Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Chloro-*N*′-(3,5-dibromo-2-hydroxybenzylidene)benzohydrazide methanol solvate

Chun-Bao Tang

Department of Chemistry, Jiaying University, Meizhou 514015, People's Republic of China
Correspondence e-mail: chunbao_tang@163.com

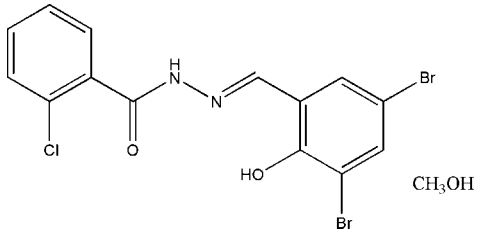
Received 25 June 2008; accepted 27 June 2008

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.012 Å; R factor = 0.069; wR factor = 0.160; data-to-parameter ratio = 16.9.

The title Schiff base compound, $C_{14}H_9Br_2ClN_2O_2\cdot CH_4O$, was derived from the condensation reaction of 3,5-dibromosalicylaldehyde with 2-chlorobenzohydrazide. The dihedral angle between the two benzene rings is 48.2 (2)°. In the crystal structure, molecules are linked through O—H···O and N—H···O intermolecular hydrogen bonds, forming layers parallel to the *bc* plane. There is also an O—H···N intramolecular hydrogen bond.

Related literature

For related structures, see: Tang (2006); Tang, (2007*a*,*b*,*c*,*d*). For reference structural data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{14}H_9Br_2ClN_2O_2\cdot CH_4O$
M_r = 464.54
Monoclinic, $P2_1/c$
a = 11.156 (4) Å
b = 9.696 (3) Å
c = 18.536 (3) Å
β = 120.356 (8)°
V = 1730.1 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 4.85 mm^{-1}
T = 298 (2) K
0.23 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.402, T_{max} = 0.444
(expected range = 0.343–0.379)
9035 measured reflections
3627 independent reflections
1895 reflections with $I > 2\sigma(I)$
R_{int} = 0.101

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.069
$wR(F^2)$ = 0.160
S = 0.92
3627 reflections
215 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.81 e Å^{-3}
$\Delta\rho_{min}$ = −0.71 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3*A*···O2^{i}	0.82	2.05	2.697 (9)	136
N2—H2···O3	0.89 (7)	1.946 (17)	2.840 (7)	173 (8)
O1—H1···N1	0.82	1.91	2.590 (6)	140

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

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*.

Financial support from the Jiaying University research fund is gratefully acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2583).

References

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). *SAINT* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Tang, C.-B. (2006). *Acta Cryst.* E**62**, m2629–m2630.
Tang, C.-B. (2007*a*). *Acta Cryst.* E**63**, m2654.
Tang, C.-B. (2007*b*). *Acta Cryst.* E**63**, m2785–m2786.
Tang, C.-B. (2007*c*). *Acta Cryst.* E**63**, o4545.
Tang, C.-B. (2007*d*). *Acta Cryst.* E**63**, o4841.

supporting information

Acta Cryst. (2008). E**64**, o1382 [doi:10.1107/S160053680801965X]

2-Chloro-*N*′-(3,5-dibromo-2-hydroxybenzylidene)benzohydrazide methanol solvate

Chun-Bao Tang

S1. Comment

Recently, the author has reported the structures of several Schiff base compounds (Tang, 2006;Tang, 2007*a*,b,c,d) and, in continuation of work in this area, reports herein the structure of the title compound, (I), a new Schiff base compound.

In the title compound (Fig. 1), the dihedral angle between the two benzene rings is 48.2 (2)°. The torsion angles C1—C7—N1—N2, C7—N1—N2—C8, and N1—N2—C8—C9 are 0.1 (2), 4.8 (2), and 4.3 (2)°, respectively. All the bond lengths are within normal values (Allen *et al.*, 1987).

In the crystal structure of the compound, molecules are linked through O–H···O intermolecular hydrogen bonds (Table 1), forming layers parallel to the *bc* plane (Fig. 2).

S2. Experimental

3,5-Dibromosalicylaldehyde (0.1 mmol, 28.0 mg) and 2-chlorobenzohydrazide (0.1 mmol, 17.0 mg) were dissolved in a methanol solution (20 ml). The mixture was stirred at reflux for 10 min to give a clear colorless solution. Colourless block-like crystals of the compound were formed by slow evaporation of the solvent over several days.

S3. Refinement

H2 was located from a difference Fourier map and refined isotropically, with U_{iso} restrained to 0.08Å^2. Other H atoms were constrained to ideal geometries, with d(C–H) = 0.93–0.96 Å, d(O–H) = 0.82 Å, and with U_{iso}(H) = 1.2U_{eq}(C), 1.5U_{eq}(C15, O1 and O3).

