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N′-(3-Bromo-5-chloro-2-hydroxybenzylidine)-2-hydroxybenzohydrazide

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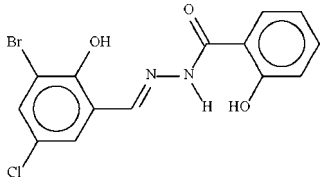
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.004 Å; R factor = 0.029; wR factor = 0.077; data-to-parameter ratio = 15.5.

In the approximately planar title molecule, $C_{14}H_{10}BrClN_3O_2$, the dihedral angle between the aromatic ring planes is 5.79 (12)°. The conformation is stabilized by intramolecular O—H···N and N—H···O hydrogen bonds and an intermolecular O—H···O link leads to chains in the crystal propagating in [001].

Related literature

For similar Schiff bases, see: Hu *et al.* (2005); Wu *et al.* (2006); Yehye *et al.* (2008*a*,*b*).



Experimental

Crystal data

$C_{14}H_{10}BrClN_2O_3$
M_r = 369.60
Monoclinic, $P2_1/c$
a = 15.8387 (3) Å
b = 6.9319 (1) Å
c = 12.9951 (3) Å
β = 106.461 (1)°
V = 1368.28 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.21 mm^{-1}
T = 100 (2) K
0.30 × 0.20 × 0.05 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.446, T_{max} = 0.856
12350 measured reflections
3136 independent reflections
2545 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.077
S = 1.02
3136 reflections
202 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1*o*···O2^{i}	0.83 (1)	1.76 (1)	2.591 (2)	175 (3)
O3—H3*o*···N2	0.84 (1)	1.88 (2)	2.632 (3)	149 (3)
N1—H1*n*···O1	0.87 (1)	1.90 (2)	2.614 (3)	139 (2)

Symmetry code: (i) $x, -y+\frac{3}{2}, z+\frac{1}{2}$.

Data collection: *APEX2* (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: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2008).

We thank the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2857).

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supporting information

Acta Cryst. (2008). E**64**, o2438 [doi:10.1107/S1600536808038634]

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S1. Comment

In the approximately planar title molecule, (I), (Fig. 1) the dihedral angle between the aromatic ring planes is 5.79 (12)°. The conformation is stabilised by intramolecular O—H···N and N—H···O hydrogen bonds and an intermolecular O—H···O link leads to chains in the crystal (Table 1).

S2. Experimental

2-Hydroxybenzohydrazide(0.60 g, 4 mmol) and 3-bromo-5-chloro-2-ydroxybenzaldehyde (0.94 g, 4 mmol) were heated in ethanol (30 ml) for 2 h. The solvent was removed by evaporation and the resulting solid was recrystallized from ethanol to yield yellow plates of (I).

S3. Refinement

The carbon-bound H-atoms were placed in calculated positions (C–H = 0.95 Å) and refined as riding with U(H) = 1.2U(C). The oxygen- and nitrogen-bound H-atoms were located in a difference map, and were refined with distance restraints of O–H 0.84±0.01 and N–H 0.88±0.01 Å. Their U_{iso} values were freely refined.

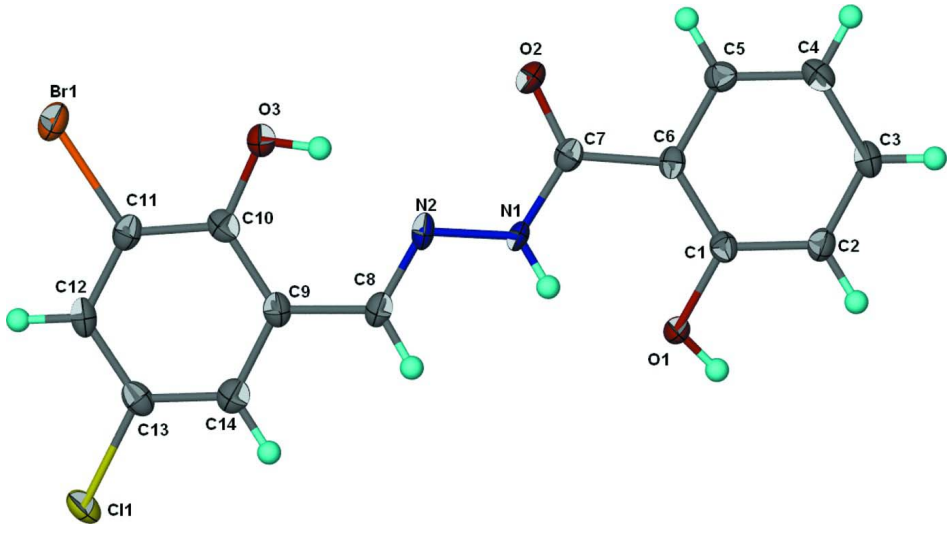

Figure 1

The molecular structure of (I) with atoms shown at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radiius.

