Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

2-Bromo-4-chloro-6-(cyclopentyliminomethyl)phenol

Yu-Mei Hao

Department of Chemistry, Baicheng Normal University, Baicheng 137000, People's Republic of China
Correspondence e-mail: jyxygzb@163.com

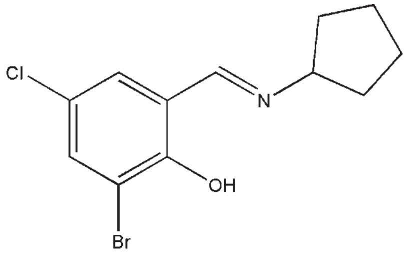
Received 24 September 2009; accepted 26 September 2009

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.007 Å; R factor = 0.044; wR factor = 0.122; data-to-parameter ratio = 16.0.

All atoms of the title molecule, $C_{12}H_{13}BrClNO$, except the C and H atoms of the cyclopentane methylene groups lie on a crystallographic mirror plane. The cyclopentane ring adopts an envelope conformation and an intramolecular O—H···N hydrogen bond is observed. In the crystal, molecules are stacked along the b axis by π–π interactions [centroid–centroid distance = 3.6424 (11) Å].

Related literature

For the pharmaceutical and medicinal activity of Schiff bases, see: Dao *et al.* (2000); Sriram *et al.* (2006); Karthikeyan *et al.* (2006). For the coordination chemistry of Schiff bases, see: Ali *et al.* (2008); Kargar *et al.* (2009); Yeap *et al.* (2009). For the crystal structures of Schiff base compounds, see: Fun *et al.* (2009); Nadeem *et al.* (2009); Eltayeb *et al.* (2008). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{12}H_{13}BrClNO$	a = 12.142 (2) Å
M_r = 302.59	b = 6.8610 (14) Å
Orthorhombic, *Pnma*	c = 15.077 (3) Å
V = 1256.0 (4) Å^3	μ = 3.46 mm^{-1}
Z = 4	T = 298 K
Mo $K\alpha$ radiation	0.20 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer	10340 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	1488 independent reflections
T_{min} = 0.544, T_{max} = 0.574	1132 reflections with $I > 2\sigma(I)$
	R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.122	$\Delta\rho_{max}$ = 0.40 e Å^{-3}
S = 1.08	$\Delta\rho_{min}$ = −0.89 e Å^{-3}
1488 reflections	
93 parameters	
6 restraints	

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.89 (6)	1.71 (6)	2.577 (5)	162 (5)

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); 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: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2924).

References

Ali, H. M., Mohamed Mustafa, M. I., Rizal, M. R. & Ng, S. W. (2008). *Acta Cryst.* E**64**, m718–m719.
Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (2002). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Dao, V.-T., Gaspard, C., Mayer, M., Werner, G. H., Nguyen, S. N. & Michelot, R. J. (2000). *Eur. J. Med. Chem.* **35**, 805–813.
Eltayeb, N. E., Teoh, S. G., Chantrapromma, S., Fun, H.-K. & Adnan, R. (2008). *Acta Cryst.* E**64**, o576–o577.
Fun, H.-K., Kia, R., Vijesh, A. M. & Isloor, A. M. (2009). *Acta Cryst.* E**65**, o349–o350.
Kargar, H., Jamshidvand, A., Fun, H.-K. & Kia, R. (2009). *Acta Cryst.* E**65**, m403–m404.
Karthikeyan, M. S., Prasad, D. J., Poojary, B., Bhat, K. S., Holla, B. S. & Kumari, N. S. (2006). *Bioorg. Med. Chem.* **14**, 7482–7489.
Nadeem, S., Shah, M. R. & VanDerveer, D. (2009). *Acta Cryst.* E**65**, o897.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sriram, D., Yogeeswari, P., Myneedu, N. S. & Saraswat, V. (2006). *Bioorg. Med. Chem. Lett.* **16**, 2127–2129.
Yeap, C. S., Kia, R., Kargar, H. & Fun, H.-K. (2009). *Acta Cryst.* E**65**, m570–m571.

supplementary materials

Acta Cryst. (2009). E**65**, o2600 [**doi:10.1107/S1600536809039142**]

2-Bromo-4-chloro-6-(cyclopentyliminomethyl)phenol

Y.-M. Hao

Comment

Schiff base compounds are a class of important materials used in pharmaceutical and medicinal appications (Dao *et al.*, 2000; Sriram *et al.*, 2006; Karthikeyan *et al.*, 2006). Schiff bases have also been used as versatile ligands in coordination chemistry (Ali *et al.*, 2008; Kargar *et al.*, 2009; Yeap *et al.*, 2009). Recently, crystal structures of a large number of Schiff base compounds have been reported (Fun *et al.*, 2009; Nadeem *et al.*, 2009; Eltayeb *et al.*, 2008). In this paper, the title new Schiff base compound (Fig. 1) is reported.

