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N′-(3,4-Dichlorobenzylidene)-isonicotinohydrazide

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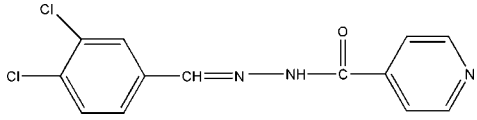
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; R factor = 0.038; wR factor = 0.109; data-to-parameter ratio = 16.3.

The molecule of the title compound, $C_{13}H_9Cl_2N_3O$, is roughly planar, the largest deviation from the mean plane being 0.168 (1) Å at the N atom next to the carbonyl group. Molecules are linked by N—H···N hydrogen bonds to form a zigzag chain.

Related literature

For related literature, see: Kahwa *et al.* (1986); Santos *et al.* (2001).



Experimental

Crystal data

$C_{13}H_9Cl_2N_3O$
M_r = 294.13
Monoclinic, $P2_1/n$
a = 8.2208 (5) Å
b = 10.9657 (6) Å
c = 14.5206 (8) Å
β = 101.881 (1)°
V = 1280.94 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.50 mm^{-1}
T = 298 (2) K
0.23 × 0.22 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.894, T_{max} = 0.907
15485 measured reflections
2797 independent reflections
2424 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.109
S = 1.07
2797 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.51 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···N3^{i}	0.86	2.28	3.0859 (18)	156

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); 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: DN2248).

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supplementary materials

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N'-(3,4-Dichlorobenzylidene)isonicotinohydrazide

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Comment

The chemistry of Schiff bases has attracted much interest in recent years. These compounds play an important role in the development of various proteins and enzymes(Kahwa *et al.*, 1986; Santos *et al.*, 2001). As part of our interest in the study of the coordination chemistry of Schiff bases, we have synthesized the title compound (I) and its cyrstal structure is reported here.

The molecule of the title compound, $C_{13}H_9Cl_2N_3O$, is roughly planar with the largest deviation from the mean plane being 0.168 (1)Å at N2 (Fig. 1). The molecules are linked by N—H···N hydrogen bonds to form a zigzag like chain (Table 1, Fig.2).

Experimental

Pyridine-4-carboxylic acid (1 mmol, 0.137 g) was dissolved in anhydrous methanol, H_2SO_4 (98% 0.5 ml) was added to this, the mixture was stirred for several minitutes at 351 K, 3,4-dichlorobenzyaldehyde (1 mmol 0.175 g) in methanol (8 ml) was added dropwise and the mixture was stirred at refluxing temperature for 2 h. The product was isolated and recrystallized in dichloromethane, brown single crystals of (I) was obtained after 5 d.

Refinement

All H atoms attached to C atoms and N atom were fixed geometrically and treated as riding with C—H = 0.93 Å (aromatic) and N—H =0.86 Å with U_{iso}(H) = 1.2U_{eq}(C or N).

Figures

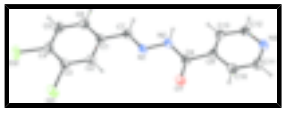

Fig. 1. Molecular view of (I) with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii.

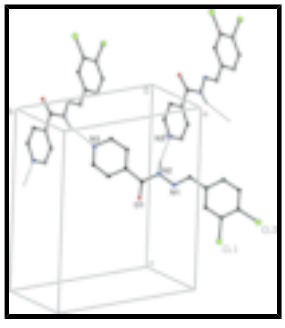

Fig. 2. Partial packing view of compound (I), showing the formation of the zigzag like chain through N—H···N hydrogen bonds. For the sake of clarity, H atoms not involved in hydrogen bonding have been omitted. [symmetry codes: (i) −*x* + 1/2, *y*-/2, −*z* + 1/2].

N'-(3,4-Dichlorobenzylidene)isonicotinohydrazide

Crystal data

$C_{13}H_9Cl_2N_3O$	$F_{000} = 600$
$M_r = 294.13$	$D_x = 1.525$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 1680 reflections
$a = 8.2208$ (5) Å	$\theta = 2.5$–24.1º
$b = 10.9657$ (6) Å	$\mu = 0.50$ mm^{-1}
$c = 14.5206$ (8) Å	$T = 298$ (2) K
$\beta = 101.8810$ (10)º	Block, brown
$V = 1280.94$ (13) Å^3	0.23 × 0.22 × 0.20 mm
$Z = 4$	

