Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N′-(2-Methoxybenzylidene)nicotinohydrazide

Ping Wang, Cong Li and Yong-Qing Su*

Faculty of Chemistry and Chemical Engineering, Yunnan Normal University, Kunming 650092, People's Republic of China
Correspondence e-mail: yongqing_su@163.com

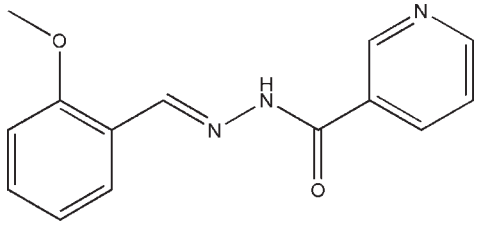
Received 29 January 2010; accepted 31 January 2010

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.035; wR factor = 0.084; data-to-parameter ratio = 8.6.

The title compound, $C_{14}H_{13}N_3O_2$, was prepared by the reaction of 2-methoxybenzyaldehyde with nicotinic acid hydrazide in methanol. The dihedral angle between the benzene and pyridine rings is 5.9 (3)°. In the crystal structure, molecules are linked by intermolecular N—H···O hydrogen bonds, leading to the formation of chains along the *c* axis; adjacent chains are linked *via* C—H···O and C—H···N hydrogen bonds.

Related literature

For general background to Schiff base compounds, see: Archibald *et al.* (1994); Harada *et al.* (1999); Ogawa *et al.* (1998). For related structures, see: Mohd Lair *et al.* (2009); Sun *et al.* (2009); Wen *et al.* (2009).



Experimental

Crystal data

$C_{14}H_{13}N_3O_2$
M_r = 255.27
Tetragonal, $P4_3$
a = 9.3264 (13) Å
c = 15.594 (3) Å
V = 1356.4 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 K
0.20 × 0.20 × 0.18 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.983, T_{max} = 0.985
6623 measured reflections
1519 independent reflections
1313 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.084
S = 1.06
1519 reflections
176 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.09 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O2^{i}	0.90 (1)	2.05 (2)	2.897 (2)	157 (3)
C4—H4···O1^{ii}	0.93	2.58	3.469 (3)	160
C11—H11···N3^{iii}	0.93	2.53	3.429 (4)	164
C13—H13···N1^{i}	0.93	2.56	3.487 (3)	176

Symmetry codes: (i) $-y+1, x, z-\frac{1}{4}$; (ii) $y+1, -x+1, z+\frac{1}{4}$; (iii) $y-1, -x+1, z+\frac{1}{4}$.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); 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: *SHELXTL*.

The authors acknowledge support by the Science and Technology Department of Yunnan Province, the Program of Yunnan Province's Young-Middle Aged Reserve Scientific and Technological Principal Culture Plan (grant No. 2006PY01–50) and the fund of Yunnan Province's Applied Research Plan (grant No. 2006E0032*M*).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI5028).

References

Archibald, S. J., Blake, A. J., Schroder, M. & Winpenny, R. E. P. (1994). *Chem. Commun.* pp. 1669–1670.
Bruker (2004). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Harada, J., Uekusa, H. & Ohashi, Y. (1999). *J. Am. Chem. Soc.* **121**, 5809–5810.
Mohd Lair, N., Mohd Ali, H. & Ng, S. W. (2009). *Acta Cryst.* E**65**, o189.
Ogawa, K., Kasahara, Y., Ohtani, Y. & Harada, J. (1998). *J. Am. Chem. Soc.* **120**, 7107–7108.
Sheldrick, G. M. (2004). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sun, Y., Li, H.-G., Wang, X., Fu, S. & Wang, D. (2009). *Acta Cryst.* E**65**, o262.
Wen, L., Yin, H., Li, W. & Li, K. (2009). *Acta Cryst.* E**65**, o2623.

supporting information

Acta Cryst. (2010). E**66**, o542 [doi:10.1107/S1600536810003831]

N′-(2-Methoxybenzylidene)nicotinohydrazide

Ping Wang, Cong Li and Yong-Qing Su

S1. Comment

Schiff bases have been received much attention in recent years (Ogawa *et al.*, 1998; Archibald *et al.*, 1994; Harada *et al.*, 1999). As a further investigation of the structures of Schiff base compounds, the title new compound is reported here.

In the title compound, the dihedral angle between the benzene ring and the pyridine ring is 5.9 (3)°. All the bond lengths are comparable with the similar Schiff bases reported previously (Wen *et al.*, 2009; Mohd Lair *et al.*, 2009; Sun *et al.*, 2009).

