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4-Hydroxy-*N'*-(3,5-dichloro-2-hydroxybenzylidene)benzohydrazide

Chong-Gui Ren

 Department of Chemistry and Chemical Engineering, Zaozhuang University,
 Zaozhuang Shandong 277160, People's Republic of China
 Correspondence e-mail: renchonggui@163.com

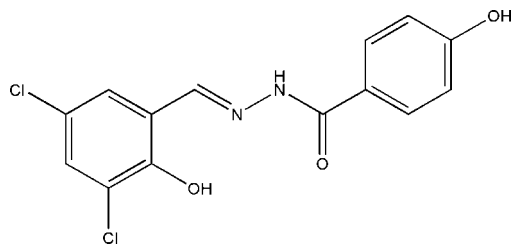
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 Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å;
 R factor = 0.039; wR factor = 0.107; data-to-parameter ratio = 15.6.

In the title compound, $\text{C}_{14}\text{H}_{10}\text{Cl}_2\text{N}_2\text{O}_3$, the dihedral angle between the two benzene rings is $5.1(2)^\circ$. The molecule adopts an *E* configuration with respect to the $\text{C}=\text{N}$ bond and an intramolecular $\text{O}-\text{H}\cdots\text{N}$ interaction is present. In the crystal structure, molecules are linked through intermolecular $\text{N}-\text{H}\cdots\text{O}$ and $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For the biological properties of Schiff base compounds, see: Jeewoth *et al.* (1999); Ren *et al.* (2002); Eltayeb *et al.* (2008); Sinha *et al.* (2008). For metal complexes of Schiff base compounds, see: Shivakumar *et al.* (2008); Prabhakaran *et al.* (2006); Dhar *et al.* (2005). For related structures, see: Cui *et al.* (2007); Jing *et al.* (2007); Ma *et al.* (2008); Salhin *et al.* (2007); Lin *et al.* (2007); Alhadi *et al.* (2008); Xue *et al.* (2008); Wang *et al.* (2008); Lu (2008); Diao *et al.* (2008); Qiu (2009); Mohd Lair *et al.* (2009*a,b*). For reference structural data, see: Allen *et al.* (1987).



Experimental

Crystal data

 $\text{C}_{14}\text{H}_{10}\text{Cl}_2\text{N}_2\text{O}_3$
 $M_r = 325.14$
 Monoclinic, $P2_1/c$
 $a = 8.030(1)$ Å
 $b = 13.546(2)$ Å
 $c = 13.433(2)$ Å
 $\beta = 107.247(2)^\circ$
 $V = 1395.5(3)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.48$ mm⁻¹
 $T = 298$ K
 $0.20 \times 0.20 \times 0.18$ mm

Data collection

 Bruker SMART CCD area-detector
 diffractometer
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.911$, $T_{\max} = 0.919$
 8463 measured reflections
 3039 independent reflections
 2324 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.023$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.107$
 $S = 1.03$
 3039 reflections
 195 parameters
 1 restraint
 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.22$ e Å⁻³
 $\Delta\rho_{\min} = -0.36$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1}\cdots\text{N1}$	0.82	1.90	2.6164 (19)	145
$\text{O3}-\text{H3}\cdots\text{O2}^{\ddagger}$	0.82	1.85	2.658 (2)	168
$\text{N2}-\text{H2}\cdots\text{O3}^{\text{ii}}$	0.899 (10)	2.204 (16)	3.000 (2)	147 (2)

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

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.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2214).