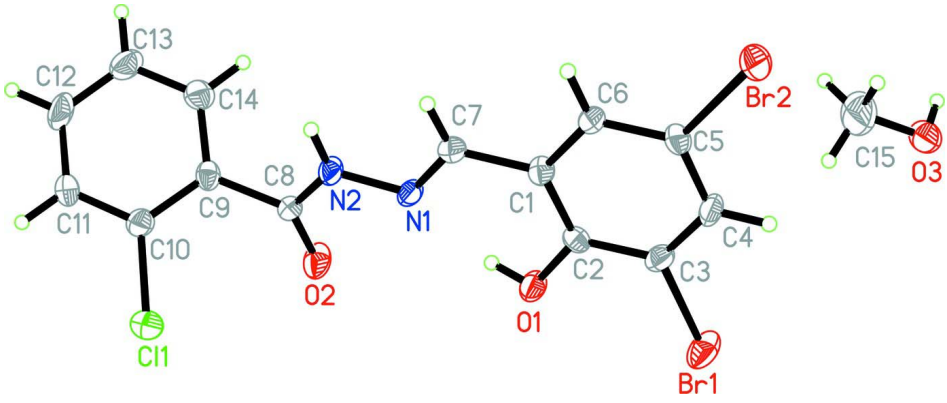


Figure 1

The molecular structure of the compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

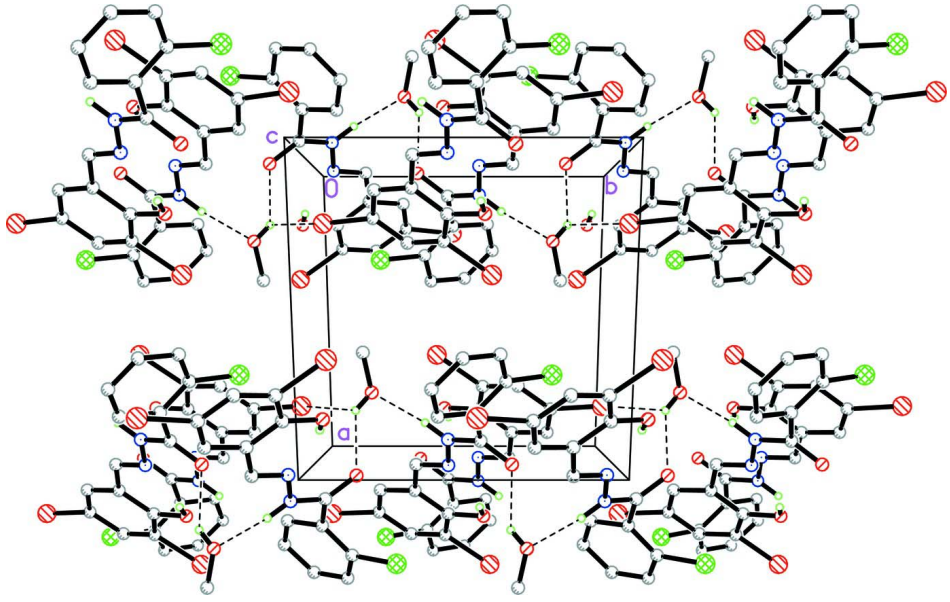


Figure 2

Molecular packing of (I) with hydrogen bonds drawn as dashed lines.