Data collection

Bruker SMART CCD area-detector diffractometer	2797 independent reflections
Radiation source: fine-focus sealed tube	2424 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.019$
$T = 298$(2) K	$\theta_{max} = 27.0$º
ω scans	$\theta_{min} = 2.4$º
Absorption correction: multi-scan (SADABS; Bruker, 1998)	$h = -10 \rightarrow 10$
$T_{min} = 0.894$, $T_{max} = 0.907$	$k = -14 \rightarrow 14$
15485 measured reflections	$l = -18 \rightarrow 18$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.038$	H-atom parameters constrained
$wR(F^2) = 0.109$	$w = 1/[\sigma^2(F_o^2) + (0.0562P)^2 + 0.3525P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{max} = 0.001$
2797 reflections	$\Delta\rho_{max} = 0.28$ e Å^{-3}
172 parameters	$\Delta\rho_{min} = -0.51$ 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 $F^2 > 2\text{sigma}(F^2)$ 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	1.09201 (8)	−0.13294 (5)	0.74298 (4)	0.0799 (2)
Cl2	1.19312 (6)	−0.38051 (5)	0.65624 (4)	0.06668 (18)
O1	0.57542 (18)	0.27617 (12)	0.51487 (8)	0.0608 (4)
N1	0.66161 (15)	0.05618 (12)	0.46137 (9)	0.0414 (3)
N2	0.54386 (16)	0.12734 (11)	0.40476 (9)	0.0417 (3)
H2	0.4928	0.1020	0.3504	0.050*
N3	0.17567 (17)	0.47619 (12)	0.25252 (10)	0.0467 (3)
C1	0.82342 (18)	−0.12390 (14)	0.48204 (11)	0.0395 (3)
C2	0.89503 (19)	−0.09540 (14)	0.57476 (11)	0.0423 (3)
H2A	0.8656	−0.0235	0.6012	0.051*
C3	1.00936 (19)	−0.17281 (15)	0.62796 (11)	0.0441 (4)
C4	1.05440 (19)	−0.27991 (15)	0.58971 (12)	0.0459 (4)
C5	0.9860 (2)	−0.30805 (17)	0.49746 (14)	0.0560 (4)
H5	1.0168	−0.3795	0.4711	0.067*
C6	0.8716 (2)	−0.23040 (17)	0.44379 (12)	0.0523 (4)
H6	0.8266	−0.2499	0.3814	0.063*
C7	0.69846 (19)	−0.04360 (15)	0.42635 (11)	0.0425 (3)
H7	0.6463	−0.0656	0.3656	0.051*
C8	0.51087 (19)	0.23852 (14)	0.43734 (10)	0.0416 (3)
C9	0.39034 (18)	0.31667 (13)	0.37003 (10)	0.0376 (3)
C10	0.3369 (2)	0.42349 (15)	0.40426 (11)	0.0450 (4)
H10	0.3703	0.4435	0.4676	0.054*
C11	0.2336 (2)	0.50024 (17)	0.34363 (12)	0.0510 (4)
H11	0.2023	0.5733	0.3675	0.061*
C12	0.2266 (2)	0.37254 (15)	0.22029 (12)	0.0509 (4)
H12	0.1879	0.3531	0.1572	0.061*
C13	0.3341 (2)	0.29202 (14)	0.27565 (11)	0.0492 (4)
H13	0.3683	0.2216	0.2494	0.059*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}

Cl1	0.0908 (4)	0.0782 (4)	0.0522 (3)	0.0102 (3)	−0.0283 (3)	−0.0046 (2)
Cl2	0.0527 (3)	0.0613 (3)	0.0828 (4)	0.0169 (2)	0.0066 (2)	0.0262 (2)
O1	0.0778 (9)	0.0515 (7)	0.0407 (6)	0.0096 (6)	−0.0166 (6)	−0.0042 (5)
N1	0.0412 (7)	0.0397 (7)	0.0383 (6)	0.0008 (5)	−0.0032 (5)	0.0074 (5)
N2	0.0449 (7)	0.0380 (7)	0.0361 (6)	0.0016 (5)	−0.0057 (5)	0.0045 (5)
N3	0.0492 (7)	0.0420 (7)	0.0440 (7)	0.0017 (6)	−0.0019 (6)	0.0076 (6)
C1	0.0390 (7)	0.0387 (8)	0.0389 (8)	−0.0011 (6)	0.0033 (6)	0.0029 (6)
C2	0.0432 (8)	0.0379 (7)	0.0421 (8)	0.0017 (6)	0.0006 (6)	−0.0008 (6)
C3	0.0399 (8)	0.0459 (8)	0.0421 (8)	−0.0032 (6)	−0.0020 (6)	0.0046 (7)
C4	0.0374 (7)	0.0431 (8)	0.0560 (9)	0.0040 (6)	0.0070 (7)	0.0126 (7)
C5	0.0624 (10)	0.0453 (9)	0.0606 (10)	0.0129 (8)	0.0131 (8)	−0.0026 (8)
C6	0.0600 (10)	0.0510 (10)	0.0425 (9)	0.0050 (8)	0.0027 (7)	−0.0058 (7)
C7	0.0425 (8)	0.0448 (8)	0.0363 (7)	−0.0008 (6)	−0.0011 (6)	0.0026 (6)
C8	0.0453 (8)	0.0396 (8)	0.0354 (7)	−0.0016 (6)	−0.0019 (6)	0.0044 (6)
C9	0.0400 (7)	0.0336 (7)	0.0358 (7)	−0.0047 (6)	−0.0002 (6)	0.0053 (6)
C10	0.0523 (9)	0.0458 (8)	0.0341 (7)	0.0045 (7)	0.0023 (6)	0.0002 (6)
C11	0.0583 (10)	0.0476 (9)	0.0455 (9)	0.0117 (8)	0.0073 (7)	0.0012 (7)
C12	0.0669 (11)	0.0391 (8)	0.0375 (8)	−0.0056 (7)	−0.0105 (7)	0.0022 (6)
C13	0.0683 (11)	0.0325 (7)	0.0394 (8)	0.0023 (7)	−0.0061 (7)	−0.0013 (6)

