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N′-(2-Chloro-5-nitrobenzylidene)-isonicotinohydrazide

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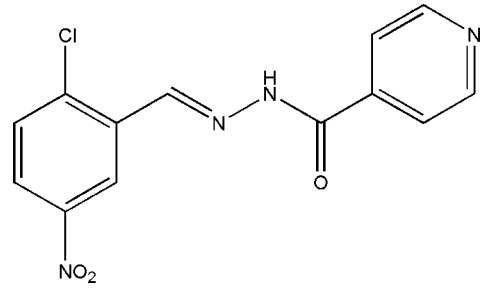
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.054; wR factor = 0.131; data-to-parameter ratio = 14.8.

The title compound, $C_{13}H_9ClN_4O_3$, was synthesized by the condensation reaction of 2-chloro-5-nitrobenzaldehyde with isonicotinohydrazide in a methanol solution. The molecule of the compound displays a *trans* configuration with respect to the C=N and C—N bonds. The dihedral angle between the benzene and pyridine rings is 12.1 (2)°. In the crystal structure, adjacent molecules are linked through intermolecular N—H···O hydrogen bonds, forming dimers.

Related literature

For Schiff base compounds, see: Fan *et al.* (2007); Kim *et al.* (2005); Nimitsiriwat *et al.* (2004). For the biological activity of Schiff base compounds, see: Chen *et al.* (1997); Ren *et al.* (2002). For similar structures, see: Mohd Lair *et al.* (2009); Fun *et al.* (2008); Yang (2008); Zhi (2008); Zhi & Yang (2007).



Experimental

Crystal data

$C_{13}H_9ClN_4O_3$
M_r = 304.69
Tetragonal, $I4_1/a$
a = 18.586 (3) Å
c = 15.183 (3) Å
V = 5244.9 (15) Å^3
Z = 16
Mo $K\alpha$ radiation
μ = 0.31 mm^{-1}
T = 298 K
0.12 × 0.10 × 0.10 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.964, T_{max} = 0.970
16616 measured reflections
2869 independent reflections
1685 reflections with $I > 2\sigma(I)$
R_{int} = 0.083

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.131
S = 1.03
2869 reflections
194 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O3^{i}	0.892 (10)	2.187 (13)	3.055 (3)	164 (3)

Symmetry code: (i) $y-\frac{1}{4}, -x+\frac{3}{4}, z-\frac{1}{4}$.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); 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: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2481).

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supporting information

Acta Cryst. (2009). E**65**, o623 [doi:10.1107/S1600536809006588]

N′-(2-Chloro-5-nitrobenzylidene)isonicotinohydrazide

Feng Zhi

S1. 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, (I), Fig. 1, derived from the condensation reaction of 2-chloro-5-nitrobenzaldehyde with isonicotinohydrazide is reported.

In (I), the molecular structure of the compound displays a *trans* configuration with respect to the C=N and C-N bonds. The dihedral angle between the benzene ring and the pyridine ring is 12.1 (2)°. The dihedral angle between the O1/N4/O2 plane and the benzene ring is 8.1 (2)°. All the bond lengths are within normal ranges and comparable to those in other similar compounds (Mohd Lair *et al.*, 2009; Fun *et al.*, 2008; Yang, 2008; Zhi, 2008; Zhi & Yang, 2007).

In the crystal structure, adjacent molecules are linked through intermolecular N—H···O hydrogen bonds (Table 1), forming dimers (Fig. 2).

S2. Experimental

2-Chloro-5-nitrobenzaldehyde (0.01 mol, 1.85 g) and isonicotinohydrazide (0.01 mol, 1.37 g) were dissolved in a methanol solution (50 ml). The mixture was stirred at room temperature to give a clear colorless solution. Crystals of the title compound were formed by gradual evaporation of the solvent for a week at room temperature.

S3. Refinement

The N proton H2 was located in a difference map and refined with N–H distance restrained to 0.90 (1) Å. All other H atoms were positioned geometrically [C–H = 0.93 Å] and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C).

