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(*E*)-6-Chloro-*N*′-(3,5-dichloro-2-hydroxybenzylidene)nicotinohydrazide

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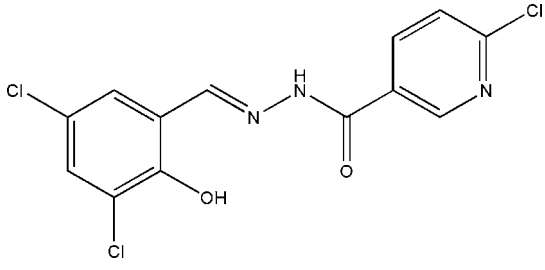
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.006 Å; R factor = 0.049; wR factor = 0.114; data-to-parameter ratio = 13.0.

The title Schiff base compound, $C_{13}H_8Cl_3N_3O_2$, was synthesized by the condensation reaction of 3,5-dichlorosalicylaldehyde with 6-chloronicotinic acid hydrazide in 95% ethanol. The molecule is nearly planar, with a dihedral angle of 1.9 (2)° between the aromatic ring planes, and an intramolecular O—H···N hydrogen bond is observed. In the crystal, the molecules are connected by intermolecular N—H···O hydrogen bonds into infinite chains propagating in [100].

Related literature

For general background, see: Kim *et al.* (2005); Fan *et al.* (2007). For background on the biological activities of Schiff bases, see: Ren *et al.* (2002); Takeuchi *et al.* (1998). For a related structure, see: Zhi (2008). For reference structural data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{13}H_8Cl_3N_3O_2$
M_r = 344.57
Monoclinic, $P2_1/c$
a = 4.8920 (10) Å
b = 18.014 (4) Å
c = 16.112 (3) Å
β = 97.90 (3)°
V = 1406.4 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.66 mm^{-1}
T = 298 K
0.27 × 0.23 × 0.23 mm

Data collection

Siemens SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Siemens, 1996)
T_{min} = 0.842, T_{max} = 0.864
7275 measured reflections
2478 independent reflections
1323 reflections with $I > 2\sigma(I)$
R_{int} = 0.078

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.114
S = 1.01
2478 reflections
191 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O2^{i}	0.86	2.20	2.930 (4)	142
O1—H1···N1	0.82	1.82	2.540 (4)	147

Symmetry code: (i) $x+1, y, z$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); 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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2920).

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supplementary materials

Acta Cryst. (2009). E**65**, o678 [**doi:10.1107/S1600536809007272**]

(*E*)-6-Chloro-*N*'-(3,5-dichloro-2-hydroxybenzylidene)nicotinohydrazide

C. Ren

Comment

Schiff base compounds have been widely investigated over a century (Fan *et al.*, 2007; Kim *et al.*, 2005). Some of the complexes derived from Schiff bases have been found to have pharmacological and antitumor properties (Ren *et al.*, 2002; Takeuchi,*et al.*, 1998). In this paper, the crystal structure of the title compound, (I), a new Schiff base compound derived from the condensation reaction of 3,5-dichlorosalicylaldehyde with 6-chloronicotinic acid hydrazide is reported.

The molecule of (I) displays a *trans* configuration with respect to the C=N and C—N bonds (Fig. 1). All the bond lengths are within normal ranges (Allen *et al.*, 1987), and are comparable to those in the related compound 6-chloro-*N*′-(2-hydroxy-1-naphthylmethylene)nicotinohydrazide (Zhi 2008). The Schiff base molecule is nearly planar, with a dihedral angle between the benzene ring and the pyridine ring of 1.9 (2)°. An intramolecular O—H···N hydrogen bond is observed. The molecules are connected *via* intermolecular N—H···O hydrogen bonds into infinite chains along the *a* axis (Table 1, Fig. 2).

Experimental

3,5-Dichlorosalicylaldehyde (0.1 mmol, 19.0 mg) and 6-chloronicotinic acid hydrazide (0.1 mmol, 17.1 mg) were dissolved in a 95% ethanol solution (10 ml). The mixture was stirred at room temperature to give a clear colorless solution. Light yellow blocks of (I) were formed by gradual evaporation of the solvent over a period of five days at room temperature.