2-Chloro-*N*′-(3,5-dibromo-2-hydroxybenzylidene)benzohydrazide methanol solvate

Crystal data

$C_{14}H_9Br_2ClN_2O_2\cdot CH_4O$
$M_r = 464.54$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 11.156\ (4)$ Å
$b = 9.696\ (3)$ Å
$c = 18.536\ (3)$ Å
$\beta = 120.356\ (8)°$
$V = 1730.1\ (9)$ Å^3
$Z = 4$
$F(000) = 912$
$D_x = 1.783$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2286 reflections
$\theta = 2.4–24.5°$
$\mu = 4.85$ mm^{-1}
$T = 298$ K
Block, colourless
0.23 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.402$, $T_{max} = 0.444$
9035 measured reflections
3627 independent reflections
1895 reflections with $I > 2\sigma(I)$
$R_{int} = 0.101$
$\theta_{max} = 27.0°$, $\theta_{min} = 2.1°$
$h = -14\rightarrow14$
$k = -12\rightarrow10$
$l = -23\rightarrow22$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.069$
$wR(F^2) = 0.160$
$S = 0.92$
3627 reflections
215 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
$w = 1/[\sigma^2(F_o^2) + (0.0746P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.81$ e Å^{-3}
$\Delta\rho_{min} = -0.71$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0119 (12)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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	−0.36755 (9)	1.09036 (8)	0.95218 (6)	0.0603 (4)
Br2	−0.19725 (9)	0.54059 (7)	1.04896 (6)	0.0557 (3)
Cl1	0.3094 (2)	1.28217 (17)	0.81645 (14)	0.0580 (6)
O1	−0.1788 (5)	1.0664 (4)	0.8816 (3)	0.0443 (14)
H1	−0.1429	1.0532	0.8531	0.067*
O2	0.0171 (5)	1.1554 (5)	0.7617 (4)	0.0543 (15)
O3	0.2494 (6)	0.7016 (5)	0.8572 (4)	0.0560 (16)
H3A	0.1924	0.6511	0.8203	0.084*
N1	0.0034 (6)	0.9492 (5)	0.8546 (4)	0.0373 (16)
N2	0.0939 (6)	0.9488 (5)	0.8251 (4)	0.0388 (16)
C1	−0.0946 (7)	0.8351 (6)	0.9261 (4)	0.0325 (16)
C2	−0.1773 (7)	0.9478 (5)	0.9204 (4)	0.0307 (16)
C3	−0.2590 (7)	0.9362 (6)	0.9571 (5)	0.0374 (18)
C4	−0.2624 (7)	0.8177 (6)	0.9961 (5)	0.0400 (19)
H4	−0.3182	0.8124	1.0200	0.048*
C5	−0.1835 (7)	0.7073 (6)	0.9999 (5)	0.0386 (18)
C6	−0.0988 (7)	0.7157 (6)	0.9662 (5)	0.0389 (19)
H6	−0.0438	0.6407	0.9702	0.047*
C7	0.0008 (7)	0.8415 (6)	0.8926 (5)	0.0415 (19)
H7	0.0583	0.7672	0.8993	0.050*
C8	0.0941 (7)	1.0545 (6)	0.7797 (5)	0.0353 (18)
C9	0.1906 (7)	1.0373 (6)	0.7455 (4)	0.0357 (17)
C10	0.2917 (8)	1.1341 (6)	0.7595 (5)	0.0415 (19)
C11	0.3785 (8)	1.1138 (7)	0.7278 (5)	0.049 (2)
H11	0.4449	1.1799	0.7366	0.058*
C12	0.3676 (9)	0.9973 (8)	0.6836 (6)	0.059 (3)
H12	0.4265	0.9842	0.6624	0.071*
C13	0.2705 (9)	0.9004 (7)	0.6706 (6)	0.056 (2)
H13	0.2645	0.8205	0.6412	0.068*
C14	0.1819 (8)	0.9187 (7)	0.7002 (5)	0.048 (2)

H14	0.1152	0.8521	0.6901	0.057*
C15	0.3807 (10)	0.6749 (9)	0.8694 (7)	0.092 (4)
H15A	0.4377	0.7555	0.8916	0.138*
H15B	0.3730	0.6508	0.8170	0.138*
H15C	0.4221	0.5998	0.9080	0.138*
H2	0.149 (7)	0.875 (5)	0.839 (5)	0.080*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0653 (7)	0.0618 (5)	0.0723 (7)	0.0269 (4)	0.0484 (6)	0.0132 (4)
Br2	0.0785 (8)	0.0421 (4)	0.0704 (7)	0.0006 (4)	0.0551 (6)	0.0114 (4)
Cl1	0.0664 (17)	0.0464 (10)	0.0727 (16)	−0.0114 (9)	0.0436 (15)	−0.0142 (10)
O1	0.055 (4)	0.040 (2)	0.055 (4)	0.009 (2)	0.040 (3)	0.008 (2)
O2	0.050 (4)	0.047 (3)	0.080 (4)	0.019 (3)	0.044 (4)	0.025 (3)
O3	0.044 (4)	0.045 (3)	0.080 (5)	0.002 (2)	0.033 (4)	0.002 (3)
N1	0.048 (4)	0.030 (3)	0.050 (4)	0.008 (2)	0.037 (4)	0.005 (3)
N2	0.047 (4)	0.030 (3)	0.057 (4)	0.007 (2)	0.039 (4)	0.011 (3)
C1	0.028 (5)	0.034 (3)	0.034 (4)	0.005 (3)	0.015 (4)	0.007 (3)
C2	0.030 (5)	0.027 (3)	0.030 (4)	−0.001 (3)	0.012 (4)	0.004 (3)
C3	0.036 (5)	0.037 (3)	0.042 (5)	0.006 (3)	0.021 (4)	−0.004 (3)
C4	0.038 (5)	0.051 (4)	0.044 (5)	0.000 (4)	0.030 (5)	−0.004 (4)
C5	0.049 (5)	0.035 (3)	0.041 (5)	−0.006 (3)	0.030 (5)	0.004 (3)
C6	0.038 (5)	0.031 (3)	0.059 (6)	0.005 (3)	0.032 (5)	−0.001 (3)
C7	0.049 (5)	0.029 (3)	0.059 (6)	0.002 (3)	0.036 (5)	−0.001 (3)
C8	0.039 (5)	0.033 (3)	0.042 (5)	−0.004 (3)	0.026 (4)	−0.007 (3)
C9	0.034 (5)	0.038 (3)	0.039 (5)	0.003 (3)	0.022 (4)	0.010 (3)
C10	0.051 (6)	0.041 (3)	0.043 (5)	−0.003 (3)	0.032 (5)	−0.003 (3)
C11	0.046 (6)	0.050 (4)	0.064 (6)	−0.009 (3)	0.039 (5)	−0.003 (4)
C12	0.065 (6)	0.071 (5)	0.074 (7)	0.004 (4)	0.059 (6)	0.004 (5)
C13	0.077 (7)	0.052 (4)	0.063 (6)	0.005 (4)	0.052 (6)	−0.004 (4)
C14	0.061 (6)	0.040 (4)	0.052 (6)	−0.003 (3)	0.036 (5)	0.003 (4)
C15	0.069 (8)	0.083 (6)	0.148 (11)	0.012 (6)	0.072 (9)	0.010 (7)