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supplementary materials

Acta Cryst. (2009). E65, o1503-o1504 [doi:10.1107/S1600536809020820]

4-Hydroxy-*N'*-(3,5-dichloro-2-hydroxybenzylidene)benzohydrazide

C.-G. Ren

Comment

The Schiff base compounds show excellent biological properties (Jeewoth *et al.*, 1999; Ren *et al.*, 2002; Eltayeb *et al.*, 2008; Sinha *et al.*, 2008). Moreover, the Schiff base compounds have been widely used as versatile ligands in coordination chemistry (Shivakumar *et al.*, 2008; Prabhakaran *et al.*, 2006; Dhar *et al.*, 2005). We report here the crystal structure of the title compound. In the title compound, Fig. 1, the dihedral angle between the two benzene rings is 5.1 (2)°. All the bond lengths are within normal values (Allen *et al.*, 1987) and comparable to those in other similar compounds (Cui *et al.*, 2007; Jing *et al.*, 2007; Ma *et al.*, 2008; Salhin *et al.*, 2007; Lin *et al.*, 2007; Alhadi *et al.*, 2008; Xue *et al.*, 2008; Wang *et al.*, 2008; Lu, 2008; Diao *et al.*, 2008; Qiu, 2009; Mohd Lair *et al.*, 2009a,b). —H···N OIn the crystal structure, molecules are linked through intermolecular N—H···O and O—H···O hydrogen bonds (Table 1), Fig. 2.

Experimental

All the starting materials were obtained with AR grade from Lancaster. 3,5-Dichloro-2-hydroxybenzaldehyde (1.0 mmol, 192.2 mg) and 4-hydroxybenzohydrazide (1.0 mmol, 152.2 mg) were refluxed in 30 ml methanol solution for 30 min giving a clear yellow solution. Yellow block-shaped single crystals of the compound were obtained by slow evaporation of the solution for a week at room temperature.

Refinement

H2 was located from a difference Fourier map and refined isotropically, with the N—H distance restrained to 0.90 (1) Å, and with U_{iso} restrained to 0.08 Å². Other H atoms were constrained to ideal geometries, with $d(\text{C—H}) = 0.93$ Å, $d(\text{O—H}) = 0.82$ Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ and $1.5U_{\text{eq}}(\text{O})$.

Figures

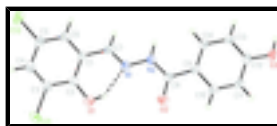


Fig. 1. The molecular structure of the compound with 30% probability ellipsoids. The intramolecular O—H···N hydrogen bond is shown as a dashed line.

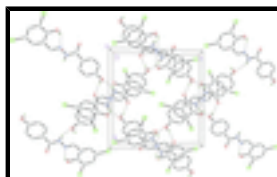


Fig. 2. Molecular packing of the compound with hydrogen bonds drawn as dashed lines. Hydrogen atoms not involved in hydrogen bonding have been omitted.

(I)

Crystal data

$C_{14}H_{10}Cl_2N_2O_3$

$M_r = 325.14$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 8.030$ (1) Å

$b = 13.546$ (2) Å

$c = 13.433$ (2) Å

$\beta = 107.247$ (2)°

$V = 1395.5$ (3) Å³

$Z = 4$

$F_{000} = 664$

$D_x = 1.547$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 2634 reflections

$\theta = 2.2$ – 26.0 °

$\mu = 0.48$ mm⁻¹

$T = 298$ K

Block, yellow

$0.20 \times 0.20 \times 0.18$ 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)

$T_{\min} = 0.911$, $T_{\max} = 0.919$

8463 measured reflections

3039 independent reflections

2324 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.023$

$\theta_{\text{max}} = 27.0$ °

$\theta_{\text{min}} = 2.2$ °

$h = -10 \rightarrow 9$

$k = -17 \rightarrow 17$

$l = -17 \rightarrow 14$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.039$