In the crystal structure, molecules form chains running along the *c* axis through intermolecular N—H···O hydrogen bonds (Table 1 and Fig. 2).

S2. Experimental

2-Methoxybenzaldehyde (1.0 mmol, 136 mg) and nicotinic acid hydrazide (1.0 mmol, 137 mg) were dissolved in methanol (30 ml). The mixture was stirred at room temperature for 1 h to give a colourless solution. After keeping the solution in air for 3 d, colourless block shaped crystals were formed.

S3. Refinement

Atom H2 was located in a difference Fourier map and refined isotropically, with the N–H distance restrained to 0.90 (1) Å. The other H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93-0.96 Å, and with U_{iso}(H) = 1.2 or 1.5U_{eq}(C). In the absence of significant anomalous dispersion effects, Friedel pairs were merged before the final refinement.

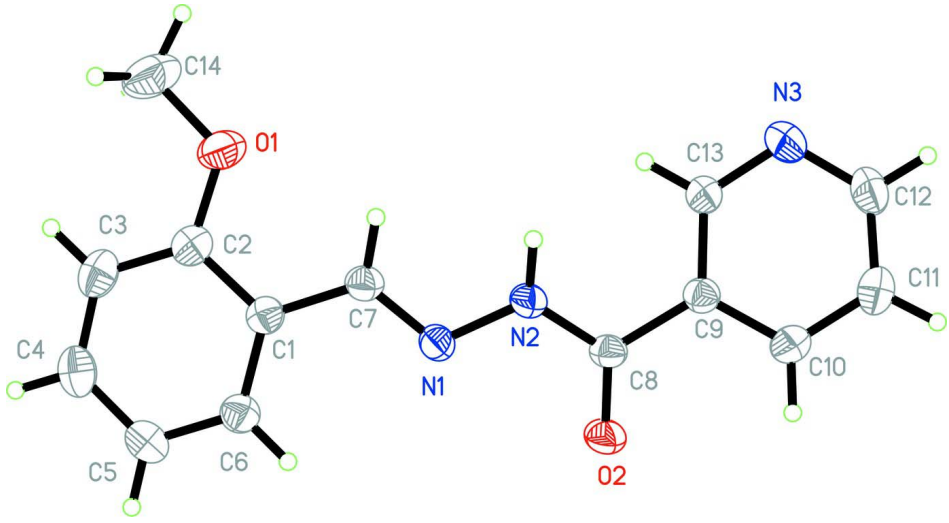


Figure 1

The molecular structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

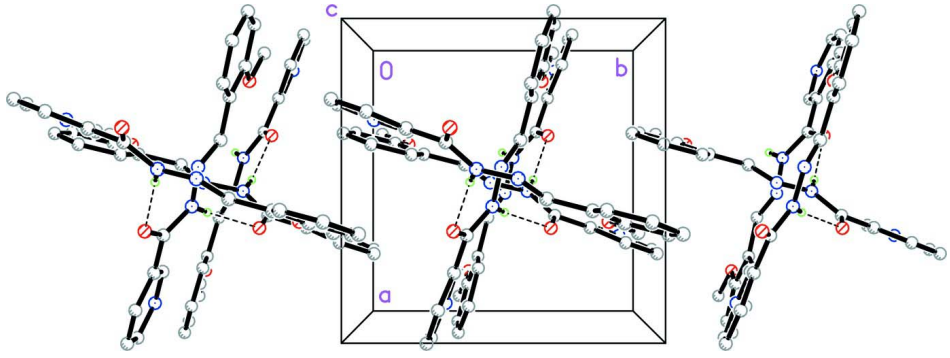


Figure 2

The crystal packing of the title compound. Intermolecular hydrogen bonds are shown as dashed lines.

N′-(2-Methoxybenzylidene)nicotinohydrazide

Crystal data

$C_{14}H_{13}N_3O_2$
$M_r = 255.27$
Tetragonal, $P4_3$
Hall symbol: P 4cw
$a = 9.3264\ (13)$ Å
$c = 15.594\ (3)$ Å
$V = 1356.4\ (4)$ Å^3
$Z = 4$
$F(000) = 536$
$D_x = 1.250$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2214 reflections
$\theta = 2.5$–$25.3°$
$\mu = 0.09$ mm^{-1}
$T = 298$ K
Block, colourless
0.20 × 0.20 × 0.18 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
$T_{min} = 0.983$, $T_{max} = 0.985$