All atoms of the title molecule, except the C and H atoms of the four methylene groups lie on a crystallographic mirror plane. The cyclopentane ring adopts a an envelope conformation. An intramolecular O—H···N hydrogen bond (Table 1) is observed. All bond lengths are within normal values (Allen *et al.*, 1987).

In the crystal, molecules are stacked along the *b* axis with π-π interactions [centroid to centroid distance = 3.6424 (11) Å].

Experimental

3-Bromo-5-chlorosalicylaldehyde (0.1 mmol, 23.5 mg) and cyclopentylamine (0.1 mmol, 8.5 mg) were refluxed in a 30 ml methanol solution for 30 min to give a clear orange solution. Yellow block-shaped single crystals of the title compound were formed by slow evaporation of the solvent over several days at room temperature.

Refinement

Atom H1 was located from a difference map and its positional parameters were refined. The remaining H atoms were constrained to ideal geometries, with C-H = 0.93–0.98 Å. The U_{iso}(H) values were set at $1.2U_{eq}$(C) and $1.5U_{eq}$(O). The U^{ij} components of atom C10 were restrained to an approximate isotropic behaviour.

Figures

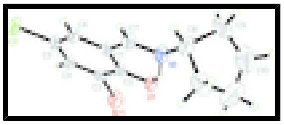


Fig. 1. The molecular structure of the title compound, with 30% probability displacement ellipsoids. The dashed line indicates an intramolecular O—H···N hydrogen bond.

2-Bromo-4-chloro-6-(cyclopentyliminomethyl)phenol

Crystal data

$C_{12}H_{13}BrClNO$	$F_{000} = 608$
$M_r = 302.59$	$D_x = 1.600$ Mg m^{-3}

Orthorhombic, *Pnma*

Hall symbol: -P 2ac 2n

$a = 12.142$ (2) Å

$b = 6.8610$ (14) Å

$c = 15.077$ (3) Å

$V = 1256.0$ (4) Å^3

$Z = 4$

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2255 reflections

$\theta = 2.6$–24.5°

$\mu = 3.46$ mm^{-1}

$T = 298$ K

Block, yellow

0.20 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298$ K

ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{\text{min}} = 0.544$, $T_{\text{max}} = 0.574$

10340 measured reflections

1488 independent reflections

1132 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.040$

$\theta_{\text{max}} = 27.5$°

$\theta_{\text{min}} = 2.2$°

$h = -15 \rightarrow 15$

$k = -8 \rightarrow 8$

$l = -19 \rightarrow 19$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.044$

$wR(F^2) = 0.122$

$S = 1.08$

1488 reflections

93 parameters

6 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0628P)^2 + 0.5702P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.001$

$\Delta\rho_{\text{max}} = 0.40$ e Å^{-3}

$\Delta\rho_{\text{min}} = -0.89$ e Å^{-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 (Å^2)

	x	*y*	*z*	$U_{\text{iso}}*/U_{\text{eq}}$
Br1	−0.24144 (4)	0.2500	0.43777 (4)	0.0689 (3)
Cl1	0.17429 (13)	0.2500	0.29606 (9)	0.0784 (5)
O1	−0.1127 (2)	0.2500	0.60853 (19)	0.0495 (7)
H1	−0.063 (5)	0.2500	0.652 (4)	0.074*
N1	0.0610 (3)	0.2500	0.7067 (2)	0.0529 (9)
C1	0.0712 (3)	0.2500	0.5489 (3)	0.0411 (9)
C2	−0.0460 (3)	0.2500	0.5392 (3)	0.0401 (9)
C3	−0.0878 (4)	0.2500	0.4537 (3)	0.0428 (9)
C4	−0.0210 (4)	0.2500	0.3801 (3)	0.0510 (11)
H4	−0.0517	0.2500	0.3235	0.061*
C5	0.0921 (4)	0.2500	0.3908 (3)	0.0515 (11)
C6	0.1374 (4)	0.2500	0.4740 (3)	0.0498 (10)
H6	0.2136	0.2500	0.4804	0.060*
C7	0.1190 (4)	0.2500	0.6365 (3)	0.0506 (10)
H7	0.1953	0.2500	0.6418	0.061*
C8	0.1145 (4)	0.2500	0.7942 (3)	0.0627 (14)
H8	0.1950	0.2500	0.7893	0.075*
C9	0.0723 (4)	0.0775 (6)	0.8482 (3)	0.0887 (14)
H9A	0.0620	−0.0359	0.8107	0.106*
H9B	0.1237	0.0447	0.8952	0.106*
C10	−0.0323 (5)	0.1421 (10)	0.8850 (4)	0.133 (2)
H10A	−0.0409	0.0934	0.9450	0.159*
H10B	−0.0928	0.0934	0.8493	0.159*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0544 (3)	0.0935 (5)	0.0588 (4)	0.000	−0.0150 (2)	0.000
Cl1	0.0888 (10)	0.0947 (11)	0.0517 (7)	0.000	0.0307 (7)	0.000
O1	0.0437 (16)	0.0639 (19)	0.0409 (16)	0.000	0.0048 (13)	0.000
N1	0.048 (2)	0.072 (3)	0.0388 (19)	0.000	−0.0052 (16)	0.000
C1	0.041 (2)	0.040 (2)	0.042 (2)	0.000	−0.0002 (16)	0.000
C2	0.049 (2)	0.032 (2)	0.039 (2)	0.000	0.0013 (17)	0.000
C3	0.051 (2)	0.035 (2)	0.042 (2)	0.000	−0.0035 (18)	0.000
C4	0.075 (3)	0.043 (2)	0.036 (2)	0.000	0.001 (2)	0.000
C5	0.063 (3)	0.049 (3)	0.042 (2)	0.000	0.015 (2)	0.000
C6	0.046 (2)	0.049 (2)	0.054 (3)	0.000	0.012 (2)	0.000
C7	0.041 (2)	0.061 (3)	0.051 (2)	0.000	−0.0026 (19)	0.000
C8	0.043 (2)	0.100 (4)	0.046 (2)	0.000	−0.010 (2)	0.000
C9	0.134 (4)	0.076 (3)	0.057 (2)	0.008 (3)	−0.034 (2)	0.007 (2)
C10	0.108 (4)	0.180 (6)	0.110 (4)	−0.023 (4)	0.015 (3)	0.040 (4)

