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(*E*)-*N*′-(3,4-Dichlorobenzylidene)-nicotinohydrazide monohydrate

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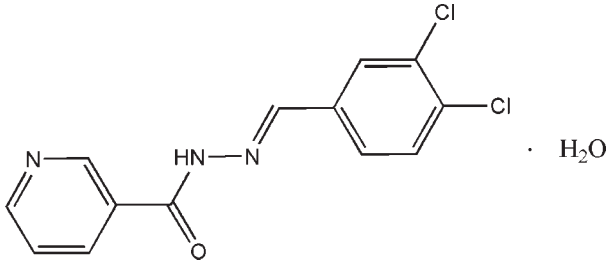
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.036; wR factor = 0.100; data-to-parameter ratio = 16.0.

In the title compound, $C_{13}H_9Cl_2N_3O\cdot H_2O$, the 3,4-dichlorobenzene ring is nearly coplanar with the pyridine ring, making a dihedral angle of 4.78 (8)°. Intermolecular O—H···O, O—H···N, N—H···O and weak C—H···O hydrogen bonding is present in the crystal structure.

Related literature

For applications of Schiff base compounds, see: Kahwa *et al.* (1986); Santos *et al.* (2001).



Experimental

Crystal data

$C_{13}H_9Cl_2N_3O\cdot H_2O$
M_r = 312.15
Monoclinic, $P2_1/c$
a = 8.2080 (3) Å
b = 12.3294 (4) Å
c = 13.7089 (4) Å
β = 91.522 (2)°
V = 1386.85 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.47 mm^{-1}
T = 296 K
0.40 × 0.20 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.893, T_{max} = 0.954
20965 measured reflections
3032 independent reflections
2150 reflections with $I > 2\sigma(I)$
R_{int} = 0.042

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.100
S = 1.02
3032 reflections
189 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···O	0.85 (2)	1.995 (16)	2.8059 (19)	160 (2)
O1—H1B···N3^{i}	0.85 (2)	2.079 (12)	2.909 (2)	166 (2)
N2—H2A···O1^{ii}	0.86	2.00	2.842 (2)	165
C7—H7A···O1^{ii}	0.93	2.55	3.314 (2)	140
C10—H10A···O1^{ii}	0.93	2.39	3.304 (2)	167

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2600).

References

Bruker (1998). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Kahwa, I. A., Selbin, I., Hsieh, T. C. Y. & Laine, R. A. (1986). *Inorg. Chim. Acta*, **118**, 179–185.
Santos, M. L. P., Bagatin, I. A., Pereira, E. M. & Ferreira, A. M. D. C. (2001). *J. Chem. Soc. Dalton Trans.* pp. 838–844.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2009). E**65**, o2336 [doi:10.1107/S1600536809034552]

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S1. Comment

The chemistry of Schiff bases has attracted a great deal of 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 coordination chemistry of Schiff bases, we have synthesized the title compound and report here its crystal structure.

The title molecule crystallizes in the E conformation (Fig. 1), with the N2—N1—C7—C6 torsion angle of 179.81 (15)°. The molecule structure is nearly planar, the dihedral angle between the 3,4-dichlorobenzene ring and the pyridine ring is 4.78 (8)°. The extensive intermolecular classic O—H···O, O—H···N, N—H···O and weak C—H···O hydrogen bonding is present in the crystal structure (Table 1 and Fig. 2).

S2. Experimental

Nicotinohydrazide (1 mmol, 0.137 g) was dissolved in ethanol (15 ml). The solution was stirred for several minitutes at 351 K, then the 3,4-dichlorobenzaldehyde (1 mmol, 0.175 g) in ethanol (8 ml) was added dropwise, and the mixture was stirred at refluxing temperature for 2 h. The solid product was isolated and recrystallized from methanol-water solution. Colourless single crystals were obtained after 3 d.

S3. Refinement

H atoms of water molecule are located in a difference Fourier map and refined isotropically, with O—H and H···H distances restrained to 0.85 (2) and 1.37 (2) Å. Other H atoms were positioned geometrically and refined as riding with C—H = 0.93 (aromatic) and N—H = 0.86 Å, U_{iso}(H) = 1.2U_{eq}(C,*N*).

