
C4—C5—C6—C7	−1.4 (6)	C11—C12—C13—C8	−0.8 (6)
C4—C5—C6—Cl1	180.0 (3)	C9—C8—C13—C12	2.2 (6)
C5—C6—C7—C2	0.4 (5)	N1—C8—C13—C12	179.8 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.82	1.86	2.588 (3)	147