Geometric parameters (Å, °)

Br1—C3	1.881 (5)	C5—C6	1.370 (6)

Cl1—C5	1.743 (4)	C6—H6	0.93
O1—C2	1.322 (5)	C7—H7	0.93
O1—H1	0.89 (6)	C8—C9	1.526 (5)
N1—C7	1.271 (6)	C8—C9^{i}	1.526 (5)
N1—C8	1.470 (5)	C8—H8	0.98
C1—C6	1.385 (6)	C9—C10	1.455 (7)
C1—C2	1.430 (6)	C9—H9A	0.97
C1—C7	1.443 (6)	C9—H9B	0.97
C2—C3	1.386 (6)	C10—C10^{i}	1.480 (14)
C3—C4	1.374 (6)	C10—H10A	0.97
C4—C5	1.383 (7)	C10—H10B	0.97
C4—H4	0.93		

C2—O1—H1	99 (4)	N1—C7—H7	118.7
C7—N1—C8	120.1 (4)	C1—C7—H7	118.7
C6—C1—C2	119.6 (4)	N1—C8—C9	109.3 (3)
C6—C1—C7	120.8 (4)	N1—C8—C9^{i}	109.3 (3)
C2—C1—C7	119.5 (4)	C9—C8—C9^{i}	101.7 (5)
O1—C2—C3	120.7 (4)	N1—C8—H8	112.0
O1—C2—C1	121.9 (4)	C9—C8—H8	112.0
C3—C2—C1	117.3 (4)	C9^{i}—C8—H8	112.0
C4—C3—C2	122.3 (4)	C10—C9—C8	105.1 (4)
C4—C3—Br1	118.9 (3)	C10—C9—H9A	110.7
C2—C3—Br1	118.8 (3)	C8—C9—H9A	110.7
C3—C4—C5	119.4 (4)	C10—C9—H9B	110.7
C3—C4—H4	120.3	C8—C9—H9B	110.7
C5—C4—H4	120.3	H9A—C9—H9B	108.8
C6—C5—C4	120.4 (4)	C9—C10—C10^{i}	107.7 (3)
C6—C5—Cl1	121.4 (4)	C9—C10—H10A	110.2
C4—C5—Cl1	118.2 (3)	C10^{i}—C10—H10A	110.2
C5—C6—C1	120.8 (4)	C9—C10—H10B	110.2
C5—C6—H6	119.6	C10^{i}—C10—H10B	110.2
C1—C6—H6	119.6	H10A—C10—H10B	108.5
N1—C7—C1	122.7 (4)		

C6—C1—C2—O1	180.0	C4—C5—C6—C1	0.0
C7—C1—C2—O1	0.0	Cl1—C5—C6—C1	180.0
C6—C1—C2—C3	0.0	C2—C1—C6—C5	0.0
C7—C1—C2—C3	180.0	C7—C1—C6—C5	180.0
O1—C2—C3—C4	180.0	C8—N1—C7—C1	180.0
C1—C2—C3—C4	0.0	C6—C1—C7—N1	180.0
O1—C2—C3—Br1	0.0	C2—C1—C7—N1	0.000 (1)
C1—C2—C3—Br1	180.0	C7—N1—C8—C9	−124.7 (3)
C2—C3—C4—C5	0.0	C7—N1—C8—C9^{i}	124.7 (3)
Br1—C3—C4—C5	180.0	N1—C8—C9—C10	−81.8 (4)
C3—C4—C5—C6	0.0	C9^{i}—C8—C9—C10	33.7 (5)
C3—C4—C5—Cl1	180.0	C8—C9—C10—C10^{i}	−21.6 (3)

Symmetry codes: (i) x, $-y+1/2$, z.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.89 (6)	1.71 (6)	2.577 (5)	162 (5)

Fig. 1