Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Bromo-4-chloro-6-[(*E*)-*p*-tolyliminomethyl]phenol

Xinli Zhang

Department of Chemistry, Baoji University of Arts and Science, Baoji, Shaanxi 721007, People's Republic of China
Correspondence e-mail: zhangxinli6008@163.com

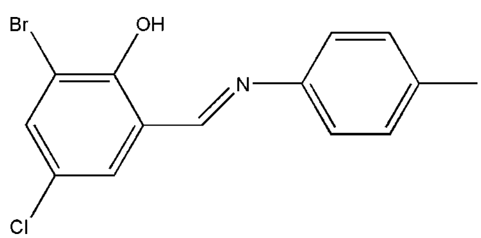
Received 1 January 2009; accepted 2 February 2009

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.007 Å; R factor = 0.045; wR factor = 0.129; data-to-parameter ratio = 14.3.

The molecule of the title compound, $C_{14}H_{11}BrClNO$, displays an E configuration with respect to the imine C═N double bond. The two aromatic rings are essentially coplanar, forming a dihedral angle of 7.9 (2)°. An intramolecular O—H···N hydrogen bond stabilizes the crystal structure.

Related literature

For the role of Schiff base ligands in catalysis and electron transfer in living organisms, see: Ueno *et al.* (2006).



Experimental

Crystal data

$C_{14}H_{11}BrClNO$
M_r = 324.60
Triclinic, $P\overline{1}$
a = 8.1354 (14) Å
b = 8.6844 (17) Å
c = 11.3740 (18) Å
α = 76.040 (2)°
β = 73.652 (12)°
γ = 62.458 (12)°
V = 677.9 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 3.22 mm^{-1}
T = 298 (2) K
0.43 × 0.18 × 0.09 mm

Data collection

Siemens SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Siemens, 1996)
T_{min} = 0.332, T_{max} = 0.745
3500 measured reflections
2351 independent reflections
1412 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.129
S = 1.00
2351 reflections
164 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.82	1.84	2.574 (4)	148

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); 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*.

The author gratefully acknowledges support from a research project (No. 08JZ09) of the Phytochemistry Key Laboratory of Shaanxi Province.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2287).

References

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Siemens (1996). *SMART*, *SAINT* and *SADABS*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Ueno, T., Yokoi, N., Unno, M., Matsui, T., Tokita, Y., Yamada, M., Ikeda-Saito, M., Nakajima, H. & Watanabe, Y. (2006). *PNAS*, **103**, 9416–9421.

supporting information

Acta Cryst. (2009). E**65**, o513   [doi:10.1107/S1600536809003912]

2-Bromo-4-chloro-6-[(*E*)-*p*-tolyliminomethyl]phenol

Xinli Zhang

S1. Comment

Recently, there has been a growing interest in Schiff base ligands because of their applications, such as catalysts and non-linear optical materials. In recent years, they were found to play an important role in the catalysis and electron transfer of the living organisms (Ueno *et al.*, 2006). This stimulated our interest in this field. As an extension of the work on the structural characterization of Schiff base compounds, the crystal structure of the title compound is reported here.

The molecular structure and crystal packing of the title compound are illustrated in Figure 1 and 2, respectively. Bond lengths and angles are not unusual, with the C1═N1 bond distance (1.263 (5) Å) slightly shorter than a normal C═N. The molecule is essentially planar, the maximum deviation from the planarity being 0.167 (6) Å for atom C10. The dihedral angle between the two aromatic rings is 7.9 (2) °. An intramolecular O—H···N hydrogen bond (Table 1) stabilizes the crystal structure.

S2. Experimental

3-Bromo-5-chlorosalicylaldehyde (0.1 mmol, 23.6 mg) and *p*-toluidine (0.1 mmol, 10.7 mg) were dissolved in methanol (10 ml). The mixture was stirred at room temperature for 10 min and then filtered. After allowing the filtrate to stand in air for 3 d, yellow block-shaped crystals of the title compound suitable for X-ray analysis were formed by slow evaporation of the solvent. The crystals were collected, washed with methanol and dried in a vacuum desiccator using anhydrous $CaCl_2$ (yield 54%).

S3. Refinement

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.93-0.96 Å, O—H = 0.82 Å, and with U_{iso}(H) = 1.2 U_{eq}(C) or 1.5 U_{eq}(C, O) for methyl and hydroxy H atoms.

