organic compounds

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

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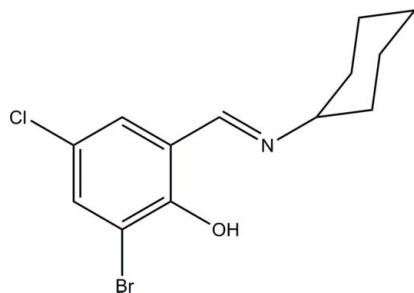Correspondence e-mail: majingjun71@yahoo.cn

The title compound, $C_{13}H_{15}BrClNO$, was prepared by the condensation of equimolar quantities of 3-bromo-5-chlorosalicylaldehyde with cyclohexylamine in methanol. There is an intramolecular O—H···N hydrogen bond in the molecule. The cyclohexyl ring adopts a chair conformation.

Related literature

For the coordination chemistry of Schiff base compounds, see: Xu *et al.* (2011); Suleiman Gwaram *et al.* (2011); Assey *et al.* (2011). For standard bond lengths, see: Allen *et al.* (1987). For similar structures, see: Miura *et al.* (2009); Damous *et al.* (2011); Şahin *et al.* (2009); Orona *et al.* (2011).

Experimental

Crystal data

$C_{13}H_{15}BrClNO$	$c = 6.969$ (1) Å
$M_r = 316.62$	$\beta = 101.634$ (2)°
Monoclinic, $P2_1/c$	$V = 1373.0$ (4) Å^3
$a = 12.296$ (2) Å	$Z = 4$
$b = 16.359$ (3) Å	Mo $K\alpha$ radiation
$\mu = 3.17$ mm^{-1}	$0.30 \times 0.30 \times 0.27$ mm
$T = 298$ K	

Data collection

Bruker SMART 1K CCD area-detector diffractometer	10912 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	2982 independent reflections
$T_{min} = 0.450$, $T_{max} = 0.481$	1705 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.042$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.073$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.227$	
$S = 1.04$	
2982 reflections	$\Delta\rho_{max} = 1.40$ e Å^{-3}
157 parameters	$\Delta\rho_{min} = -0.42$ e Å^{-3}
1 restraint	

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.90 (1)	1.71 (2)	2.564 (6)	159 (6)

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); 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 materials

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

D.-Y. Wang, X.-F. Meng and J.-J. Ma

Comment

Schiff bases are versatile ligands in coordination chemistry (Xu *et al.*, 2011; Suleiman Gwaram *et al.*, 2011; Assey *et al.*, 2011). As a contribution to a structural study on Schiff base compounds, we present here the crystal structure of the title compound, that was obtained as the product of the reaction of 3-bromo-5-chlorosalicylaldehyde with cyclohexylamine in methanol.

In the title compound, Fig. 1, there in an intramolecular O1—H1···N1 hydrogen bond (Table 1). The C1—C6 benzene ring is approximately perpendicular to the C8—C13 cyclohexyl ring. As expected, the cyclohexyl ring adopts a chair conformation. The bond distances and angles are within normal ranges (Allen *et al.*, 1987), and agree well with the corresponding bond distances and angles reported in closely related compounds (Miura *et al.*, 2009; Damous *et al.*, 2011; Şahin *et al.*, 2009; Orona *et al.*, 2011).

Experimental

To a methanol solution (10 ml) of 3-bromo-5-chlorosalicylaldehyde (0.1 mmol, 23.5 mg) and cyclohexylamine (0.1 mmol, 9.9 mg), a few drops of acetic acid were added. The mixture was refluxed for 1 h and then cooled to room temperature. The yellow crystalline solid was collected by filtration, washed with cold methanol and dried in air. Single crystals, suitable for X-ray diffraction, were obtained by slow evaporation of a methanol solution of the product in air.

Refinement

The OH H-atom was located in a difference Fourier map and was refined with a distance restraint, O—H = 0.90 (1) Å, and U_{iso}(H) = 0.08 Å^2. The C-bound H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93–0.98 Å, with U_{iso}(H) = 1.2U_{eq}(C).
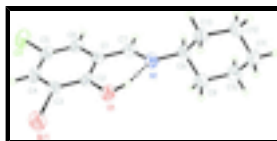
Figures

Fig. 1. The molecular structure of the title compound, with the numbering scheme and displacement ellipsoids drawn at the 30% probability level. The intramolecular O—H···N hydrogen bond is drawn as a dashed line.