Refinement

All H atoms were placed in geometrically idealized positions, with C—H = 0.93 Å, O—H = 0.82 Å and N—H = 0.86 Å and refined as riding with U_{iso}(H) = 1.2U_{eq}(C,*N*) or 1.5U_{eq}(O).

Figures

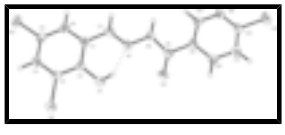

Fig. 1. The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level. The dashed lines indicate hydrogen bonds.

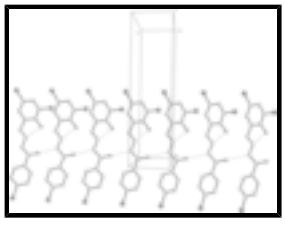

Fig. 2. The infinite chains structure formed *via* hydrogen bonds, H atoms have been omitted for clarity. The dashed lines indicate the connections between the donor and acceptor atoms of the hydrogen bonds.

(*E*)-6-Chloro-*N*′-(3,5-dichloro-2- hydroxybenzylidene)nicotinohydrazide

Crystal data

$C_{13}H_8Cl_3N_3O_2$	F_{000} = 696
M_r = 344.57	D_x = 1.627 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 669 reflections
a = 4.8920 (10) Å	θ = 2.6–18.8º
b = 18.014 (4) Å	μ = 0.66 mm^{-1}
c = 16.112 (3) Å	T = 298 K
β = 97.90 (3)º	Block, light yellow
V = 1406.4 (5) Å^3	0.27 × 0.23 × 0.23 mm
Z = 4	

Data collection

Siemens SMART CCD diffractometer	2478 independent reflections
Radiation source: fine-focus sealed tube	1323 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.078
T = 298 K	θ_{max} = 25.1º
φ and ω scans	θ_{min} = 1.7º
Absorption correction: multi-scan (SADABS; Siemens, 1996)	h = −5→5
T_{min} = 0.842, T_{max} = 0.864	k = −15→21
7275 measured reflections	l = −19→15

