organic compounds

(E)-N′-(2,4-Dichlorobenzylidene)-nicotinohydrazide

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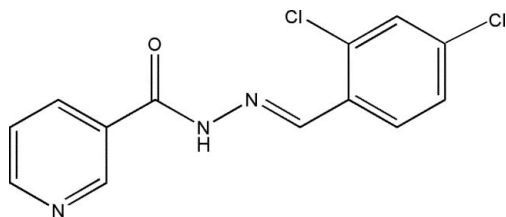
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The title compound, $C_{13}H_9Cl_2N_3O$, displays a *trans* configuration with respect to the C=N double bond. The central portion of the molecule is planar to within 0.025 Å, and forms dihedral angles of 31.7 (3) and 32.0 (3)° with the dichlorobenzene and pyridine rings, respectively. N—H···O hydrogen bonds link the molecules into chains

Related literature

For related Schiff-base structures, see: Qiu, Fang, *et al.* (2006); Qiu, Luo, *et al.* (2006).

Experimental

Crystal data

$C_{13}H_9Cl_2N_3O$	c = 11.9420 (12) Å
M_r = 294.13	β = 111.081 (4)°
Monoclinic, $P2_1/c$	V = 1331.3 (3) Å^3
a = 4.7646 (6) Å	Z = 4
b = 25.075 (3) Å	Mo $K\alpha$ radiation

μ = 0.48 mm^{-1}	0.31 × 0.12 × 0.11 mm
T = 298 (2) K	

Data collection

Bruker SMART APEX CCD diffractometer	7889 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	2290 independent reflections
T_{min} = 0.930, T_{max} = 0.945	1206 reflections with $I > 2\sigma(I)$
	R_{int} = 0.046

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047	173 parameters
$wR(F^2)$ = 0.127	H-atom parameters constrained
S = 0.85	$\Delta\rho_{max}$ = 0.21 e Å^{-3}
2290 reflections	$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1^i	0.90	2.07	2.827 (3)	141

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

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: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

References

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supplementary materials

(*E*)-*N'*-(2,4-Dichlorobenzylidene)nicotinohydrazide

X.-Y. Qiu

Comment

As an extension of our work on the structural characterization of Schiff-base compounds (Qiu, Fang *et al.*, 2006; Qiu, Luo *et al.*, 2006), the crystal structure of the title compound is reported here. In the molecule (Fig. 1), the C7=N3 bond length of 1.279 (3) Å conforms to the expected value for a double bond. The N2—C6 bond length of 1.341 (3) Å is between that expected for a single and a double bond, because of conjugation effects in molecule. The central portion of the molecule is planar to within 0.025 Å, and forms dihedral angles of 31.7 (3) and 32.0 (3) ° to the dichlorobenzene and pyridine rings, respectively. N—H⋯O hydrogen bonds link molecules into chains (Fig. 2).

Experimental

The reagents were commercial products used without further purification. 2,4-Dichlorobenzaldehyde (0.1 mmol, 17.5 mg) and nicotinohydrazide (0.1 mmol, 13.7 mg) were dissolved in ethanol (15 ml). The reaction mixture was stirred for 20 min to give a clear solution. After allowing the resulting clear solution to stand at room temperature in air for 10 d, large white crystals were formed at the bottom of the vessel on slow evaporation of the solvent. The crystals were isolated, washed three times with ethanol and dried in a vacuum desiccator over anhydrous $CaCl_2$ (yield 52%).