Geometric parameters (Å, °)

Cl1—C3	1.7249 (16)	C4—C5	1.377 (3)
Cl2—C4	1.7312 (16)	C5—C6	1.384 (3)
O1—C8	1.2138 (19)	C5—H5	0.9300
N1—C7	1.269 (2)	C6—H6	0.9300
N1—N2	1.3762 (17)	C7—H7	0.9300
N2—C8	1.355 (2)	C8—C9	1.508 (2)
N2—H2	0.8600	C9—C10	1.379 (2)
N3—C12	1.329 (2)	C9—C13	1.380 (2)
N3—C11	1.337 (2)	C10—C11	1.377 (2)
C1—C6	1.386 (2)	C10—H10	0.9300
C1—C2	1.389 (2)	C11—H11	0.9300
C1—C7	1.464 (2)	C12—C13	1.384 (2)
C2—C3	1.379 (2)	C12—H12	0.9300
C2—H2A	0.9300	C13—H13	0.9300
C3—C4	1.382 (2)		
C7—N1—N2	116.49 (13)	C1—C6—H6	119.7
C8—N2—N1	117.92 (12)	N1—C7—C1	119.70 (14)
C8—N2—H2	121.0	N1—C7—H7	120.1
N1—N2—H2	121.0	C1—C7—H7	120.1
C12—N3—C11	116.30 (14)	O1—C8—N2	123.30 (14)
C6—C1—C2	118.62 (14)	O1—C8—C9	120.76 (14)
C6—C1—C7	120.77 (14)	N2—C8—C9	115.92 (13)
C2—C1—C7	120.61 (14)	C10—C9—C13	117.25 (14)
C3—C2—C1	120.55 (15)	C10—C9—C8	117.69 (13)
C3—C2—H2A	119.7	C13—C9—C8	125.01 (14)
C1—C2—H2A	119.7	C11—C10—C9	119.26 (15)
C2—C3—C4	120.41 (15)	C11—C10—H10	120.4

C2—C3—Cl1	118.27 (13)	C9—C10—H10	120.4
C4—C3—Cl1	121.32 (12)	N3—C11—C10	124.01 (16)
C5—C4—C3	119.46 (15)	N3—C11—H11	118.0
C5—C4—Cl2	119.37 (13)	C10—C11—H11	118.0
C3—C4—Cl2	121.16 (13)	N3—C12—C13	123.43 (15)
C4—C5—C6	120.28 (16)	N3—C12—H12	118.3
C4—C5—H5	119.9	C13—C12—H12	118.3
C6—C5—H5	119.9	C9—C13—C12	119.69 (15)
C5—C6—C1	120.66 (16)	C9—C13—H13	120.2
C5—C6—H6	119.7	C12—C13—H13	120.2

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···N3^{i}	0.86	2.28	3.0859 (18)	156

Symmetry codes: (i) −*x*+1/2, *y*−1/2, −*z*+1/2.

Fig. 1

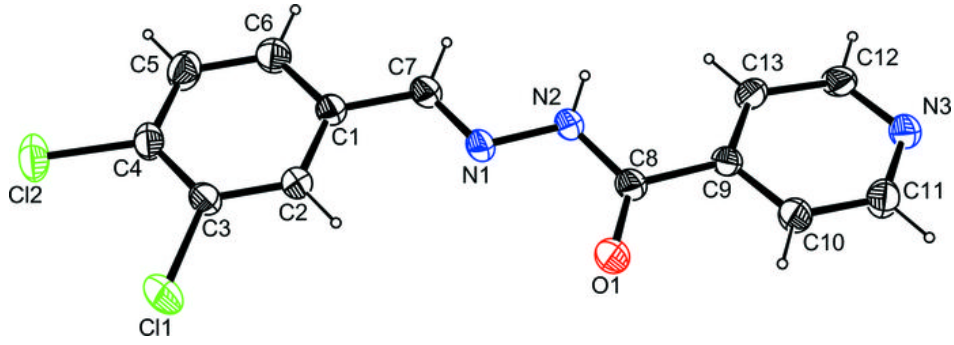

Fig. 2