N′-(3-Bromo-5-chloro-2-hydroxybenzylidine)-2-hydroxybenzohydrazide

Crystal data

$C_{14}H_{10}BrClN_2O_3$
$M_r = 369.60$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 15.8387$ (3) Å
$b = 6.9319$ (1) Å
$c = 12.9951$ (3) Å
$\beta = 106.461$ (1)°
$V = 1368.28$ (5) Å^3
$Z = 4$
$F(000) = 736$
$D_x = 1.794$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3691 reflections
$\theta = 3.2$–$28.2°$
$\mu = 3.21$ mm^{-1}
$T = 100$ K
Plate, yellow
0.30 × 0.20 × 0.05 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.446$, $T_{max} = 0.856$
12350 measured reflections
3136 independent reflections
2545 reflections with $I > 2\sigma(I)$
$R_{int} = 0.038$
$\theta_{max} = 27.5°$, $\theta_{min} = 1.3°$
$h = -20 \rightarrow 20$
$k = -9 \rightarrow 8$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.077$
$S = 1.02$
3136 reflections
202 parameters
3 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difmap and geom
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0353P)^2 + 1.3959P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.41$ 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.145097 (18)	0.43521 (4)	0.04463 (2)	0.02196 (9)
Cl1	−0.04591 (4)	0.45367 (9)	0.33930 (5)	0.01932 (14)
O1	0.48671 (11)	0.8436 (3)	0.72174 (14)	0.0186 (4)
H1O	0.495 (2)	0.837 (5)	0.7879 (9)	0.033 (9)*
O2	0.52114 (12)	0.6952 (3)	0.42779 (14)	0.0224 (4)
O3	0.28679 (12)	0.5844 (3)	0.23371 (14)	0.0184 (4)
H3O	0.3254 (15)	0.616 (4)	0.2897 (15)	0.027 (9)*
N1	0.42237 (13)	0.7471 (3)	0.52022 (16)	0.0142 (4)