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

Crystal data

C$_{13}$H$_{15}$BrClNO	$F(000) = 640$

$M_r = 316.62$

Monoclinic, $P2_1/c$

$a = 12.296\ (2)$ Å

$b = 16.359\ (3)$ Å

$c = 6.969\ (1)$ Å

$\beta = 101.634\ (2)°$

$V = 1373.0\ (4)$ Å^3

$Z = 4$

$D_x = 1.532$ Mg m^{-3}

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

Cell parameters from 2374 reflections

$\theta = 2.5–24.1°$

$\mu = 3.17$ mm^{-1}

$T = 298$ K

Block, yellow

$0.30 \times 0.30 \times 0.27$ mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

graphite

ω scan

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

$T_{min} = 0.450$, $T_{max} = 0.481$

10912 measured reflections

2982 independent reflections

1705 reflections with $I > 2\sigma(I)$

$R_{int} = 0.042$

$\theta_{max} = 27.0°$, $\theta_{min} = 2.5°$

$h = -15 \rightarrow 15$

$k = -20 \rightarrow 19$

$l = -8 \rightarrow 8$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.073$

$wR(F^2) = 0.227$

$S = 1.04$

2982 reflections

157 parameters

1 restraint

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

$w = 1/[\sigma^2(F_o^2) + (0.1329P)^2 + 0.243P]$
where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 1.40$ e Å^{-3}

$\Delta\rho_{min} = -0.42$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.36373 (6)	0.05122 (4)	−0.11130 (11)	0.0888 (4)
Cl1	0.09344 (12)	0.31936 (13)	−0.2918 (3)	0.0948 (6)
N1	0.6092 (4)	0.3288 (3)	0.0492 (7)	0.0594 (11)
O1	0.5335 (3)	0.1823 (2)	0.0130 (6)	0.0619 (9)
C1	0.4189 (4)	0.3005 (3)	−0.0749 (6)	0.0479 (11)
C2	0.4337 (4)	0.2154 (3)	−0.0606 (6)	0.0490 (11)
C3	0.3424 (4)	0.1648 (3)	−0.1214 (7)	0.0550 (12)
C4	0.2398 (4)	0.1959 (4)	−0.1955 (7)	0.0624 (14)
H4	0.1801	0.1612	−0.2402	0.075*
C5	0.2255 (4)	0.2799 (4)	−0.2032 (7)	0.0616 (14)
C6	0.3129 (4)	0.3316 (3)	−0.1472 (7)	0.0576 (13)
H6	0.3020	0.3878	−0.1572	0.069*
C7	0.5119 (4)	0.3548 (3)	−0.0185 (7)	0.0564 (12)
H7	0.5003	0.4109	−0.0322	0.068*
C8	0.7014 (4)	0.3862 (3)	0.0990 (8)	0.0601 (13)
H8	0.6719	0.4419	0.0785	0.072*
C9	0.7837 (5)	0.3731 (4)	−0.0324 (9)	0.0809 (18)
H9A	0.7478	0.3837	−0.1672	0.097*
H9B	0.8083	0.3167	−0.0231	0.097*
C10	0.8835 (6)	0.4293 (5)	0.0252 (12)	0.095 (2)
H10A	0.9367	0.4179	−0.0569	0.114*
H10B	0.8598	0.4857	0.0036	0.114*
C11	0.9385 (5)	0.4176 (4)	0.2383 (11)	0.089 (2)
H11A	0.9677	0.3625	0.2579	0.107*
H11B	1.0000	0.4555	0.2730	0.107*
C12	0.8578 (5)	0.4318 (5)	0.3658 (9)	0.0772 (18)
H12A	0.8343	0.4885	0.3550	0.093*
H12B	0.8937	0.4218	0.5010	0.093*
C13	0.7562 (5)	0.3768 (4)	0.3122 (8)	0.0674 (14)
H13A	0.7782	0.3203	0.3381	0.081*
H13B	0.7033	0.3907	0.3934	0.081*
H1	0.576 (4)	0.227 (2)	0.035 (9)	0.080*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0837 (6)	0.0650 (5)	0.1181 (7)	−0.0128 (3)	0.0212 (4)	−0.0034 (3)
Cl1	0.0463 (8)	0.1216 (15)	0.1084 (13)	0.0132 (8)	−0.0034 (8)	0.0139 (11)
N1	0.050 (2)	0.062 (3)	0.063 (3)	−0.004 (2)	0.0047 (19)	−0.006 (2)
O1	0.0451 (19)	0.063 (2)	0.076 (2)	0.0007 (16)	0.0071 (17)	−0.0017 (18)
C1	0.045 (2)	0.059 (3)	0.039 (2)	0.003 (2)	0.0040 (19)	0.003 (2)
C2	0.039 (2)	0.071 (3)	0.038 (2)	0.004 (2)	0.0115 (18)	0.002 (2)
C3	0.054 (3)	0.064 (3)	0.050 (3)	−0.009 (2)	0.016 (2)	−0.004 (2)
C4	0.042 (3)	0.089 (4)	0.054 (3)	−0.015 (3)	0.005 (2)	0.002 (3)

