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6-Chloro-*N*′-(2,4-dichlorobenzylidene)-nicotinohydrazide

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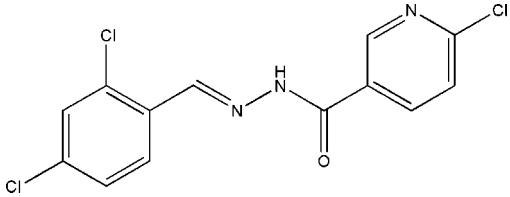
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.009 Å; R factor = 0.086; wR factor = 0.190; data-to-parameter ratio = 15.4.

The title compound, $C_{13}H_8Cl_3N_3O$, was synthesized by the condensation reaction of 2,4-dichlorobenzaldehyde with 6-chloronicotinic acid hydrazide in a methanol solution. The Schiff base molecule displays a *trans* configuration with respect to the C=N and C—N bonds. The dihedral angle between the benzene and pyridine rings is 5.5 (3)°. The crystal structure is stabilized by intermolecular N—H···O, C—H···O and C—H···N hydrogen bonds.

Related literature

For related literature, see: Allen *et al.* (1987); Chen *et al.* (1997); Fan *et al.* (2007); Kim *et al.* (2005); Nimitsiriwat *et al.* (2004); Ren *et al.* (2002).



Experimental

Crystal data

$C_{13}H_8Cl_3N_3O$
M_r = 328.57
Triclinic, $P\overline{1}$
a = 4.6670 (9) Å
b = 12.202 (2) Å
c = 12.935 (3) Å
α = 106.70 (3)°
β = 92.36 (3)°
γ = 96.94 (3)°
V = 698.1 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.65 mm^{-1}
T = 298 (2) K
0.27 × 0.23 × 0.22 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.843, T_{max} = 0.870
5653 measured reflections
2827 independent reflections
1141 reflections with $I > 2\sigma(I)$
R_{int} = 0.081

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.086
$wR(F^2)$ = 0.190
S = 0.97
2827 reflections
184 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1^{i}	0.90 (3)	1.95 (3)	2.816 (6)	160 (6)
C5—H5···O1^{ii}	0.93	2.46	3.291 (6)	148
C13—H13···N3^{iii}	0.93	2.57	3.379 (6)	145

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXL97*.

Financial support from the Third Affiliated Hospital of Suzhou University is acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2327).

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supplementary materials

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6-Chloro-*N*'-(2,4-dichlorobenzylidene)nicotinohydrazide

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Comment

Schiff base compounds have been widely investigated over a century (Fan *et al.*, 2007; Kim *et al.*, 2005; Nimitsiriwat *et al.*, 2004). Some of the compounds have been found to have pharmacological and antibacterial activity (Chen *et al.*, 1997; Ren *et al.*, 2002). In this paper, the crystal structure of a new Schiff base compound derived from the condensation reaction of 2,4-dichlorobenzaldehyde with 6-chloronicotinic acid hydrazide is reported.

The Schiff base molecule of the compound displays a *trans* configuration with respect to the C═N and C—N bonds (Fig. 1). The dihedral angle between the C1—C6 phenyl ring and the C9—C13/N3 pyridine ring is 5.5 (3)°. All the bond lengths are within normal ranges (Allen *et al.*, 1987). The crystal structure is stabilized by intermolecular N–H···O, C–H···O and C–H···N hydrogen bonds (Table 1 and Fig. 2).

Experimental

2,4-Dichlorobenzaldehyde (0.1 mmol, 17.5 mg) and 6-chloronicotinic acid hydrazide (0.1 mmol, 17.1 mg) were dissolved in a methanol solution (10 ml). The mixture was stirred at room temperature to give a clear yellow solution. Crystals of the title compound were formed by gradual evaporation of the solvent for 5 days at room temperature.

Refinement

Atom H2 was located from a difference Fourier map and refined isotropically, with N–H distance restrained to 0.90 (1) Å. Other H atoms were constrained to ideal geometries, with C–H = 0.93 Å, and with U_{iso}(H) set to 1.2U_{eq}(C).

Figures

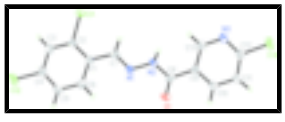

Fig. 1. The structure of (I) at the 30% probability level.

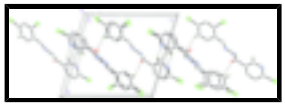

Fig. 2. Molecular packing of (I), viewed along the *b* axis. Intermolecular hydrogen bonds are shown as dashed lines.