H1N	0.4157 (17)	0.778 (4)	0.5825 (12)	0.015 (7)*
N2	0.35545 (13)	0.6804 (3)	0.43620 (16)	0.0154 (4)
C1	0.56768 (15)	0.8577 (4)	0.70555 (19)	0.0140 (5)
C2	0.64075 (16)	0.9151 (4)	0.7881 (2)	0.0163 (5)
H2	0.6343	0.9445	0.8569	0.020*
C3	0.72242 (16)	0.9295 (4)	0.7702 (2)	0.0171 (5)
H3	0.7720	0.9670	0.8271	0.021*
C4	0.73279 (16)	0.8897 (4)	0.6701 (2)	0.0174 (5)
H4	0.7890	0.9010	0.6581	0.021*
C5	0.66035 (16)	0.8333 (4)	0.5875 (2)	0.0158 (5)
H5	0.6674	0.8061	0.5187	0.019*
C6	0.57684 (15)	0.8157 (3)	0.60395 (19)	0.0131 (5)
C7	0.50521 (16)	0.7480 (3)	0.51062 (19)	0.0149 (5)
C8	0.27901 (16)	0.6691 (4)	0.4507 (2)	0.0154 (5)
H8	0.2706	0.7070	0.5174	0.018*
C9	0.20470 (16)	0.5978 (3)	0.3648 (2)	0.0155 (5)
C10	0.21190 (16)	0.5580 (3)	0.2611 (2)	0.0153 (5)
C11	0.13777 (17)	0.4882 (4)	0.1845 (2)	0.0162 (5)
C12	0.05895 (16)	0.4549 (3)	0.2073 (2)	0.0169 (5)
H12	0.0095	0.4062	0.1538	0.020*
C13	0.05363 (16)	0.4941 (4)	0.3096 (2)	0.0163 (5)
C14	0.12487 (17)	0.5652 (3)	0.3880 (2)	0.0171 (5)
H14	0.1197	0.5921	0.4577	0.020*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.02322 (15)	0.02735 (15)	0.01301 (13)	−0.00167 (11)	0.00141 (10)	−0.00143 (11)
Cl1	0.0119 (3)	0.0207 (3)	0.0259 (3)	−0.0026 (2)	0.0063 (2)	−0.0016 (3)
O1	0.0127 (9)	0.0334 (10)	0.0096 (8)	−0.0027 (8)	0.0032 (7)	−0.0009 (8)
O2	0.0184 (9)	0.0369 (11)	0.0111 (9)	0.0009 (8)	0.0027 (7)	−0.0044 (8)
O3	0.0149 (9)	0.0247 (10)	0.0149 (9)	−0.0035 (7)	0.0029 (7)	−0.0025 (8)
N1	0.0129 (10)	0.0201 (11)	0.0079 (10)	−0.0002 (8)	0.0000 (8)	−0.0028 (8)
N2	0.0148 (10)	0.0162 (10)	0.0113 (10)	0.0002 (8)	−0.0027 (8)	−0.0009 (8)
C1	0.0124 (12)	0.0151 (12)	0.0136 (12)	0.0003 (9)	0.0020 (9)	0.0018 (10)
C2	0.0162 (12)	0.0192 (12)	0.0122 (12)	−0.0016 (10)	0.0018 (10)	−0.0003 (10)
C3	0.0131 (12)	0.0181 (12)	0.0176 (12)	−0.0002 (10)	0.0000 (10)	0.0005 (11)
C4	0.0127 (12)	0.0188 (13)	0.0209 (13)	0.0007 (10)	0.0052 (10)	0.0017 (10)
C5	0.0167 (12)	0.0163 (12)	0.0150 (12)	0.0017 (10)	0.0054 (10)	0.0012 (10)
C6	0.0137 (11)	0.0127 (11)	0.0113 (11)	0.0010 (9)	0.0008 (9)	0.0021 (9)
C7	0.0168 (12)	0.0151 (11)	0.0116 (12)	0.0017 (10)	0.0018 (10)	0.0028 (10)
C8	0.0170 (12)	0.0144 (12)	0.0124 (11)	0.0013 (10)	0.0005 (10)	−0.0014 (10)
C9	0.0140 (12)	0.0147 (12)	0.0157 (12)	0.0005 (9)	0.0009 (10)	−0.0004 (10)
C10	0.0144 (12)	0.0133 (12)	0.0177 (12)	0.0022 (9)	0.0038 (10)	0.0040 (10)
C11	0.0185 (13)	0.0147 (11)	0.0132 (12)	0.0014 (10)	0.0011 (10)	0.0004 (10)
C12	0.0140 (12)	0.0152 (12)	0.0175 (13)	−0.0008 (10)	−0.0021 (10)	−0.0002 (10)
C13	0.0126 (12)	0.0132 (11)	0.0224 (13)	0.0007 (9)	0.0038 (10)	0.0010 (10)
C14	0.0188 (12)	0.0150 (12)	0.0166 (12)	0.0020 (10)	0.0037 (10)	0.0003 (10)

Geometric parameters (Å, º)