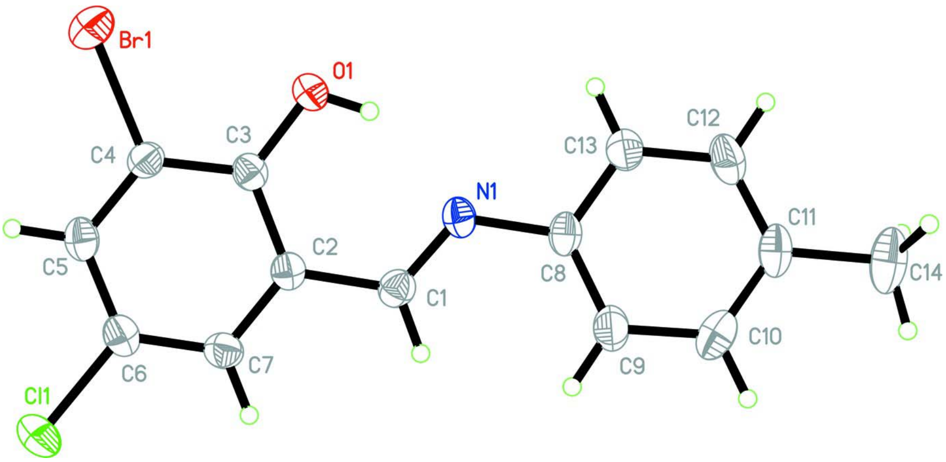


Figure 1

The molecular structure of the title compound with 30% probability ellipsoids. H atoms are shown as spheres of arbitrary radii. The dashed line represents a hydrogen bond.

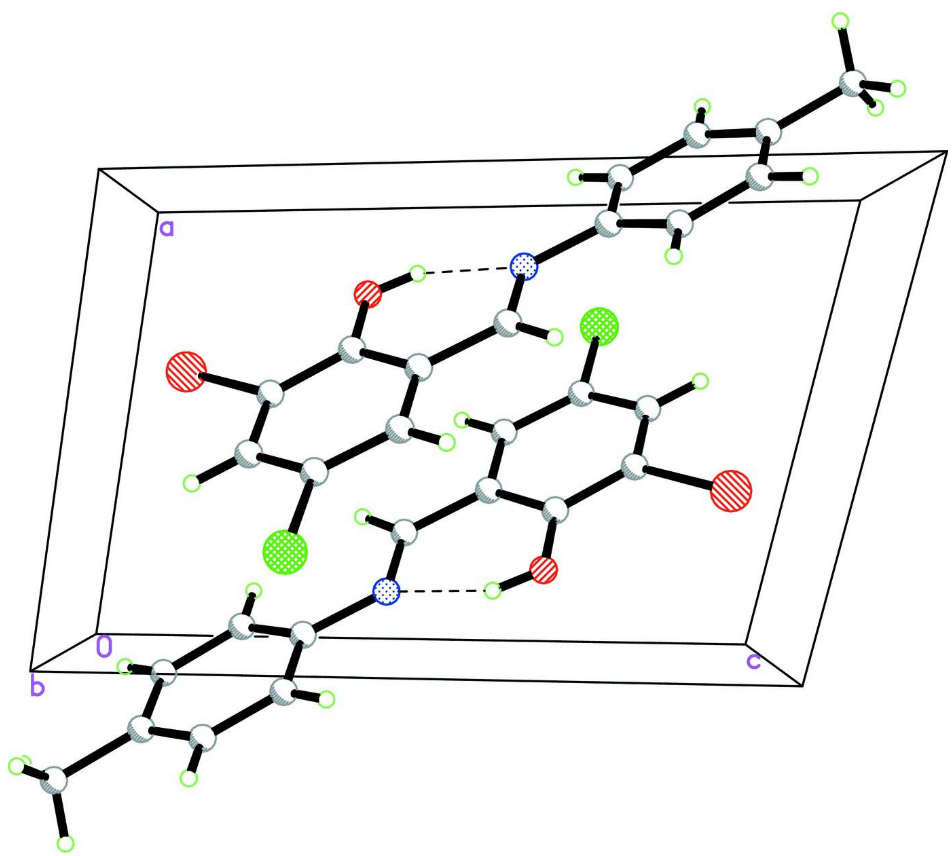


Figure 2

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

2-Bromo-4-chloro-6-[(*E*)-*p*-tolyliminomethyl]phenol

Crystal data

$C_{14}H_{11}BrClNO$
M_r = 324.60
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 8.1354 (14) Å
b = 8.6844 (17) Å
c = 11.3740 (18) Å
α = 76.040 (2)°
β = 73.652 (12)°
γ = 62.458 (12)°
V = 677.9 (2) Å^3
Z = 2
F(000) = 324
D_x = 1.590 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1148 reflections
θ = 2.7–24.9°
μ = 3.22 mm^{-1}
T = 298 K
Block-shaped, yellow
0.43 × 0.18 × 0.09 mm