Refinement

All H were placed in geometrically idealized positions (C—H 0.93 Å N—H 0.90 Å), and constrained to ride on their parent atoms. They were treated as riding atoms, with $U_{iso}(H) = 1.2U_{eq}(C/N)$.
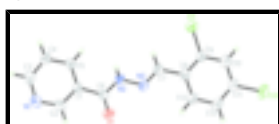
Figures

Fig. 1. The molecular structure of the title compound, showing displacement ellipsoids at 30% probability for non-H atoms.
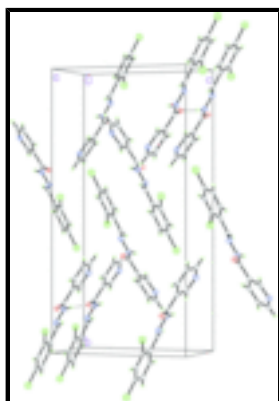

Fig. 2. Packing diagram, viewed approximately along the *a* axis. Dashed lines show N—H⋯O hydrogen bonds.

(*E*)-*N*'-(2,4-Dichlorobenzylidene)nicotinohydrazide

Crystal data

$C_{13}H_9Cl_2N_3O$	$F_{000} = 600$
$M_r = 294.13$	$D_x = 1.468$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 1785 reflections
$a = 4.7646$ (6) Å	$\theta = 4.2$–$25°$
$b = 25.075$ (3) Å	$\mu = 0.48$ mm^{-1}
$c = 11.9420$ (12) Å	$T = 298$ (2) K
$\beta = 111.081$ (4)$°$	Block, white
$V = 1331.3$ (3) Å^3	$0.31 \times 0.12 \times 0.11$ mm
$Z = 4$	

Data collection

Bruker SMART APEX CCD diffractometer	2290 independent reflections
Radiation source: fine-focus sealed tube	1206 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.046$
$T = 298(2)$ K	$\theta_{max} = 25.0°$
ω scans	$\theta_{min} = 2.0°$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -5 \rightarrow 5$
$T_{min} = 0.930$, $T_{max} = 0.945$	$k = -29 \rightarrow 29$
7889 measured reflections	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.047$	$w = 1/[\sigma^2(F_o^2) + (0.0774P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.127$	$(\Delta/\sigma)_{max} = 0.001$
$S = 0.85$	$\Delta\rho_{max} = 0.21$ e Å^{-3}
2290 reflections	$\Delta\rho_{min} = -0.19$ e Å^{-3}
173 parameters	Extinction correction: SHELXTL (Bruker, 1997), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.014 (3)
Secondary atom site location: difference Fourier map	

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	−0.2420 (2)	0.95073 (3)	0.62765 (8)	0.0809 (4)
Cl2	0.3137 (2)	1.13761 (3)	0.63521 (9)	0.1026 (4)
O1	1.0158 (5)	0.84278 (8)	1.04441 (19)	0.0712 (6)
C3	0.6738 (9)	0.68767 (13)	1.2109 (3)	0.0718 (9)
H3	0.6556	0.6573	1.2523	0.086*
N2	0.5113 (5)	0.85361 (8)	0.9690 (2)	0.0564 (6)
H2A	0.3301	0.8402	0.9613	0.068*
N3	0.5329 (6)	0.90164 (9)	0.9153 (2)	0.0571 (6)
C1	0.7164 (6)	0.77671 (11)	1.0901 (3)	0.0496 (7)
C2	0.9336 (7)	0.76173 (13)	1.1980 (3)	0.0687 (9)
H2	1.1026	0.7833	1.2295	0.082*
N1	0.9163 (7)	0.71808 (11)	1.2600 (2)	0.0794 (8)
C4	0.4503 (8)	0.69823 (12)	1.1037 (3)	0.0716 (9)
H4	0.2882	0.6751	1.0727	0.086*
C5	0.4689 (7)	0.74424 (11)	1.0413 (3)	0.0610 (8)
H5	0.3185	0.7528	0.9686	0.073*
C6	0.7634 (7)	0.82730 (11)	1.0325 (2)	0.0515 (7)
C7	0.2834 (7)	0.92486 (11)	0.8581 (2)	0.0576 (8)
H7	0.1019	0.9090	0.8520	0.069*
C8	0.2873 (7)	0.97689 (10)	0.8022 (3)	0.0540 (7)
C9	0.0573 (6)	0.99328 (11)	0.6987 (3)	0.0584 (8)
C10	0.0620 (7)	1.04245 (11)	0.6468 (3)	0.0672 (9)
H10	−0.0945	1.0528	0.5775	0.081*
C11	0.3031 (8)	1.07572 (11)	0.6999 (3)	0.0692 (9)
C12	0.5376 (8)	1.06099 (12)	0.8028 (3)	0.0691 (9)
H12	0.6994	1.0838	0.8378	0.083*
C13	0.5272 (7)	1.01154 (11)	0.8527 (3)	0.0660 (9)
H13	0.6848	1.0013	0.9217	0.079*