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.049	H-atom parameters constrained
$wR(F^2)$ = 0.114	$w = 1/[\sigma^2(F_o^2) + (0.0343P)^2 + 0.2069P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.01	$(\Delta/\sigma)_{max}$ = 0.001
2478 reflections	$\Delta\rho_{max}$ = 0.25 e Å^{-3}
191 parameters	$\Delta\rho_{min}$ = −0.40 e Å^{-3}
Primary atom site location: structure-invariant direct methods	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_{iso}*/U_{eq}
Cl1	0.3842 (2)	0.62724 (6)	0.45177 (7)	0.0534 (3)
Cl2	1.2166 (3)	0.50424 (7)	0.65897 (7)	0.0651 (4)
Cl3	1.2331 (3)	1.28010 (7)	0.69130 (8)	0.0698 (4)
N1	0.9665 (7)	0.8466 (2)	0.6071 (2)	0.0471 (9)
N2	1.0608 (7)	0.91708 (18)	0.6266 (2)	0.0474 (9)
H2	1.2318	0.9261	0.6437	0.057*
O1	0.6119 (6)	0.76626 (15)	0.51866 (18)	0.0542 (8)
H1	0.6800	0.8046	0.5397	0.081*
O2	0.6214 (6)	0.95721 (16)	0.59872 (19)	0.0619 (9)
C1	1.0059 (8)	0.7165 (2)	0.6081 (2)	0.0408 (10)
C2	0.7633 (8)	0.7076 (2)	0.5502 (2)	0.0399 (10)
C3	0.6750 (8)	0.6374 (2)	0.5262 (2)	0.0411 (11)
C4	0.8115 (8)	0.5751 (2)	0.5595 (3)	0.0455 (11)
H4	0.7479	0.5280	0.5428	0.055*
C5	1.0456 (9)	0.5834 (2)	0.6182 (3)	0.0461 (11)
C6	1.1444 (9)	0.6532 (2)	0.6417 (3)	0.0450 (11)
H6	1.3036	0.6581	0.6801	0.054*
C7	1.1106 (9)	0.7906 (3)	0.6314 (2)	0.0452 (11)
H7	1.2817	0.7964	0.6639	0.054*
C8	0.8673 (9)	0.9716 (2)	0.6171 (3)	0.0450 (11)
C9	0.9672 (8)	1.0483 (2)	0.6324 (3)	0.0410 (10)
C10	0.8110 (9)	1.1062 (2)	0.5936 (3)	0.0506 (12)
H10	0.6519	1.0962	0.5566	0.061*
C11	0.8935 (9)	1.1786 (3)	0.6102 (3)	0.0543 (12)
H11	0.7949	1.2183	0.5842	0.065*
C12	1.1270 (9)	1.1895 (2)	0.6666 (3)	0.0477 (12)
N3	1.2807 (7)	1.1365 (2)	0.7059 (2)	0.0502 (10)
C13	1.1988 (9)	1.0670 (2)	0.6874 (3)	0.0493 (11)
H13	1.3051	1.0285	0.7133	0.059*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0452 (7)	0.0496 (7)	0.0632 (8)	−0.0075 (6)	0.0002 (5)	−0.0027 (6)
Cl2	0.0715 (9)	0.0529 (8)	0.0676 (8)	0.0141 (7)	−0.0015 (6)	0.0027 (6)
Cl3	0.0842 (10)	0.0520 (8)	0.0722 (9)	−0.0184 (7)	0.0071 (7)	−0.0060 (7)
N1	0.040 (2)	0.040 (2)	0.062 (3)	−0.0058 (19)	0.0091 (18)	−0.0038 (19)
N2	0.036 (2)	0.037 (2)	0.067 (3)	−0.0058 (19)	−0.0010 (18)	−0.0074 (19)
O1	0.0454 (19)	0.045 (2)	0.069 (2)	−0.0041 (16)	−0.0032 (15)	−0.0010 (16)
O2	0.0317 (19)	0.060 (2)	0.092 (2)	−0.0082 (16)	0.0027 (16)	−0.0115 (18)
C1	0.030 (2)	0.045 (3)	0.048 (3)	−0.004 (2)	0.010 (2)	−0.005 (2)
C2	0.034 (3)	0.035 (3)	0.050 (3)	−0.002 (2)	0.005 (2)	−0.001 (2)
C3	0.030 (2)	0.049 (3)	0.043 (3)	−0.002 (2)	0.0032 (19)	0.000 (2)
C4	0.048 (3)	0.038 (3)	0.053 (3)	−0.005 (2)	0.016 (2)	−0.003 (2)
C5	0.048 (3)	0.047 (3)	0.044 (3)	0.006 (2)	0.010 (2)	0.002 (2)
C6	0.041 (3)	0.049 (3)	0.045 (3)	0.005 (2)	0.007 (2)	−0.004 (2)
C7	0.035 (3)	0.053 (3)	0.048 (3)	−0.007 (2)	0.006 (2)	−0.008 (2)
C8	0.040 (3)	0.046 (3)	0.049 (3)	−0.008 (2)	0.006 (2)	−0.005 (2)
C9	0.033 (3)	0.041 (3)	0.049 (3)	−0.005 (2)	0.006 (2)	−0.005 (2)
C10	0.041 (3)	0.053 (3)	0.055 (3)	−0.008 (2)	−0.001 (2)	−0.001 (2)
C11	0.053 (3)	0.048 (3)	0.061 (3)	−0.003 (2)	0.003 (2)	0.006 (2)
C12	0.052 (3)	0.046 (3)	0.047 (3)	−0.015 (2)	0.013 (2)	−0.001 (2)
N3	0.043 (2)	0.048 (3)	0.057 (2)	−0.004 (2)	−0.0001 (18)	−0.006 (2)
C13	0.043 (3)	0.044 (3)	0.061 (3)	−0.002 (2)	0.004 (2)	−0.006 (2)

Geometric parameters (Å, °)