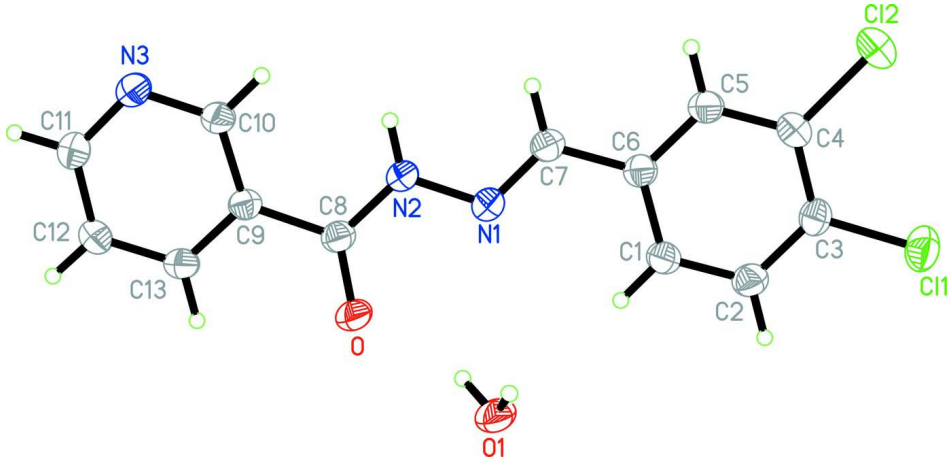

Figure 1

The molecular structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level.

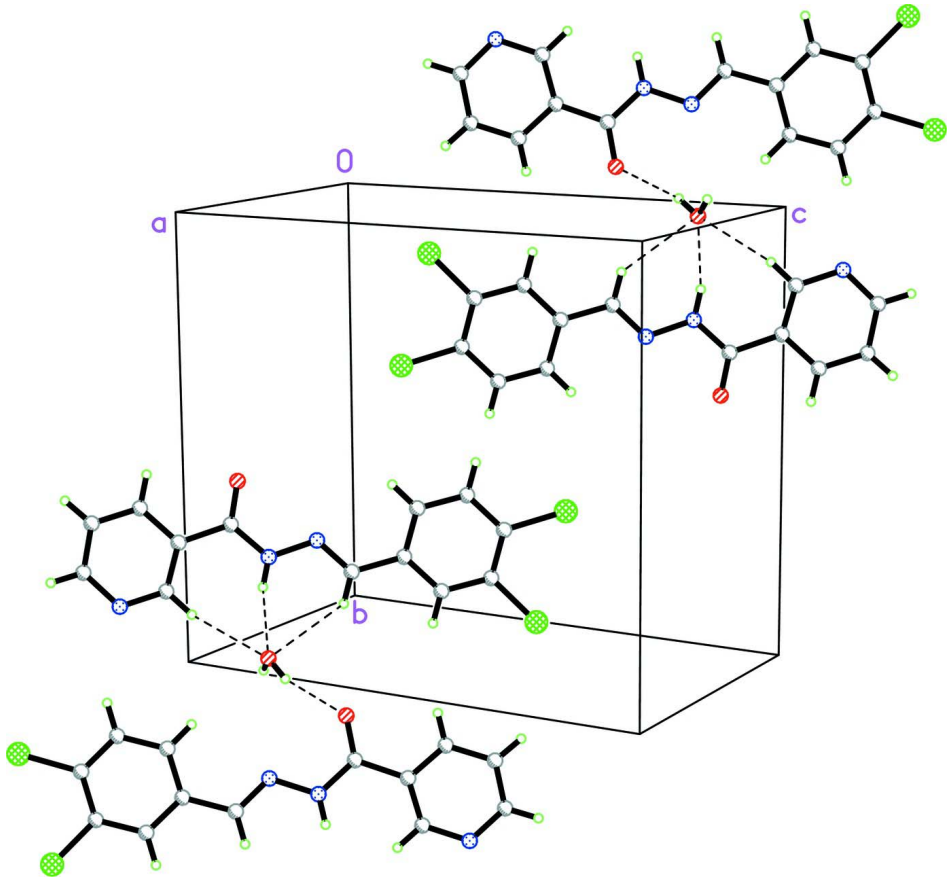

Figure 2

The unit cell packing diagram showing the intermolecular hydrogen bonding as dashed lines.

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Crystal data

$C_{13}H_9Cl_2N_3O·H_2O$
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Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 8.2080\ (3)$ Å
$b = 12.3294\ (4)$ Å
$c = 13.7089\ (4)$ Å
$\beta = 91.522\ (2)°$
$V = 1386.85\ (8)$ Å^3
$Z = 4$
$F(000) = 640$
$D_x = 1.495$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3887 reflections
$\theta = 2.5–27.0°$
$\mu = 0.47$ mm^{-1}
$T = 296$ K
Block, colourless
0.40 × 0.20 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
$T_{min} = 0.893$, $T_{max} = 0.954$
20965 measured reflections
3032 independent reflections