C5	0.042 (3)	0.091 (4)	0.052 (3)	0.007 (3)	0.007 (2)	0.006 (3)
C6	0.051 (3)	0.066 (3)	0.054 (3)	0.011 (3)	0.009 (2)	0.006 (2)
C7	0.055 (3)	0.056 (3)	0.057 (3)	0.000 (2)	0.010 (2)	0.002 (2)
C8	0.050 (3)	0.048 (3)	0.076 (4)	−0.006 (2)	−0.001 (2)	−0.001 (2)
C9	0.082 (4)	0.088 (4)	0.075 (4)	−0.025 (3)	0.022 (3)	−0.019 (3)
C10	0.082 (5)	0.108 (5)	0.104 (6)	−0.033 (4)	0.039 (4)	−0.026 (4)
C11	0.048 (3)	0.088 (4)	0.127 (6)	−0.008 (3)	0.009 (4)	−0.006 (4)
C12	0.064 (4)	0.086 (4)	0.077 (4)	−0.020 (3)	0.003 (3)	−0.011 (3)
C13	0.062 (3)	0.073 (4)	0.067 (3)	−0.016 (3)	0.015 (3)	−0.008 (3)

Geometric parameters (Å, °)

Br1—C3	1.875 (5)	C8—C13	1.510 (7)
Cl1—C5	1.741 (5)	C8—H8	0.9800
N1—C7	1.268 (6)	C9—C10	1.521 (8)
N1—C8	1.460 (6)	C9—H9A	0.9700
O1—C2	1.344 (5)	C9—H9B	0.9700
O1—H1	0.900 (10)	C10—C11	1.515 (10)
C1—C6	1.395 (7)	C10—H10A	0.9700
C1—C2	1.405 (7)	C10—H10B	0.9700
C1—C7	1.440 (7)	C11—C12	1.478 (9)
C2—C3	1.391 (7)	C11—H11A	0.9700
C3—C4	1.363 (7)	C11—H11B	0.9700
C4—C5	1.384 (8)	C12—C13	1.524 (7)
C4—H4	0.9300	C12—H12A	0.9700
C5—C6	1.362 (7)	C12—H12B	0.9700
C6—H6	0.9300	C13—H13A	0.9700
C7—H7	0.9300	C13—H13B	0.9700
C8—C9	1.510 (8)		
C7—N1—C8	120.1 (5)	C8—C9—H9A	109.4
C2—O1—H1	101 (4)	C10—C9—H9A	109.4
C6—C1—C2	119.1 (4)	C8—C9—H9B	109.4
C6—C1—C7	120.4 (5)	C10—C9—H9B	109.4
C2—C1—C7	120.5 (4)	H9A—C9—H9B	108.0
O1—C2—C3	119.7 (5)	C11—C10—C9	111.0 (6)
O1—C2—C1	121.4 (4)	C11—C10—H10A	109.4
C3—C2—C1	118.9 (4)	C9—C10—H10A	109.4
C4—C3—C2	121.5 (5)	C11—C10—H10B	109.4
C4—C3—Br1	119.7 (4)	C9—C10—H10B	109.4
C2—C3—Br1	118.7 (4)	H10A—C10—H10B	108.0
C3—C4—C5	119.1 (5)	C12—C11—C10	110.4 (5)
C3—C4—H4	120.5	C12—C11—H11A	109.6
C5—C4—H4	120.5	C10—C11—H11A	109.6
C6—C5—C4	121.3 (4)	C12—C11—H11B	109.6
C6—C5—Cl1	119.8 (5)	C10—C11—H11B	109.6
C4—C5—Cl1	118.9 (4)	H11A—C11—H11B	108.1
C5—C6—C1	120.1 (5)	C11—C12—C13	112.2 (5)
C5—C6—H6	119.9	C11—C12—H12A	109.2
C1—C6—H6	119.9	C13—C12—H12A	109.2

N1—C7—C1	122.2 (5)	C11—C12—H12B	109.2
N1—C7—H7	118.9	C13—C12—H12B	109.2
C1—C7—H7	118.9	H12A—C12—H12B	107.9
N1—C8—C9	110.4 (4)	C8—C13—C12	111.2 (5)
N1—C8—C13	109.8 (4)	C8—C13—H13A	109.4
C9—C8—C13	111.2 (5)	C12—C13—H13A	109.4
N1—C8—H8	108.4	C8—C13—H13B	109.4
C9—C8—H8	108.4	C12—C13—H13B	109.4
C13—C8—H8	108.4	H13A—C13—H13B	108.0
C8—C9—C10	111.0 (5)		

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.90 (1)	1.71 (2)	2.564 (6)	159 (6)

Fig. 1