Atomic displacement parameters ($Å^2$)

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

Cl1	0.0815 (7)	0.0744 (6)	0.0757 (6)	−0.0159 (5)	0.0149 (5)	0.0034 (4)
Cl2	0.1464 (10)	0.0508 (5)	0.1095 (8)	−0.0097 (5)	0.0445 (7)	0.0140 (5)
O1	0.0508 (14)	0.0715 (14)	0.0928 (17)	−0.0066 (11)	0.0277 (12)	0.0013 (11)
C3	0.089 (3)	0.057 (2)	0.075 (2)	0.0097 (19)	0.037 (2)	0.0134 (18)
N2	0.0482 (15)	0.0513 (14)	0.0742 (17)	−0.0030 (12)	0.0272 (12)	0.0118 (12)
N3	0.0631 (17)	0.0467 (14)	0.0654 (16)	−0.0030 (12)	0.0279 (13)	0.0034 (12)
C1	0.0438 (18)	0.0500 (17)	0.0556 (18)	0.0033 (14)	0.0188 (14)	0.0014 (14)
C2	0.060 (2)	0.075 (2)	0.064 (2)	−0.0036 (18)	0.0132 (16)	0.0023 (18)
N1	0.083 (2)	0.0792 (19)	0.0690 (19)	0.0032 (17)	0.0181 (16)	0.0188 (16)
C4	0.077 (2)	0.0484 (18)	0.087 (3)	−0.0103 (16)	0.027 (2)	−0.0019 (17)
C5	0.062 (2)	0.0508 (17)	0.0633 (19)	0.0019 (16)	0.0145 (16)	0.0039 (15)
C6	0.0459 (19)	0.0558 (18)	0.0528 (18)	−0.0001 (16)	0.0178 (14)	−0.0036 (14)
C7	0.060 (2)	0.0501 (17)	0.065 (2)	−0.0054 (16)	0.0250 (16)	0.0033 (15)
C8	0.0603 (19)	0.0478 (16)	0.0581 (18)	0.0012 (15)	0.0262 (15)	0.0004 (14)
C9	0.062 (2)	0.0500 (17)	0.0648 (19)	−0.0034 (15)	0.0248 (16)	−0.0013 (15)
C10	0.079 (2)	0.0532 (19)	0.071 (2)	0.0036 (18)	0.0290 (18)	0.0064 (16)
C11	0.096 (3)	0.0437 (17)	0.078 (2)	−0.0004 (18)	0.043 (2)	0.0040 (16)
C12	0.085 (2)	0.0523 (19)	0.074 (2)	−0.0170 (17)	0.034 (2)	−0.0102 (17)
C13	0.076 (2)	0.0513 (18)	0.068 (2)	−0.0079 (17)	0.0237 (17)	−0.0008 (16)

Geometric parameters (Å, °)