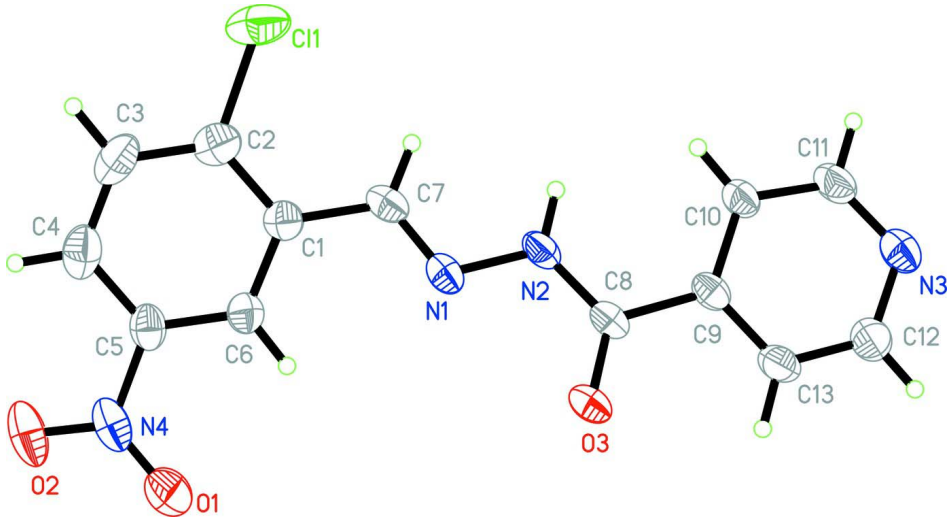

Figure 1

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

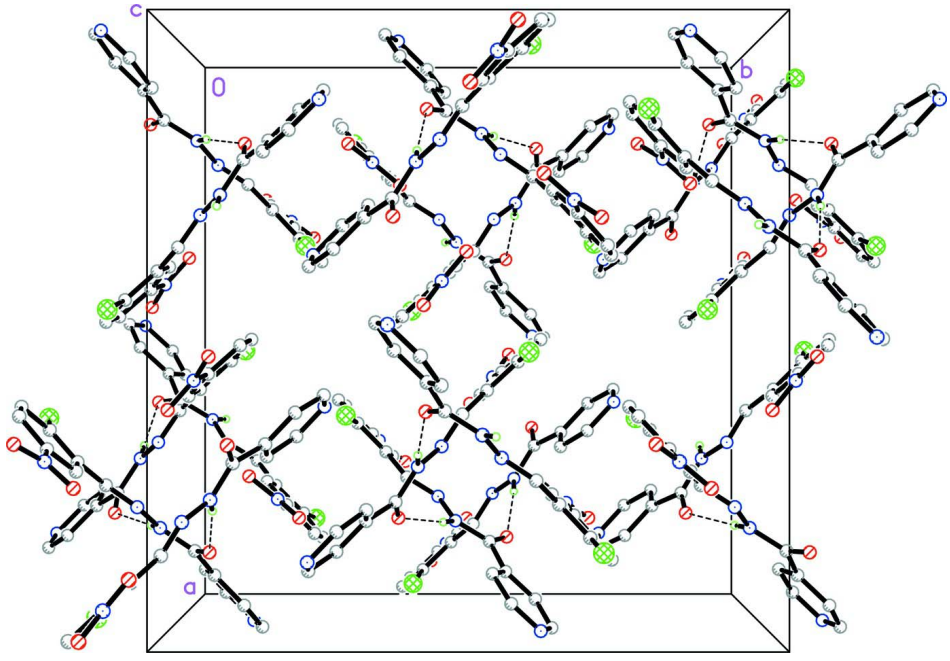

Figure 2

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

N′-(2-Chloro-5-nitrobenzylidene)isonicotinohydrazide

Crystal data

$C_{13}H_9ClN_4O_3$
M_r = 304.69
Tetragonal, $I4_1/a$
Hall symbol: -I 4ad
a = 18.586 (3) Å
c = 15.183 (3) Å
V = 5244.9 (15) Å^3
Z = 16
F(000) = 2496
D_x = 1.543 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1528 reflections
θ = 2.2–24.5°
μ = 0.31 mm^{-1}

T = 298 K
Block, colorless

0.12 × 0.10 × 0.10 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.964, T_{max} = 0.970

16616 measured reflections
2869 independent reflections
1685 reflections with $I > 2\sigma(I)$
R_{int} = 0.083
θ_{max} = 27.0°, θ_{min} = 1.7°
h = −23→22
k = −21→23
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.131
S = 1.03
2869 reflections
194 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0346P)^2 + 2.6778P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00084 (18)