6-Chloro-*N*'-(2,4-dichlorobenzylidene)nicotinohydrazide

Crystal data

$C_{13}H_8Cl_3N_3O$	$Z = 2$
$M_r = 328.57$	$F_{000} = 332$

Triclinic, $P\bar{1}$	D_x = 1.563 Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 4.6670 (9) Å	Cell parameters from 872 reflections
b = 12.202 (2) Å	θ = 2.5–24.7º
c = 12.935 (3) Å	μ = 0.65 mm^{-1}
α = 106.70 (3)º	T = 298 (2) K
β = 92.36 (3)º	Block, yellow
γ = 96.94 (3)º	0.27 × 0.23 × 0.22 mm
V = 698.1 (2) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	2827 independent reflections
Radiation source: fine-focus sealed tube	1141 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.081
T = 298(2) K	θ_{max} = 26.5º
ω scans	θ_{min} = 1.7º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −5→5
T_{min} = 0.843, T_{max} = 0.870	k = −15→15
5653 measured reflections	l = −15→16

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.086	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.190	$w = 1/[\sigma^2(F_o^2) + (0.0596P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.97	$(\Delta/\sigma)_{max} < 0.001$
2827 reflections	$\Delta\rho_{max}$ = 0.29 e Å^{-3}
184 parameters	$\Delta\rho_{min}$ = −0.34 e Å^{-3}
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.3082 (4)	0.10650 (15)	0.49252 (14)	0.0547 (6)
Cl2	−0.3745 (5)	0.04955 (17)	0.13214 (15)	0.0752 (7)
Cl3	0.3929 (5)	0.79974 (18)	1.24132 (15)	0.0810 (8)
O1	−0.3459 (9)	0.5648 (4)	0.7653 (3)	0.0499 (13)
N1	−0.0622 (11)	0.4126 (4)	0.6298 (4)	0.0408 (14)
N2	0.0597 (10)	0.4785 (5)	0.7306 (4)	0.0396 (14)
N3	0.3450 (11)	0.6333 (5)	1.0617 (4)	0.0488 (15)
C1	−0.0062 (13)	0.2716 (5)	0.4667 (5)	0.0347 (15)
C2	0.0704 (12)	0.1627 (5)	0.4208 (5)	0.0378 (16)
C3	−0.0399 (15)	0.0942 (6)	0.3192 (5)	0.055 (2)
H3	0.0146	0.0214	0.2908	0.066*
C4	−0.2311 (15)	0.1346 (6)	0.2602 (5)	0.0475 (18)
C5	−0.3120 (14)	0.2426 (6)	0.3028 (5)	0.0504 (19)
H5	−0.4419	0.2696	0.2628	0.060*
C6	−0.2010 (13)	0.3109 (5)	0.4046 (5)	0.0452 (18)
H6	−0.2560	0.3837	0.4324	0.054*
C7	0.1035 (14)	0.3441 (6)	0.5761 (5)	0.0439 (18)
H7	0.2863	0.3405	0.6053	0.053*
C8	−0.1012 (14)	0.5535 (5)	0.7941 (5)	0.0390 (17)
C9	0.0401 (13)	0.6207 (6)	0.9038 (5)	0.0382 (16)
C10	−0.0519 (14)	0.7242 (6)	0.9563 (5)	0.0504 (19)
H10	−0.1877	0.7541	0.9215	0.061*
C11	0.0586 (15)	0.7831 (6)	1.0609 (5)	0.056 (2)
H11	0.0034	0.8537	1.0979	0.067*
C12	0.2527 (15)	0.7327 (6)	1.1074 (5)	0.0494 (19)
C13	0.2408 (13)	0.5797 (6)	0.9598 (5)	0.0445 (18)
H13	0.3066	0.5108	0.9243	0.053*
H2	0.251 (4)	0.493 (6)	0.750 (5)	0.080*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0553 (12)	0.0540 (12)	0.0534 (12)	0.0200 (9)	−0.0092 (9)	0.0104 (9)
Cl2	0.1053 (17)	0.0664 (14)	0.0410 (12)	0.0000 (12)	−0.0214 (11)	0.0042 (10)
Cl3	0.1154 (18)	0.0737 (15)	0.0401 (12)	0.0009 (13)	−0.0198 (12)	0.0035 (11)
O1	0.029 (3)	0.067 (3)	0.051 (3)	0.014 (2)	−0.003 (2)	0.010 (3)
N1	0.037 (3)	0.040 (3)	0.038 (3)	−0.006 (3)	−0.010 (3)	0.006 (3)
N2	0.029 (3)	0.045 (3)	0.038 (3)	0.009 (3)	−0.008 (3)	0.002 (3)
N3	0.054 (4)	0.053 (4)	0.037 (4)	0.008 (3)	−0.002 (3)	0.011 (3)