Geometric parameters (Å, º)

Br1—C3	1.897 (6)	C4—H4	0.9300
Br2—C5	1.899 (6)	C5—C6	1.372 (8)
Cl1—C10	1.733 (6)	C6—H6	0.9300
O1—C2	1.352 (6)	C7—H7	0.9300
O1—H1	0.8200	C8—C9	1.509 (8)
O2—C8	1.232 (7)	C9—C10	1.387 (9)
O3—C15	1.389 (9)	C9—C14	1.399 (9)
O3—H3A	0.8200	C10—C11	1.377 (8)
N1—C7	1.269 (7)	C11—C12	1.365 (10)
N1—N2	1.369 (6)	C11—H11	0.9300
N2—C8	1.327 (7)	C12—C13	1.361 (10)
N2—H2	0.89 (7)	C12—H12	0.9300

C1—C6	1.390 (8)	C13—C14	1.362 (9)
C1—C2	1.400 (8)	C13—H13	0.9300
C1—C7	1.480 (8)	C14—H14	0.9300
C2—C3	1.389 (8)	C15—H15A	0.9600
C3—C4	1.368 (8)	C15—H15B	0.9600
C4—C5	1.366 (8)	C15—H15C	0.9600
C2—O1—H1	109.5	O2—C8—N2	124.1 (5)
C15—O3—H3A	109.5	O2—C8—C9	121.5 (6)
C7—N1—N2	116.6 (5)	N2—C8—C9	114.3 (5)
C8—N2—N1	119.3 (5)	C10—C9—C14	118.1 (5)
C8—N2—H2	125 (5)	C10—C9—C8	122.2 (6)
N1—N2—H2	115 (5)	C14—C9—C8	119.7 (5)
C6—C1—C2	119.4 (5)	C11—C10—C9	120.3 (6)
C6—C1—C7	119.0 (5)	C11—C10—Cl1	119.3 (5)
C2—C1—C7	121.5 (5)	C9—C10—Cl1	120.4 (4)
O1—C2—C3	119.6 (5)	C12—C11—C10	120.4 (6)
O1—C2—C1	122.3 (5)	C12—C11—H11	119.8
C3—C2—C1	118.1 (5)	C10—C11—H11	119.8
C4—C3—C2	121.8 (5)	C13—C12—C11	120.0 (6)
C4—C3—Br1	119.9 (4)	C13—C12—H12	120.0
C2—C3—Br1	118.3 (5)	C11—C12—H12	120.0
C5—C4—C3	119.7 (5)	C12—C13—C14	120.9 (7)
C5—C4—H4	120.1	C12—C13—H13	119.6
C3—C4—H4	120.1	C14—C13—H13	119.6
C4—C5—C6	120.3 (5)	C13—C14—C9	120.3 (7)
C4—C5—Br2	119.0 (4)	C13—C14—H14	119.8
C6—C5—Br2	120.6 (5)	C9—C14—H14	119.8
C5—C6—C1	120.7 (5)	O3—C15—H15A	109.5
C5—C6—H6	119.7	O3—C15—H15B	109.5
C1—C6—H6	119.7	H15A—C15—H15B	109.5
N1—C7—C1	119.4 (5)	O3—C15—H15C	109.5
N1—C7—H7	120.3	H15A—C15—H15C	109.5
C1—C7—H7	120.3	H15B—C15—H15C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3*A*···O2^{i}	0.82	2.05	2.697 (9)	136
N2—H2···O3	0.89 (7)	1.95 (2)	2.840 (7)	173 (8)
O1—H1···N1	0.82	1.91	2.590 (6)	140

Symmetry code: (i) $-x, y-1/2, -z+3/2$.