Br1—C11	1.890 (3)	C3—H3	0.9500
Cl1—C13	1.749 (3)	C4—C5	1.387 (4)
O1—C1	1.361 (3)	C4—H4	0.9500
O1—H1O	0.833 (10)	C5—C6	1.404 (3)
O2—C7	1.229 (3)	C5—H5	0.9500
O3—C10	1.344 (3)	C6—C7	1.483 (3)
O3—H3O	0.836 (10)	C8—C9	1.459 (3)
N1—C7	1.353 (3)	C8—H8	0.9500
N1—N2	1.369 (3)	C9—C14	1.399 (4)
N1—H1N	0.872 (10)	C9—C10	1.412 (4)
N2—C8	1.279 (3)	C10—C11	1.393 (3)
C1—C2	1.395 (3)	C11—C12	1.382 (4)
C1—C6	1.399 (3)	C12—C13	1.383 (4)
C2—C3	1.382 (4)	C12—H12	0.9500
C2—H2	0.9500	C13—C14	1.379 (4)
C3—C4	1.384 (4)	C14—H14	0.9500
C1—O1—H1O	106 (2)	O2—C7—N1	121.6 (2)
C10—O3—H3O	107 (2)	O2—C7—C6	120.8 (2)
C7—N1—N2	118.7 (2)	N1—C7—C6	117.6 (2)
C7—N1—H1N	117.4 (18)	N2—C8—C9	120.0 (2)
N2—N1—H1N	123.5 (18)	N2—C8—H8	120.0
C8—N2—N1	117.0 (2)	C9—C8—H8	120.0
O1—C1—C2	121.0 (2)	C14—C9—C10	119.8 (2)
O1—C1—C6	119.0 (2)	C14—C9—C8	118.2 (2)
C2—C1—C6	120.0 (2)	C10—C9—C8	122.0 (2)
C3—C2—C1	120.2 (2)	O3—C10—C11	119.1 (2)
C3—C2—H2	119.9	O3—C10—C9	122.8 (2)
C1—C2—H2	119.9	C11—C10—C9	118.0 (2)
C2—C3—C4	120.7 (2)	C12—C11—C10	122.3 (2)
C2—C3—H3	119.6	C12—C11—Br1	118.63 (19)
C4—C3—H3	119.6	C10—C11—Br1	119.05 (19)
C3—C4—C5	119.4 (2)	C11—C12—C13	118.6 (2)
C3—C4—H4	120.3	C11—C12—H12	120.7
C5—C4—H4	120.3	C13—C12—H12	120.7
C4—C5—C6	121.0 (2)	C14—C13—C12	121.3 (2)
C4—C5—H5	119.5	C14—C13—Cl1	119.6 (2)
C6—C5—H5	119.5	C12—C13—Cl1	119.01 (19)
C1—C6—C5	118.7 (2)	C13—C14—C9	119.9 (2)
C1—C6—C7	125.3 (2)	C13—C14—H14	120.0
C5—C6—C7	116.0 (2)	C9—C14—H14	120.0
C7—N1—N2—C8	175.3 (2)	N2—C8—C9—C14	172.3 (2)
O1—C1—C2—C3	−179.7 (2)	N2—C8—C9—C10	−6.4 (4)
C6—C1—C2—C3	−0.5 (4)	C14—C9—C10—O3	−179.4 (2)
C1—C2—C3—C4	0.8 (4)	C8—C9—C10—O3	−0.7 (4)

C2—C3—C4—C5	−0.6 (4)	C14—C9—C10—C11	0.5 (4)
C3—C4—C5—C6	−0.1 (4)	C8—C9—C10—C11	179.2 (2)
O1—C1—C6—C5	179.1 (2)	O3—C10—C11—C12	179.1 (2)
C2—C1—C6—C5	−0.2 (4)	C9—C10—C11—C12	−0.9 (4)
O1—C1—C6—C7	−2.6 (4)	O3—C10—C11—Br1	−0.5 (3)
C2—C1—C6—C7	178.1 (2)	C9—C10—C11—Br1	179.55 (18)
C4—C5—C6—C1	0.4 (4)	C10—C11—C12—C13	0.6 (4)
C4—C5—C6—C7	−178.0 (2)	Br1—C11—C12—C13	−179.88 (18)
N2—N1—C7—O2	1.8 (4)	C11—C12—C13—C14	0.1 (4)
N2—N1—C7—C6	−178.3 (2)	C11—C12—C13—Cl1	179.70 (19)
C1—C6—C7—O2	−172.2 (2)	C12—C13—C14—C9	−0.5 (4)
C5—C6—C7—O2	6.1 (3)	Cl1—C13—C14—C9	179.98 (18)
C1—C6—C7—N1	7.9 (4)	C10—C9—C14—C13	0.1 (4)
C5—C6—C7—N1	−173.8 (2)	C8—C9—C14—C13	−178.6 (2)
N1—N2—C8—C9	−179.6 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1o···O2^{i}	0.83 (1)	1.76 (1)	2.591 (2)	175 (3)
O3—H3o···N2	0.84 (1)	1.88 (2)	2.632 (3)	149 (3)
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