Data collection

Siemens SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Siemens, 1996)
T_{min} = 0.332, T_{max} = 0.745
3500 measured reflections
2351 independent reflections
1412 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 25.0°, θ_{min} = 1.9°
h = −9→9
k = −6→10
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.129
S = 1.00
2351 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
$w = 1/[\sigma^2(F_o^2) + (0.065P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 e Å^{-3}

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Br1	0.62666 (9)	0.48664 (8)	0.11313 (5)	0.0881 (3)
Cl1	0.2620 (2)	1.09756 (16)	0.31821 (13)	0.0714 (4)
O1	0.7924 (4)	0.3591 (4)	0.3427 (3)	0.0554 (9)

H1	0.8367	0.3272	0.4054	0.083*
N1	0.8449 (5)	0.3835 (5)	0.5491 (3)	0.0414 (9)
C1	0.7224 (6)	0.5406 (6)	0.5417 (4)	0.0434 (11)
H1A	0.6902	0.6050	0.6058	0.052*
C2	0.6298 (6)	0.6243 (6)	0.4356 (4)	0.0381 (10)
C3	0.6708 (6)	0.5270 (6)	0.3398 (4)	0.0387 (10)
C4	0.5770 (6)	0.6130 (6)	0.2422 (4)	0.0439 (11)
C5	0.4522 (6)	0.7843 (6)	0.2351 (4)	0.0457 (11)
H5	0.3916	0.8380	0.1683	0.055*
C6	0.4168 (6)	0.8776 (6)	0.3294 (4)	0.0467 (11)
C7	0.5034 (6)	0.7974 (6)	0.4289 (4)	0.0476 (11)
H7	0.4763	0.8608	0.4925	0.057*
C8	0.9370 (6)	0.3016 (6)	0.6518 (4)	0.0416 (11)
C9	0.9271 (7)	0.3898 (7)	0.7423 (4)	0.0553 (13)
H9	0.8552	0.5102	0.7394	0.066*
C10	1.0267 (7)	0.2956 (8)	0.8377 (4)	0.0619 (14)
H10	1.0201	0.3549	0.8981	0.074*
C11	1.1339 (7)	0.1177 (7)	0.8446 (4)	0.0541 (13)
C12	1.1407 (7)	0.0343 (7)	0.7539 (4)	0.0604 (14)
H12	1.2122	−0.0862	0.7571	0.072*
C13	1.0454 (6)	0.1229 (6)	0.6584 (4)	0.0522 (12)
H13	1.0540	0.0623	0.5981	0.063*
C14	1.2405 (8)	0.0205 (8)	0.9481 (4)	0.0813 (18)
H14A	1.2169	0.1011	1.0021	0.122*
H14B	1.3735	−0.0336	0.9144	0.122*
H14C	1.1990	−0.0679	0.9938	0.122*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.1111 (6)	0.0784 (5)	0.0686 (4)	−0.0123 (4)	−0.0436 (4)	−0.0269 (3)
Cl1	0.0773 (9)	0.0392 (7)	0.0848 (9)	−0.0057 (7)	−0.0321 (8)	−0.0064 (6)
O1	0.058 (2)	0.0406 (19)	0.0586 (19)	−0.0026 (17)	−0.0275 (16)	−0.0109 (15)
N1	0.037 (2)	0.042 (2)	0.046 (2)	−0.0165 (19)	−0.0143 (17)	0.0014 (17)
C1	0.047 (3)	0.046 (3)	0.042 (2)	−0.023 (3)	−0.010 (2)	−0.005 (2)
C2	0.036 (2)	0.041 (3)	0.043 (2)	−0.021 (2)	−0.010 (2)	−0.002 (2)
C3	0.033 (2)	0.039 (3)	0.046 (2)	−0.015 (2)	−0.011 (2)	−0.004 (2)
C4	0.043 (3)	0.047 (3)	0.042 (2)	−0.015 (2)	−0.011 (2)	−0.011 (2)
C5	0.042 (3)	0.048 (3)	0.048 (3)	−0.020 (2)	−0.017 (2)	0.004 (2)
C6	0.045 (3)	0.042 (3)	0.053 (3)	−0.020 (2)	−0.015 (2)	0.002 (2)
C7	0.051 (3)	0.040 (3)	0.055 (3)	−0.018 (2)	−0.015 (2)	−0.009 (2)
C8	0.034 (2)	0.052 (3)	0.041 (2)	−0.023 (2)	−0.012 (2)	0.004 (2)
C9	0.056 (3)	0.053 (3)	0.055 (3)	−0.019 (3)	−0.024 (2)	0.002 (2)
C10	0.062 (3)	0.087 (4)	0.052 (3)	−0.040 (3)	−0.019 (3)	−0.008 (3)
C11	0.049 (3)	0.068 (4)	0.047 (3)	−0.031 (3)	−0.020 (2)	0.016 (3)
C12	0.061 (3)	0.047 (3)	0.069 (3)	−0.021 (3)	−0.029 (3)	0.016 (3)
C13	0.053 (3)	0.048 (3)	0.055 (3)	−0.018 (3)	−0.019 (2)	−0.003 (2)
C14	0.080 (4)	0.110 (5)	0.061 (3)	−0.052 (4)	−0.037 (3)	0.028 (3)