$wR(F^2) = 0.107$

$S = 1.03$

3039 reflections

195 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

$$w = 1/[\sigma^2(F_o^2) + (0.0503P)^2 + 0.3954P]$$

where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\text{max}} = 0.22$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.36$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	-0.05205 (8)	0.19042 (4)	1.13049 (5)	0.0682 (2)
C12	0.17799 (11)	0.43904 (5)	1.45283 (5)	0.0843 (2)
N1	0.25047 (18)	0.49746 (11)	1.00303 (11)	0.0382 (3)
N2	0.31078 (19)	0.56552 (10)	0.94624 (11)	0.0387 (3)
O1	0.08301 (19)	0.33479 (10)	1.01678 (10)	0.0512 (3)
H1	0.1314	0.3745	0.9885	0.077*
O2	0.26379 (19)	0.45814 (10)	0.81320 (10)	0.0540 (4)
O3	0.55673 (19)	0.80001 (10)	0.59831 (10)	0.0512 (3)
H3	0.6200	0.8433	0.6322	0.077*
C1	0.1834 (2)	0.45241 (12)	1.15726 (13)	0.0369 (4)
C2	0.1046 (2)	0.36288 (13)	1.11609 (14)	0.0384 (4)
C3	0.0458 (2)	0.30054 (13)	1.18096 (15)	0.0422 (4)
C4	0.0667 (2)	0.32330 (14)	1.28362 (15)	0.0467 (5)
H4	0.0275	0.2804	1.3259	0.056*
C5	0.1470 (3)	0.41115 (15)	1.32275 (14)	0.0479 (5)
C6	0.2037 (2)	0.47558 (14)	1.26095 (14)	0.0436 (4)
H6	0.2558	0.5348	1.2885	0.052*
C7	0.2478 (2)	0.52136 (13)	1.09426 (13)	0.0388 (4)
H7	0.2873	0.5833	1.1208	0.047*
C8	0.3119 (2)	0.54031 (13)	0.84892 (13)	0.0366 (4)
C9	0.3736 (2)	0.61545 (12)	0.78824 (13)	0.0354 (4)
C10	0.3425 (3)	0.59603 (15)	0.68236 (14)	0.0468 (4)
H10	0.2799	0.5400	0.6532	0.056*
C11	0.4035 (3)	0.65890 (16)	0.62059 (14)	0.0497 (5)
H11	0.3815	0.6454	0.5500	0.060*
C12	0.4974 (2)	0.74196 (13)	0.66340 (13)	0.0402 (4)
C13	0.5276 (2)	0.76339 (13)	0.76825 (13)	0.0404 (4)
H13	0.5893	0.8199	0.7969	0.049*
C14	0.4656 (2)	0.70024 (13)	0.82994 (13)	0.0383 (4)
H14	0.4856	0.7147	0.9002	0.046*
H2	0.352 (3)	0.6232 (11)	0.9766 (18)	0.080*

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0753 (4)	0.0500 (3)	0.0806 (4)	-0.0224 (3)	0.0252 (3)	-0.0019 (3)
C12	0.1359 (6)	0.0822 (5)	0.0491 (3)	-0.0081 (4)	0.0492 (4)	-0.0052 (3)
N1	0.0398 (8)	0.0373 (7)	0.0391 (8)	0.0010 (6)	0.0143 (6)	0.0045 (6)
N2	0.0462 (8)	0.0364 (8)	0.0361 (8)	-0.0036 (6)	0.0165 (7)	0.0022 (6)
O1	0.0629 (9)	0.0480 (8)	0.0433 (7)	-0.0110 (6)	0.0165 (6)	-0.0057 (6)
O2	0.0730 (10)	0.0428 (7)	0.0462 (8)	-0.0120 (7)	0.0174 (7)	-0.0068 (6)
O3	0.0679 (10)	0.0503 (8)	0.0392 (7)	0.0019 (6)	0.0218 (7)	0.0104 (6)
C1	0.0357 (9)	0.0358 (9)	0.0422 (9)	0.0033 (7)	0.0160 (7)	0.0027 (7)
C2	0.0340 (9)	0.0394 (9)	0.0421 (9)	0.0042 (7)	0.0117 (7)	0.0021 (7)
C3	0.0347 (9)	0.0383 (9)	0.0543 (11)	0.0006 (7)	0.0140 (8)	0.0040 (8)
C4	0.0438 (10)	0.0478 (11)	0.0553 (11)	0.0066 (8)	0.0249 (9)	0.0130 (9)
C5	0.0563 (12)	0.0525 (11)	0.0413 (10)	0.0068 (9)	0.0241 (9)	0.0032 (8)
C6	0.0500 (11)	0.0404 (9)	0.0452 (10)	0.0000 (8)	0.0212 (9)	-0.0030 (8)
C7	0.0399 (9)	0.0355 (9)	0.0427 (10)	-0.0012 (7)	0.0147 (8)	-0.0003 (7)
C8	0.0359 (9)	0.0382 (9)	0.0343 (9)	0.0031 (7)	0.0080 (7)	0.0008 (7)
C9	0.0345 (9)	0.0392 (9)	0.0326 (8)	0.0059 (7)	0.0102 (7)	0.0010 (7)
C10	0.0527 (11)	0.0510 (11)	0.0347 (9)	-0.0067 (9)	0.0098 (8)	-0.0043 (8)
C11	0.0588 (12)	0.0615 (12)	0.0276 (9)	-0.0009 (10)	0.0107 (8)	-0.0009 (8)
C12	0.0453 (10)	0.0419 (10)	0.0351 (9)	0.0116 (8)	0.0144 (7)	0.0106 (7)
C13	0.0468 (10)	0.0376 (9)	0.0373 (9)	0.0016 (8)	0.0132 (8)	0.0003 (7)
C14	0.0453 (10)	0.0412 (9)	0.0299 (8)	0.0037 (7)	0.0135 (7)	-0.0014 (7)