Cl1—C3	1.739 (4)	C4—C5	1.389 (5)
Cl2—C5	1.736 (4)	C4—H4	0.9300
Cl3—C12	1.742 (4)	C5—C6	1.382 (5)
N1—C7	1.263 (5)	C6—H6	0.9300
N1—N2	1.372 (4)	C7—H7	0.9300
N2—C8	1.358 (5)	C8—C9	1.474 (5)
N2—H2	0.8600	C9—C13	1.380 (5)
O1—C2	1.349 (4)	C9—C10	1.391 (5)
O1—H1	0.8200	C10—C11	1.380 (6)
O2—C8	1.227 (5)	C10—H10	0.9300
C1—C6	1.396 (5)	C11—C12	1.373 (6)
C1—C2	1.414 (5)	C11—H11	0.9300
C1—C7	1.460 (5)	C12—N3	1.321 (5)
C2—C3	1.375 (5)	N3—C13	1.337 (5)
C3—C4	1.376 (5)	C13—H13	0.9300
C7—N1—N2	120.9 (4)	N1—C7—C1	119.3 (4)
C8—N2—N1	115.9 (4)	N1—C7—H7	120.3
C8—N2—H2	122.1	C1—C7—H7	120.3
N1—N2—H2	122.1	O2—C8—N2	121.3 (4)
C2—O1—H1	109.5	O2—C8—C9	122.0 (4)

C6—C1—C2	118.8 (4)	N2—C8—C9	116.7 (4)
C6—C1—C7	120.8 (4)	C13—C9—C10	117.2 (4)
C2—C1—C7	120.4 (4)	C13—C9—C8	124.0 (4)
O1—C2—C3	118.6 (4)	C10—C9—C8	118.6 (4)
O1—C2—C1	121.8 (4)	C11—C10—C9	119.6 (4)
C3—C2—C1	119.5 (4)	C11—C10—H10	120.2
C2—C3—C4	121.5 (4)	C9—C10—H10	120.2
C2—C3—Cl1	119.1 (3)	C12—C11—C10	117.3 (4)
C4—C3—Cl1	119.4 (3)	C12—C11—H11	121.4
C3—C4—C5	119.3 (4)	C10—C11—H11	121.4
C3—C4—H4	120.4	N3—C12—C11	125.5 (4)
C5—C4—H4	120.4	N3—C12—Cl3	115.8 (3)
C6—C5—C4	120.6 (4)	C11—C12—Cl3	118.7 (4)
C6—C5—Cl2	120.8 (4)	C12—N3—C13	115.9 (4)
C4—C5—Cl2	118.6 (3)	N3—C13—C9	124.5 (4)
C5—C6—C1	120.3 (4)	N3—C13—H13	117.7
C5—C6—H6	119.9	C9—C13—H13	117.7
C1—C6—H6	119.9		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O2^{i}	0.86	2.20	2.930 (4)	142
O1—H1···N1	0.82	1.82	2.540 (4)	147

Symmetry codes: (i) *x*+1, *y*, *z*.

Fig. 1

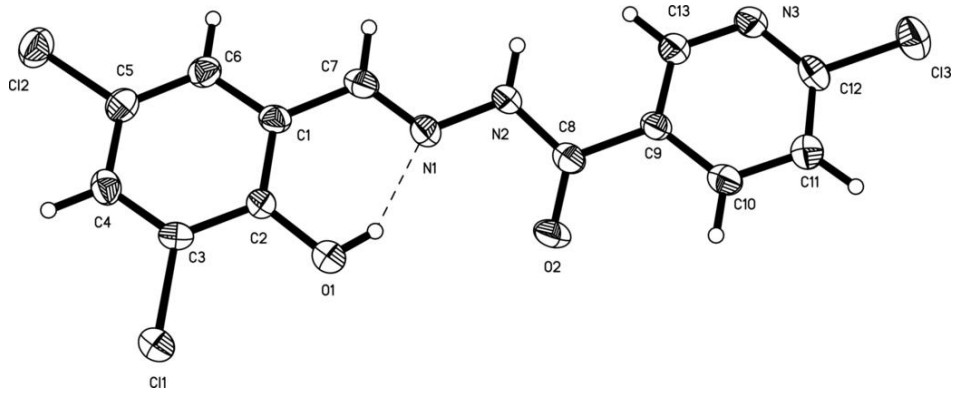

Fig. 2