Geometric parameters (Å, º)

Br1—C4	1.885 (4)	C7—H7	0.9300
Cl1—C6	1.732 (5)	C8—C13	1.381 (6)
O1—C3	1.329 (5)	C8—C9	1.389 (6)
O1—H1	0.8200	C9—C10	1.399 (6)
N1—C1	1.263 (5)	C9—H9	0.9300
N1—C8	1.425 (5)	C10—C11	1.374 (7)
C1—C2	1.462 (5)	C10—H10	0.9300
C1—H1A	0.9300	C11—C12	1.372 (7)
C2—C7	1.372 (6)	C11—C14	1.506 (6)
C2—C3	1.411 (5)	C12—C13	1.376 (6)
C3—C4	1.387 (5)	C12—H12	0.9300
C4—C5	1.357 (6)	C13—H13	0.9300
C5—C6	1.386 (6)	C14—H14A	0.9600
C5—H5	0.9300	C14—H14B	0.9600
C6—C7	1.370 (6)	C14—H14C	0.9600
C3—O1—H1	109.5	C13—C8—N1	116.6 (4)
C1—N1—C8	122.3 (4)	C9—C8—N1	124.4 (4)
N1—C1—C2	121.9 (4)	C8—C9—C10	119.3 (5)
N1—C1—H1A	119.0	C8—C9—H9	120.4
C2—C1—H1A	119.0	C10—C9—H9	120.4
C7—C2—C3	120.0 (4)	C11—C10—C9	121.8 (5)
C7—C2—C1	120.0 (4)	C11—C10—H10	119.1
C3—C2—C1	119.9 (4)	C9—C10—H10	119.1
O1—C3—C4	120.8 (4)	C12—C11—C10	117.6 (4)
O1—C3—C2	121.9 (4)	C12—C11—C14	122.0 (5)
C4—C3—C2	117.3 (4)	C10—C11—C14	120.4 (5)
C5—C4—C3	122.8 (4)	C11—C12—C13	122.2 (5)
C5—C4—Br1	118.7 (3)	C11—C12—H12	118.9
C3—C4—Br1	118.5 (3)	C13—C12—H12	118.9
C4—C5—C6	118.7 (4)	C12—C13—C8	120.2 (5)
C4—C5—H5	120.7	C12—C13—H13	119.9
C6—C5—H5	120.7	C8—C13—H13	119.9
C7—C6—C5	120.5 (4)	C11—C14—H14A	109.5
C7—C6—Cl1	120.9 (4)	C11—C14—H14B	109.5
C5—C6—Cl1	118.5 (3)	H14A—C14—H14B	109.5
C6—C7—C2	120.6 (4)	C11—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	119.0 (4)		
C8—N1—C1—C2	179.4 (4)	Cl1—C6—C7—C2	−178.2 (3)
N1—C1—C2—C7	−177.3 (4)	C3—C2—C7—C6	−0.4 (6)
N1—C1—C2—C3	2.6 (6)	C1—C2—C7—C6	179.5 (4)
C7—C2—C3—O1	179.9 (4)	C1—N1—C8—C13	170.2 (4)
C1—C2—C3—O1	0.1 (6)	C1—N1—C8—C9	−11.1 (6)

C7—C2—C3—C4	−0.8 (6)	C13—C8—C9—C10	−0.2 (7)
C1—C2—C3—C4	179.3 (4)	N1—C8—C9—C10	−178.8 (4)
O1—C3—C4—C5	−179.8 (4)	C8—C9—C10—C11	−0.1 (7)
C2—C3—C4—C5	1.0 (6)	C9—C10—C11—C12	0.1 (7)
O1—C3—C4—Br1	−0.2 (6)	C9—C10—C11—C14	179.5 (4)
C2—C3—C4—Br1	−179.5 (3)	C10—C11—C12—C13	0.2 (7)
C3—C4—C5—C6	0.1 (7)	C14—C11—C12—C13	−179.2 (4)
Br1—C4—C5—C6	−179.5 (3)	C11—C12—C13—C8	−0.5 (7)
C4—C5—C6—C7	−1.3 (6)	C9—C8—C13—C12	0.5 (7)
C4—C5—C6—Cl1	178.4 (3)	N1—C8—C13—C12	179.2 (4)
C5—C6—C7—C2	1.4 (7)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.82	1.84	2.574 (4)	148