Geometric parameters (\AA , $^\circ$)

C11—C3	1.7281 (19)	C4—C5	1.381 (3)
C12—C5	1.7321 (19)	C4—H4	0.9300
N1—C7	1.274 (2)	C5—C6	1.372 (3)
N1—N2	1.3732 (19)	C6—H6	0.9300
N2—C8	1.354 (2)	C7—H7	0.9300
N2—H2	0.899 (10)	C8—C9	1.478 (2)
O1—C2	1.348 (2)	C9—C14	1.390 (2)
O1—H1	0.8200	C9—C10	1.394 (2)
O2—C8	1.228 (2)	C10—C11	1.376 (3)
O3—C12	1.363 (2)	C10—H10	0.9300
O3—H3	0.8200	C11—C12	1.381 (3)
C1—C6	1.389 (2)	C11—H11	0.9300
C1—C2	1.403 (2)	C12—C13	1.387 (2)
C1—C7	1.455 (2)	C13—C14	1.383 (2)
C2—C3	1.393 (2)	C13—H13	0.9300
C3—C4	1.374 (3)	C14—H14	0.9300
C7—N1—N2	118.23 (15)	N1—C7—C1	120.45 (16)
C8—N2—N1	118.12 (14)	N1—C7—H7	119.8
C8—N2—H2	123.3 (16)	C1—C7—H7	119.8
N1—N2—H2	118.5 (16)	O2—C8—N2	120.98 (16)