6623 measured reflections
1519 independent reflections
1313 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 27.0°$, $\theta_{min} = 2.2°$
$h = -11 \rightarrow 10$
$k = -4 \rightarrow 11$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.084$
$S = 1.06$
1519 reflections
176 parameters
2 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
$w = 1/[\sigma^2(F_o^2) + (0.0427P)^2 + 0.084P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.09$ e Å^{-3}
$\Delta\rho_{min} = -0.14$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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}
N1	0.54565 (17)	0.50653 (18)	0.21981 (11)	0.0443 (4)
N2	0.41517 (19)	0.53627 (19)	0.18161 (11)	0.0457 (4)
N3	0.0801 (2)	0.6980 (3)	0.03464 (14)	0.0763 (7)
O1	0.84079 (19)	0.3686 (2)	0.05708 (12)	0.0681 (5)
O2	0.33671 (17)	0.66164 (18)	0.29682 (10)	0.0586 (4)
C1	0.7782 (2)	0.4068 (2)	0.20059 (15)	0.0454 (5)
C2	0.8837 (2)	0.3667 (2)	0.14119 (15)	0.0508 (5)
C3	1.0208 (3)	0.3312 (3)	0.1677 (2)	0.0635 (7)
H3	1.0901	0.3046	0.1279	0.076*
C4	1.0535 (3)	0.3360 (3)	0.2543 (2)	0.0662 (7)
H4	1.1455	0.3123	0.2724	0.079*
C5	0.9519 (3)	0.3753 (3)	0.31421 (19)	0.0621 (6)
H5	0.9751	0.3784	0.3722	0.075*
C6	0.8156 (3)	0.4098 (2)	0.28694 (15)	0.0516 (5)
H6	0.7469	0.4358	0.3273	0.062*
C7	0.6357 (2)	0.4433 (2)	0.17089 (15)	0.0466 (5)
H7	0.6091	0.4202	0.1151	0.056*
C8	0.3205 (2)	0.6206 (2)	0.22245 (13)	0.0420 (5)
C9	0.1932 (2)	0.6646 (2)	0.17108 (14)	0.0428 (5)
C10	0.0723 (3)	0.7159 (3)	0.21159 (16)	0.0623 (7)

H10	0.0690	0.7228	0.2710	0.075*
C11	−0.0433 (3)	0.7567 (4)	0.1626 (2)	0.0797 (9)
H11	−0.1265	0.7907	0.1884	0.096*
C12	−0.0339 (3)	0.7463 (4)	0.07564 (19)	0.0779 (9)
H12	−0.1125	0.7749	0.0432	0.093*
C13	0.1914 (3)	0.6590 (3)	0.08281 (14)	0.0571 (6)
H13	0.2732	0.6259	0.0550	0.069*
C14	0.9423 (4)	0.3294 (4)	−0.0070 (2)	0.0921 (11)
H14A	1.0234	0.3926	−0.0041	0.138*
H14B	0.8987	0.3366	−0.0626	0.138*
H14C	0.9734	0.2325	0.0026	0.138*
H2	0.394 (3)	0.496 (3)	0.1309 (11)	0.080*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0447 (10)	0.0450 (9)	0.0430 (10)	0.0038 (8)	−0.0060 (8)	−0.0027 (8)
N2	0.0473 (10)	0.0510 (10)	0.0387 (10)	0.0048 (8)	−0.0067 (8)	−0.0073 (8)
N3	0.0612 (14)	0.118 (2)	0.0500 (13)	0.0151 (13)	−0.0096 (11)	0.0096 (13)
O1	0.0652 (10)	0.0875 (12)	0.0516 (10)	−0.0048 (9)	0.0080 (9)	−0.0131 (9)
O2	0.0685 (10)	0.0673 (10)	0.0399 (8)	0.0156 (8)	−0.0110 (8)	−0.0140 (8)
C1	0.0491 (12)	0.0404 (11)	0.0468 (12)	−0.0005 (9)	0.0015 (9)	−0.0005 (9)
C2	0.0522 (12)	0.0462 (11)	0.0538 (14)	−0.0060 (9)	0.0038 (10)	−0.0048 (10)
C3	0.0490 (13)	0.0615 (14)	0.0799 (18)	−0.0002 (11)	0.0098 (13)	−0.0091 (13)
C4	0.0479 (13)	0.0646 (16)	0.086 (2)	0.0025 (11)	−0.0054 (14)	0.0038 (14)
C5	0.0647 (15)	0.0641 (15)	0.0576 (15)	0.0002 (12)	−0.0114 (13)	0.0085 (12)
C6	0.0554 (13)	0.0517 (12)	0.0478 (13)	0.0016 (10)	0.0009 (10)	0.0040 (10)
C7	0.0519 (12)	0.0485 (11)	0.0395 (10)	0.0022 (9)	−0.0030 (10)	−0.0042 (9)
C8	0.0492 (11)	0.0405 (10)	0.0361 (11)	0.0013 (8)	−0.0022 (9)	−0.0018 (8)
C9	0.0449 (11)	0.0421 (10)	0.0414 (11)	−0.0001 (8)	−0.0009 (9)	−0.0020 (9)
C10	0.0606 (16)	0.0807 (17)	0.0458 (14)	0.0162 (12)	0.0040 (12)	−0.0068 (13)
C11	0.0526 (14)	0.111 (2)	0.076 (2)	0.0233 (14)	0.0058 (14)	−0.0014 (19)
C12	0.0502 (15)	0.114 (2)	0.0692 (19)	0.0146 (14)	−0.0098 (13)	0.0141 (17)
C13	0.0467 (13)	0.0809 (16)	0.0439 (13)	0.0107 (11)	0.0008 (10)	0.0058 (11)
C14	0.080 (2)	0.127 (3)	0.069 (2)	−0.0180 (19)	0.0249 (16)	−0.0256 (19)