C1	0.033 (4)	0.042 (4)	0.031 (4)	0.003 (3)	0.005 (3)	0.015 (3)
C2	0.032 (4)	0.039 (4)	0.040 (4)	0.007 (3)	−0.007 (3)	0.009 (3)
C3	0.069 (5)	0.049 (5)	0.045 (5)	0.008 (4)	0.001 (4)	0.011 (4)
C4	0.059 (5)	0.040 (4)	0.036 (4)	−0.007 (4)	−0.009 (4)	0.007 (3)
C5	0.057 (5)	0.049 (5)	0.048 (5)	0.004 (4)	−0.015 (4)	0.022 (4)
C6	0.052 (4)	0.032 (4)	0.047 (5)	0.003 (3)	−0.002 (4)	0.007 (3)
C7	0.033 (4)	0.044 (4)	0.051 (5)	0.006 (3)	−0.001 (3)	0.008 (4)
C8	0.031 (4)	0.033 (4)	0.052 (5)	0.006 (3)	0.013 (3)	0.009 (3)
C9	0.034 (4)	0.040 (4)	0.036 (4)	0.003 (3)	−0.002 (3)	0.005 (3)
C10	0.046 (4)	0.054 (5)	0.050 (5)	0.018 (4)	−0.010 (4)	0.010 (4)
C11	0.065 (5)	0.050 (5)	0.042 (5)	0.014 (4)	−0.005 (4)	−0.004 (4)
C12	0.067 (5)	0.042 (5)	0.036 (4)	0.001 (4)	0.000 (4)	0.008 (4)
C13	0.045 (4)	0.043 (4)	0.045 (5)	0.004 (4)	0.003 (3)	0.014 (4)

Geometric parameters (Å, °)

Cl1—C2	1.740 (6)	C3—H3	0.9300
Cl2—C4	1.739 (6)	C4—C5	1.380 (9)
Cl3—C12	1.750 (7)	C5—C6	1.380 (8)
O1—C8	1.223 (7)	C5—H5	0.9300
N1—C7	1.283 (7)	C6—H6	0.9300
N1—N2	1.377 (6)	C7—H7	0.9300
N2—C8	1.364 (7)	C8—C9	1.498 (8)
N2—H2	0.90 (3)	C9—C10	1.379 (8)
N3—C12	1.319 (8)	C9—C13	1.386 (8)
N3—C13	1.332 (7)	C10—C11	1.380 (8)
C1—C2	1.388 (8)	C10—H10	0.9300
C1—C6	1.403 (8)	C11—C12	1.365 (9)
C1—C7	1.469 (8)	C11—H11	0.9300
C2—C3	1.378 (8)	C13—H13	0.9300
C3—C4	1.373 (8)		
C7—N1—N2	113.6 (5)	C1—C6—H6	119.6
C8—N2—N1	117.9 (5)	N1—C7—C1	117.5 (6)
C8—N2—H2	115 (5)	N1—C7—H7	121.2
N1—N2—H2	124 (4)	C1—C7—H7	121.2
C12—N3—C13	115.9 (6)	O1—C8—N2	123.0 (6)
C2—C1—C6	117.1 (6)	O1—C8—C9	121.6 (6)
C2—C1—C7	122.9 (6)	N2—C8—C9	115.4 (6)
C6—C1—C7	120.0 (6)	C10—C9—C13	117.7 (6)
C3—C2—C1	122.3 (6)	C10—C9—C8	118.7 (6)
C3—C2—Cl1	117.5 (5)	C13—C9—C8	123.4 (6)
C1—C2—Cl1	120.2 (5)	C9—C10—C11	119.7 (6)
C4—C3—C2	119.4 (7)	C9—C10—H10	120.2
C4—C3—H3	120.3	C11—C10—H10	120.2
C2—C3—H3	120.3	C12—C11—C10	116.8 (7)
C3—C4—C5	120.1 (6)	C12—C11—H11	121.6
C3—C4—Cl2	120.3 (6)	C10—C11—H11	121.6
C5—C4—Cl2	119.7 (6)	N3—C12—C11	126.1 (6)
C6—C5—C4	120.3 (6)	N3—C12—Cl3	114.4 (5)

C6—C5—H5	119.9	C11—C12—Cl3	119.5 (6)
C4—C5—H5	119.9	N3—C13—C9	123.8 (6)
C5—C6—C1	120.8 (6)	N3—C13—H13	118.1
C5—C6—H6	119.6	C9—C13—H13	118.1

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1^{i}	0.90 (3)	1.95 (3)	2.816 (6)	160 (6)
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Fig. 1

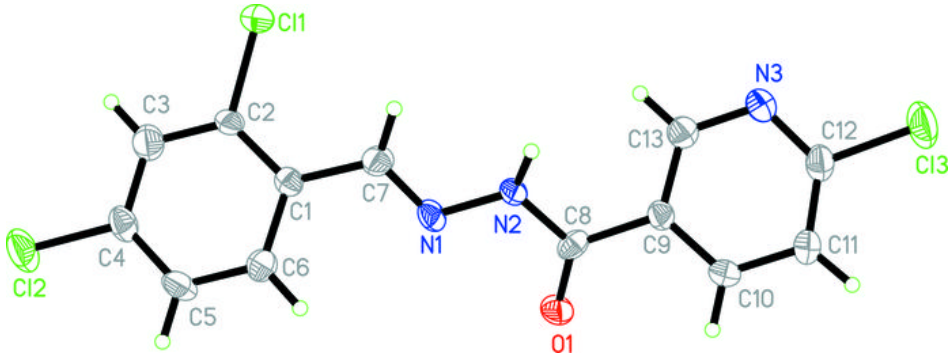

Fig. 2