C2—O1—H1	109.5	O2—C8—C9	121.77 (15)
C12—O3—H3	109.5	N2—C8—C9	117.26 (15)
C6—C1—C2	119.72 (16)	C14—C9—C10	118.68 (16)
C6—C1—C7	118.80 (16)	C14—C9—C8	124.85 (15)
C2—C1—C7	121.47 (15)	C10—C9—C8	116.40 (16)
O1—C2—C3	118.68 (16)	C11—C10—C9	120.67 (18)
O1—C2—C1	123.01 (16)	C11—C10—H10	119.7
C3—C2—C1	118.30 (16)	C9—C10—H10	119.7
C4—C3—C2	121.93 (17)	C10—C11—C12	120.06 (16)
C4—C3—C11	119.57 (14)	C10—C11—H11	120.0
C2—C3—C11	118.49 (15)	C12—C11—H11	120.0
C3—C4—C5	118.66 (17)	O3—C12—C11	117.03 (15)
C3—C4—H4	120.7	O3—C12—C13	122.78 (17)
C5—C4—H4	120.7	C11—C12—C13	120.19 (16)
C6—C5—C4	121.27 (17)	C14—C13—C12	119.56 (17)
C6—C5—C12	119.84 (16)	C14—C13—H13	120.2
C4—C5—C12	118.89 (15)	C12—C13—H13	120.2
C5—C6—C1	120.10 (17)	C13—C14—C9	120.82 (15)
C5—C6—H6	120.0	C13—C14—H14	119.6
C1—C6—H6	120.0	C9—C14—H14	119.6
C7—N1—N2—C8	-179.36 (15)	C6—C1—C7—N1	171.54 (16)
C6—C1—C2—O1	-178.94 (16)	C2—C1—C7—N1	-7.3 (3)
C7—C1—C2—O1	-0.1 (3)	N1—N2—C8—O2	-1.5 (2)
C6—C1—C2—C3	1.3 (2)	N1—N2—C8—C9	178.67 (14)
C7—C1—C2—C3	-179.91 (16)	O2—C8—C9—C14	-164.75 (17)
O1—C2—C3—C4	178.59 (17)	N2—C8—C9—C14	15.1 (2)
C1—C2—C3—C4	-1.6 (3)	O2—C8—C9—C10	12.3 (2)
O1—C2—C3—C11	0.0 (2)	N2—C8—C9—C10	-167.92 (16)
C1—C2—C3—C11	179.75 (13)	C14—C9—C10—C11	0.8 (3)
C2—C3—C4—C5	0.6 (3)	C8—C9—C10—C11	-176.42 (17)
C11—C3—C4—C5	179.25 (14)	C9—C10—C11—C12	0.3 (3)
C3—C4—C5—C6	0.7 (3)	C10—C11—C12—O3	178.78 (17)
C3—C4—C5—C12	-178.61 (15)	C10—C11—C12—C13	-1.3 (3)
C4—C5—C6—C1	-1.0 (3)	O3—C12—C13—C14	-179.03 (16)
C12—C5—C6—C1	178.29 (14)	C11—C12—C13—C14	1.0 (3)
C2—C1—C6—C5	0.0 (3)	C12—C13—C14—C9	0.1 (3)
C7—C1—C6—C5	-178.85 (16)	C10—C9—C14—C13	-1.0 (3)
N2—N1—C7—C1	179.32 (14)	C8—C9—C14—C13	175.92 (16)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O1—H1 \cdots N1	0.82	1.90	2.6164 (19)	145
O3—H3 \cdots O2 ⁱ	0.82	1.85	2.658 (2)	168
N2—H2 \cdots O3 ⁱⁱ	0.899 (10)	2.204 (16)	3.000 (2)	147 (2)

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

Fig. 1

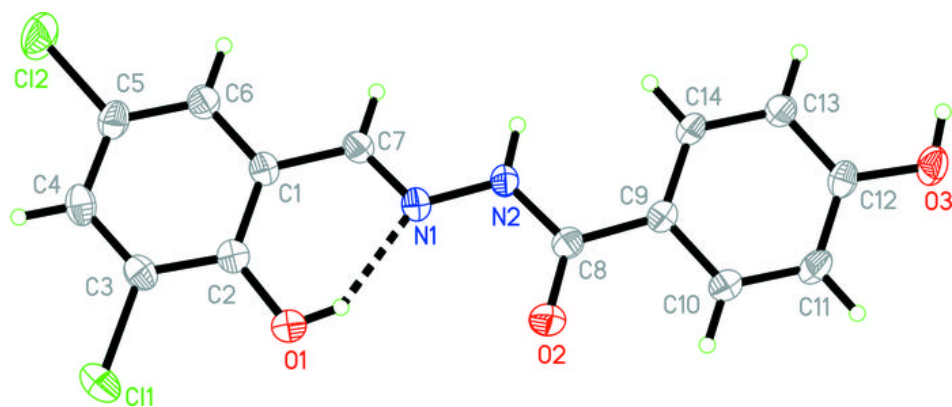


Fig. 2