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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
Cl1	0.14870 (5)	0.28558 (6)	0.06873 (6)	0.0950 (4)
O1	0.24702 (14)	0.38109 (14)	0.46884 (14)	0.0866 (7)
O2	0.17681 (13)	0.29247 (14)	0.49968 (15)	0.0980 (8)
O3	0.36910 (10)	0.56885 (10)	0.18178 (11)	0.0646 (5)
N1	0.29514 (11)	0.44612 (12)	0.16009 (13)	0.0507 (5)
N2	0.33431 (12)	0.47740 (12)	0.09354 (12)	0.0520 (6)
N3	0.50947 (13)	0.62326 (13)	−0.09159 (15)	0.0627 (6)
N4	0.20600 (15)	0.33277 (15)	0.44731 (16)	0.0674 (7)
C1	0.21758 (13)	0.35202 (14)	0.20311 (17)	0.0497 (6)
C2	0.16520 (15)	0.30249 (16)	0.17913 (19)	0.0624 (8)
C3	0.12506 (16)	0.26504 (17)	0.2413 (3)	0.0767 (10)
H3	0.0893	0.2333	0.2231	0.092*

C4	0.13799 (16)	0.27471 (16)	0.3291 (2)	0.0723 (9)
H4	0.1120	0.2494	0.3713	0.087*
C5	0.19049 (14)	0.32300 (14)	0.35332 (18)	0.0540 (7)
C6	0.22899 (13)	0.36163 (14)	0.29285 (16)	0.0505 (6)
H6	0.2632	0.3947	0.3120	0.061*
C7	0.25958 (14)	0.39094 (15)	0.13746 (17)	0.0544 (7)
H7	0.2603	0.3753	0.0793	0.065*
C8	0.37206 (13)	0.53777 (14)	0.11106 (15)	0.0471 (6)
C9	0.41915 (13)	0.56487 (13)	0.03806 (15)	0.0446 (6)
C10	0.41588 (15)	0.54180 (14)	−0.04791 (15)	0.0543 (7)
H10	0.3836	0.5060	−0.0645	0.065*
C11	0.46139 (17)	0.57273 (16)	−0.10915 (17)	0.0643 (8)
H11	0.4580	0.5568	−0.1671	0.077*
C12	0.51235 (15)	0.64389 (16)	−0.00807 (19)	0.0632 (8)
H12	0.5459	0.6789	0.0070	0.076*
C13	0.46919 (15)	0.61719 (15)	0.05767 (17)	0.0588 (7)
H13	0.4737	0.6343	0.1150	0.071*
H2	0.3334 (16)	0.4566 (14)	0.0406 (10)	0.080*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0684 (6)	0.1253 (8)	0.0914 (6)	0.0108 (5)	−0.0135 (4)	−0.0546 (6)
O1	0.1009 (18)	0.1033 (19)	0.0558 (13)	−0.0094 (15)	−0.0011 (12)	0.0119 (12)
O2	0.1027 (18)	0.1125 (19)	0.0787 (15)	0.0079 (15)	0.0333 (13)	0.0459 (14)
O3	0.0837 (14)	0.0749 (13)	0.0351 (10)	−0.0072 (11)	0.0146 (9)	−0.0096 (9)
N1	0.0517 (13)	0.0618 (14)	0.0385 (11)	0.0045 (11)	0.0075 (10)	0.0043 (10)
N2	0.0645 (14)	0.0606 (15)	0.0308 (11)	0.0020 (12)	0.0090 (10)	−0.0004 (10)
N3	0.0636 (16)	0.0770 (17)	0.0476 (15)	0.0023 (13)	0.0087 (11)	0.0066 (12)
N4	0.0682 (17)	0.0753 (18)	0.0588 (16)	0.0173 (15)	0.0191 (13)	0.0226 (14)
C1	0.0445 (15)	0.0519 (16)	0.0526 (15)	0.0061 (12)	0.0028 (12)	−0.0037 (12)
C2	0.0493 (17)	0.0646 (19)	0.073 (2)	0.0108 (14)	0.0018 (15)	−0.0201 (15)
C3	0.0525 (19)	0.062 (2)	0.116 (3)	−0.0120 (15)	0.0152 (19)	−0.0258 (19)
C4	0.064 (2)	0.0575 (19)	0.095 (3)	−0.0031 (16)	0.0257 (18)	−0.0006 (17)
C5	0.0489 (16)	0.0497 (16)	0.0635 (18)	0.0065 (13)	0.0140 (13)	0.0059 (13)
C6	0.0469 (15)	0.0528 (16)	0.0519 (16)	0.0001 (12)	0.0037 (12)	0.0029 (12)
C7	0.0588 (17)	0.0681 (19)	0.0364 (14)	0.0076 (15)	0.0028 (12)	−0.0043 (13)
C8	0.0546 (16)	0.0541 (16)	0.0325 (13)	0.0091 (13)	0.0035 (11)	0.0015 (11)
C9	0.0470 (15)	0.0507 (15)	0.0359 (13)	0.0129 (12)	0.0034 (11)	0.0023 (11)
C10	0.0679 (18)	0.0570 (16)	0.0378 (14)	−0.0026 (14)	0.0091 (12)	−0.0018 (12)
C11	0.081 (2)	0.074 (2)	0.0377 (15)	0.0014 (18)	0.0124 (14)	−0.0056 (14)
C12	0.0542 (17)	0.078 (2)	0.0572 (18)	−0.0075 (15)	−0.0030 (14)	0.0078 (15)
C13	0.0644 (18)	0.0738 (19)	0.0383 (14)	−0.0027 (15)	−0.0019 (13)	−0.0018 (13)