2150 reflections with $I > 2\sigma(I)$
$R_{int} = 0.042$
$\theta_{max} = 27.0°$, $\theta_{min} = 2.2°$
$h = -10 \rightarrow 10$
$k = -15 \rightarrow 15$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.100$
$S = 1.02$
3032 reflections
189 parameters
3 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0432P)^2 + 0.3048P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.022$
$\Delta\rho_{max} = 0.15$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}

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	−0.01836 (7)	0.35397 (5)	0.67808 (4)	0.07194 (19)
Cl2	0.02126 (7)	0.10476 (5)	0.62576 (4)	0.0725 (2)
N2	0.46330 (18)	0.25736 (12)	0.18259 (10)	0.0462 (4)
H2A	0.4694	0.1892	0.1698	0.055*
N1	0.38507 (18)	0.29373 (12)	0.26366 (10)	0.0471 (4)
O	0.52773 (19)	0.42935 (10)	0.14108 (9)	0.0657 (4)
C6	0.2369 (2)	0.25413 (14)	0.40566 (13)	0.0448 (4)
C8	0.5303 (2)	0.33162 (14)	0.12366 (12)	0.0457 (4)
C4	0.0960 (2)	0.20492 (15)	0.55082 (13)	0.0482 (4)
C3	0.0803 (2)	0.31321 (16)	0.57470 (13)	0.0487 (4)
C10	0.6343 (2)	0.18316 (14)	0.01178 (13)	0.0501 (4)
H10A	0.5992	0.1314	0.0559	0.060*
C2	0.1439 (2)	0.39176 (15)	0.51490 (14)	0.0527 (5)
H2	0.1344	0.4646	0.5315	0.063*
C9	0.6100 (2)	0.29128 (13)	0.03370 (12)	0.0420 (4)
C13	0.6619 (2)	0.36685 (15)	−0.03277 (13)	0.0519 (5)
H13A	0.6463	0.4404	−0.0214	0.062*
C5	0.1740 (2)	0.17586 (15)	0.46627 (13)	0.0483 (4)
H5A	0.1841	0.1029	0.4501	0.058*
C7	0.3221 (2)	0.22160 (15)	0.31757 (13)	0.0480 (4)
H7A	0.3301	0.1487	0.3011	0.058*

C1	0.2212 (2)	0.36294 (14)	0.43093 (14)	0.0508 (5)
H1	0.2632	0.4164	0.3909	0.061*
O1	0.5421 (2)	0.52708 (11)	0.32595 (11)	0.0651 (4)
N3	0.7054 (2)	0.14870 (12)	−0.06903 (11)	0.0564 (4)
C11	0.7563 (2)	0.22393 (16)	−0.13096 (14)	0.0558 (5)
H11A	0.8077	0.2014	−0.1871	0.067*
C12	0.7366 (3)	0.33289 (16)	−0.11592 (14)	0.0575 (5)
H12A	0.7731	0.3830	−0.1611	0.069*
H1B	0.599 (3)	0.4849 (16)	0.3622 (14)	0.092 (9)*
H1A	0.515 (3)	0.4919 (18)	0.2749 (11)	0.102 (10)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0761 (4)	0.0845 (4)	0.0562 (3)	0.0074 (3)	0.0190 (3)	−0.0066 (3)
Cl2	0.0856 (4)	0.0646 (3)	0.0679 (3)	−0.0098 (3)	0.0148 (3)	0.0166 (3)
N2	0.0585 (9)	0.0371 (7)	0.0434 (8)	0.0010 (7)	0.0081 (7)	−0.0042 (6)
N1	0.0519 (9)	0.0439 (8)	0.0458 (8)	0.0025 (7)	0.0058 (6)	−0.0041 (7)
O	0.1088 (12)	0.0353 (7)	0.0541 (8)	0.0024 (7)	0.0196 (7)	−0.0034 (6)
C6	0.0423 (10)	0.0429 (9)	0.0492 (9)	−0.0002 (8)	0.0026 (7)	−0.0017 (8)
C8	0.0558 (11)	0.0386 (9)	0.0427 (9)	0.0027 (8)	0.0004 (8)	−0.0014 (7)
C4	0.0463 (11)	0.0494 (10)	0.0490 (10)	−0.0031 (8)	0.0013 (8)	0.0069 (8)
C3	0.0436 (10)	0.0562 (11)	0.0466 (10)	0.0029 (9)	0.0052 (8)	−0.0032 (8)
C10	0.0666 (12)	0.0379 (9)	0.0461 (10)	0.0021 (9)	0.0079 (8)	0.0030 (8)
C2	0.0546 (12)	0.0435 (10)	0.0603 (11)	0.0024 (9)	0.0084 (9)	−0.0050 (9)
C9	0.0466 (10)	0.0379 (9)	0.0414 (9)	−0.0006 (8)	−0.0016 (7)	−0.0014 (7)
C13	0.0654 (12)	0.0379 (9)	0.0527 (10)	−0.0046 (8)	0.0061 (9)	−0.0011 (8)
C5	0.0502 (11)	0.0411 (9)	0.0535 (10)	−0.0017 (8)	0.0018 (8)	−0.0016 (8)
C7	0.0508 (11)	0.0424 (10)	0.0509 (10)	−0.0001 (8)	0.0040 (8)	−0.0050 (8)
C1	0.0528 (11)	0.0413 (10)	0.0590 (11)	−0.0007 (8)	0.0119 (9)	0.0013 (8)
O1	0.1057 (13)	0.0364 (7)	0.0533 (8)	0.0038 (8)	0.0052 (8)	−0.0022 (7)
N3	0.0752 (11)	0.0435 (9)	0.0512 (9)	0.0039 (8)	0.0131 (8)	−0.0031 (7)
C11	0.0632 (13)	0.0558 (12)	0.0490 (10)	−0.0018 (10)	0.0123 (9)	−0.0050 (9)
C12	0.0703 (13)	0.0488 (11)	0.0541 (11)	−0.0098 (10)	0.0163 (10)	0.0021 (9)