Geometric parameters (Å, º)

N1—C7	1.279 (3)	C5—C6	1.379 (3)
N1—N2	1.383 (2)	C5—H5	0.93
N2—C8	1.343 (3)	C6—H6	0.93
N2—H2	0.897 (10)	C7—H7	0.93
N3—C12	1.320 (4)	C8—C9	1.489 (3)
N3—C13	1.332 (3)	C9—C13	1.378 (3)
O1—C2	1.371 (3)	C9—C10	1.378 (3)
O1—C14	1.424 (3)	C10—C11	1.375 (4)
O2—C8	1.231 (2)	C10—H10	0.93
C1—C6	1.391 (3)	C11—C12	1.362 (4)

C1—C2	1.402 (3)	C11—H11	0.93
C1—C7	1.448 (3)	C12—H12	0.93
C2—C3	1.385 (3)	C13—H13	0.93
C3—C4	1.385 (4)	C14—H14A	0.96
C3—H3	0.93	C14—H14B	0.96
C4—C5	1.380 (4)	C14—H14C	0.96
C4—H4	0.93		

C7—N1—N2	114.42 (17)	C1—C7—H7	119.3
C8—N2—N1	119.46 (17)	O2—C8—N2	123.23 (19)
C8—N2—H2	120.9 (19)	O2—C8—C9	121.28 (18)
N1—N2—H2	119.6 (19)	N2—C8—C9	115.49 (17)
C12—N3—C13	116.6 (2)	C13—C9—C10	117.4 (2)
C2—O1—C14	118.3 (2)	C13—C9—C8	122.5 (2)
C6—C1—C2	118.0 (2)	C10—C9—C8	120.1 (2)
C6—C1—C7	122.3 (2)	C11—C10—C9	118.9 (2)
C2—C1—C7	119.7 (2)	C11—C10—H10	120.6
O1—C2—C3	123.9 (2)	C9—C10—H10	120.6
O1—C2—C1	115.1 (2)	C12—C11—C10	118.8 (3)
C3—C2—C1	121.0 (2)	C12—C11—H11	120.6
C4—C3—C2	119.1 (2)	C10—C11—H11	120.6
C4—C3—H3	120.4	N3—C12—C11	124.0 (3)
C2—C3—H3	120.4	N3—C12—H12	118.0
C5—C4—C3	121.1 (2)	C11—C12—H12	118.0
C5—C4—H4	119.4	N3—C13—C9	124.3 (2)
C3—C4—H4	119.4	N3—C13—H13	117.9
C6—C5—C4	119.1 (3)	C9—C13—H13	117.9
C6—C5—H5	120.4	O1—C14—H14A	109.5
C4—C5—H5	120.4	O1—C14—H14B	109.5
C5—C6—C1	121.7 (2)	H14A—C14—H14B	109.5
C5—C6—H6	119.2	O1—C14—H14C	109.5
C1—C6—H6	119.2	H14A—C14—H14C	109.5
N1—C7—C1	121.4 (2)	H14B—C14—H14C	109.5
N1—C7—H7	119.3		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O2^{i}	0.90 (1)	2.05 (2)	2.897 (2)	157 (3)
C4—H4···O1^{ii}	0.93	2.58	3.469 (3)	160
C11—H11···N3^{iii}	0.93	2.53	3.429 (4)	164
C13—H13···N1^{i}	0.93	2.56	3.487 (3)	176

Symmetry codes: (i) $-y+1, x, z-1/4$; (ii) $y+1, -x+1, z+1/4$; (iii) $y-1, -x+1, z+1/4$.