Geometric parameters (Å, º)

Cl1—C2	1.733 (3)	C3—H3	0.9300
O1—N4	1.222 (3)	C4—C5	1.376 (4)

O2—N4	1.220 (3)	C4—H4	0.9300
O3—C8	1.221 (3)	C5—C6	1.368 (3)
N1—C7	1.268 (3)	C6—H6	0.9300
N1—N2	1.374 (3)	C7—H7	0.9300
N2—C8	1.350 (3)	C8—C9	1.499 (3)
N2—H2	0.892 (10)	C9—C10	1.375 (3)
N3—C11	1.324 (4)	C9—C13	1.378 (3)
N3—C12	1.326 (3)	C10—C11	1.382 (4)
N4—C5	1.467 (4)	C10—H10	0.9300
C1—C2	1.388 (4)	C11—H11	0.9300
C1—C6	1.391 (3)	C12—C13	1.373 (4)
C1—C7	1.458 (4)	C12—H12	0.9300
C2—C3	1.390 (4)	C13—H13	0.9300
C3—C4	1.367 (4)		

C7—N1—N2	114.8 (2)	C5—C6—H6	119.6
C8—N2—N1	118.8 (2)	C1—C6—H6	119.6
C8—N2—H2	123 (2)	N1—C7—C1	119.7 (2)
N1—N2—H2	118 (2)	N1—C7—H7	120.2
C11—N3—C12	115.2 (2)	C1—C7—H7	120.2
O2—N4—O1	123.7 (3)	O3—C8—N2	122.9 (2)
O2—N4—C5	118.1 (3)	O3—C8—C9	121.2 (2)
O1—N4—C5	118.3 (2)	N2—C8—C9	115.9 (2)
C2—C1—C6	116.7 (2)	C10—C9—C13	117.1 (2)
C2—C1—C7	121.7 (3)	C10—C9—C8	124.8 (2)
C6—C1—C7	121.6 (2)	C13—C9—C8	118.1 (2)
C1—C2—C3	122.0 (3)	C9—C10—C11	118.8 (3)
C1—C2—Cl1	119.9 (2)	C9—C10—H10	120.6
C3—C2—Cl1	118.1 (2)	C11—C10—H10	120.6
C4—C3—C2	120.1 (3)	N3—C11—C10	124.9 (3)
C4—C3—H3	119.9	N3—C11—H11	117.5
C2—C3—H3	119.9	C10—C11—H11	117.5
C3—C4—C5	118.1 (3)	N3—C12—C13	124.6 (3)
C3—C4—H4	120.9	N3—C12—H12	117.7
C5—C4—H4	120.9	C13—C12—H12	117.7
C6—C5—C4	122.3 (3)	C12—C13—C9	119.5 (2)
C6—C5—N4	119.0 (3)	C12—C13—H13	120.3
C4—C5—N4	118.7 (3)	C9—C13—H13	120.3
C5—C6—C1	120.7 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O3^{i}	0.89 (1)	2.19 (1)	3.055 (3)	164 (3)

Symmetry code: (i) *y*−1/4, −*x*+3/4, *z*−1/4.