Geometric parameters (Å, º)

Cl1—C3	1.7255 (18)	C10—H10A	0.9300
Cl2—C4	1.7291 (18)	C2—C1	1.376 (2)
N2—C8	1.348 (2)	C2—H2	0.9300
N2—N1	1.3736 (19)	C9—C13	1.378 (2)
N2—H2A	0.8600	C13—C12	1.374 (3)
N1—C7	1.275 (2)	C13—H13A	0.9300
O—C8	1.229 (2)	C5—H5A	0.9300
C6—C5	1.383 (2)	C7—H7A	0.9300
C6—C1	1.392 (2)	C1—H1	0.9300
C6—C7	1.467 (2)	O1—H1B	0.85 (2)
C8—C9	1.497 (2)	O1—H1A	0.85 (2)

C4—C3	1.381 (3)	N3—C11	1.332 (2)
C4—C5	1.386 (2)	C11—C12	1.369 (3)
C3—C2	1.380 (3)	C11—H11A	0.9300
C10—N3	1.335 (2)	C12—H12A	0.9300
C10—C9	1.382 (2)		

C8—N2—N1	118.04 (14)	C13—C9—C8	118.00 (15)
C8—N2—H2A	121.0	C10—C9—C8	124.65 (15)
N1—N2—H2A	121.0	C12—C13—C9	119.66 (17)
C7—N1—N2	116.54 (15)	C12—C13—H13A	120.2
C5—C6—C1	118.97 (17)	C9—C13—H13A	120.2
C5—C6—C7	119.85 (16)	C6—C5—C4	120.70 (17)
C1—C6—C7	121.15 (16)	C6—C5—H5A	119.7
O—C8—N2	122.71 (16)	C4—C5—H5A	119.7
O—C8—C9	119.75 (16)	N1—C7—C6	119.73 (16)
N2—C8—C9	117.54 (15)	N1—C7—H7A	120.1
C3—C4—C5	119.74 (16)	C6—C7—H7A	120.1
C3—C4—Cl2	120.84 (14)	C2—C1—C6	120.31 (17)
C5—C4—Cl2	119.42 (14)	C2—C1—H1	119.8
C4—C3—C2	119.88 (16)	C6—C1—H1	119.8
C4—C3—Cl1	121.65 (14)	H1B—O1—H1A	107.2 (18)
C2—C3—Cl1	118.47 (15)	C11—N3—C10	117.30 (16)
N3—C10—C9	123.78 (17)	N3—C11—C12	123.14 (17)
N3—C10—H10A	118.1	N3—C11—H11A	118.4
C9—C10—H10A	118.1	C12—C11—H11A	118.4
C1—C2—C3	120.39 (17)	C11—C12—C13	118.75 (18)
C1—C2—H2	119.8	C11—C12—H12A	120.6
C3—C2—H2	119.8	C13—C12—H12A	120.6
C13—C9—C10	117.35 (16)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···O	0.85 (2)	2.00 (2)	2.8059 (19)	160 (2)
O1—H1*B*···N3^{i}	0.85 (2)	2.08 (1)	2.909 (2)	166 (2)
N2—H2*A*···O1^{ii}	0.86	2.00	2.842 (2)	165
C7—H7*A*···O1^{ii}	0.93	2.55	3.314 (2)	140
C10—H10*A*···O1^{ii}	0.93	2.39	3.304 (2)	167

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