Cl1—C9	1.738 (3)	C4—C5	1.393 (4)
Cl2—C11	1.743 (3)	C4—H4	0.930
O1—C6	1.222 (3)	C5—H5	0.930
C3—N1	1.331 (4)	C7—C8	1.469 (4)
C3—C4	1.365 (4)	C7—H7	0.930
C3—H3	0.930	C8—C9	1.387 (4)
N2—C6	1.341 (3)	C8—C13	1.389 (4)
N2—N3	1.385 (3)	C9—C10	1.383 (4)
N2—H2A	0.900	C10—C11	1.376 (4)
N3—C7	1.279 (3)	C10—H10	0.930
C1—C5	1.378 (4)	C11—C12	1.381 (4)
C1—C2	1.383 (4)	C12—C13	1.384 (4)
C1—C6	1.498 (4)	C12—H12	0.930
C2—N1	1.341 (4)	C13—H13	0.930
C2—H2	0.930		
N1—C3—C4	124.3 (3)	N2—C6—C1	115.1 (3)
N1—C3—H3	117.9	N3—C7—C8	119.1 (3)
C4—C3—H3	117.9	N3—C7—H7	120.5
C6—N2—N3	119.3 (2)	C8—C7—H7	120.5
C6—N2—H2A	120.9	C9—C8—C13	117.7 (3)
N3—N2—H2A	120.4	C9—C8—C7	122.2 (3)
C7—N3—N2	115.7 (2)	C13—C8—C7	120.1 (3)
C5—C1—C2	118.3 (3)	C10—C9—C8	121.9 (3)
C5—C1—C6	123.5 (3)	C10—C9—Cl1	118.1 (2)
C2—C1—C6	118.2 (3)	C8—C9—Cl1	120.0 (2)
N1—C2—C1	124.2 (3)	C11—C10—C9	118.6 (3)
N1—C2—H2	117.9	C11—C10—H10	120.7

C1—C2—H2	117.9	C9—C10—H10	120.7
C3—N1—C2	116.1 (3)	C10—C11—C12	121.5 (3)
C3—C4—C5	118.9 (3)	C10—C11—Cl2	119.2 (3)
C3—C4—H4	120.5	C12—C11—Cl2	119.3 (3)
C5—C4—H4	120.5	C11—C12—C13	118.7 (3)
C1—C5—C4	118.2 (3)	C11—C12—H12	120.7
C1—C5—H5	120.9	C13—C12—H12	120.7
C4—C5—H5	120.9	C12—C13—C8	121.7 (3)
O1—C6—N2	123.6 (3)	C12—C13—H13	119.2
O1—C6—C1	121.3 (3)	C8—C13—H13	119.2
C6—N2—N3—C7	−178.8 (2)	N3—C7—C8—C9	149.4 (3)
C5—C1—C2—N1	−1.6 (5)	N3—C7—C8—C13	−30.4 (4)
C6—C1—C2—N1	178.6 (3)	C13—C8—C9—C10	−0.5 (4)
C4—C3—N1—C2	0.4 (5)	C7—C8—C9—C10	179.7 (3)
C1—C2—N1—C3	1.2 (5)	C13—C8—C9—Cl1	177.4 (2)
N1—C3—C4—C5	−1.5 (5)	C7—C8—C9—Cl1	−2.4 (4)
C2—C1—C5—C4	0.4 (4)	C8—C9—C10—C11	0.2 (4)
C6—C1—C5—C4	−179.8 (3)	Cl1—C9—C10—C11	−177.8 (2)
C3—C4—C5—C1	1.0 (5)	C9—C10—C11—C12	0.1 (5)
N3—N2—C6—O1	−1.5 (4)	C9—C10—C11—Cl2	179.6 (2)
N3—N2—C6—C1	178.1 (2)	C10—C11—C12—C13	0.0 (5)
C5—C1—C6—O1	−148.3 (3)	Cl2—C11—C12—C13	−179.5 (2)
C2—C1—C6—O1	31.4 (4)	C11—C12—C13—C8	−0.3 (5)
C5—C1—C6—N2	32.1 (4)	C9—C8—C13—C12	0.5 (4)
C2—C1—C6—N2	−148.2 (3)	C7—C8—C13—C12	−179.6 (3)
N2—N3—C7—C8	177.9 (2)		

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1[i]	0.90	2.07	2.827 (3)	141

Symmetry codes: (i) x−1, y, z.

Fig. 1

Fig. 2