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2-Methoxybenzaldehyde isonicotinoylhydrazone

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.011 Å
R factor = 0.144
wR factor = 0.278
Data-to-parameter ratio = 13.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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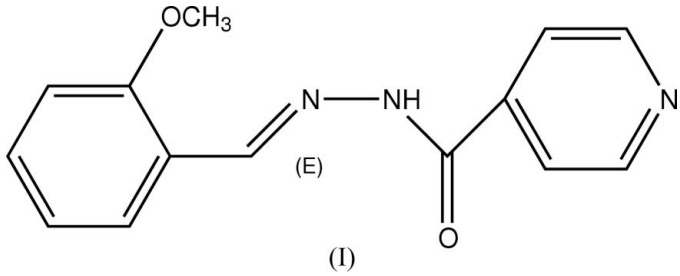
The title molecule, $C_{14}H_{13}N_3O_2$, adopts a *trans* configuration with respect to the C=N double bond. There are two molecules in the asymmetric unit. The dihedral angles between the two rings are 39.1 (4) and 19.7 (4)°. In the crystal structure, intermolecular N—H···O and N—H···N hydrogen bonds generate a network structure.

Comment

The background to this study is described in the previous paper (Xie *et al.*, 2006). As an extension of work on the structural characterization of hydrazone Schiff base compounds, we report here the crystal structure of (I), a new isonicotinohydrazone with 2-methoxybenzaldehyde.



(I)

In the title compound, (I), which crystallizes with two unique molecules in the asymmetric unit (Fig. 1), the C—N bonds in the hydrazone units are characteristically short (Table 1) because of conjugation effects. All other bond lengths are within normal ranges (Allen *et al.*, 1987). The dihedral angles between the benzene and pyridine rings are 39.1 (4)° (C1/C2/C3/N1/C4/C5 with C8/C9/C10/C11/C12/C13) and 19.7 (4)° (C15/C16/C17/N4/C18/C19 with C22/C23/C24/C25/C26/C27); these are slightly larger than normal (Fun *et al.*, 1997) due to the steric effect of the C13 and C27 methoxy substituents. The crystal structure is stabilized by intermolecular N—H···O and N—H···N hydrogen bonds (Table 2 and Fig. 2)

Experimental

2-Methoxybenzaldehyde (0.2 mmol, 27.2 mg) and isonicotinohydrazide (0.2 mmol, 27.4 mg) were dissolved in methanol (10 ml). The mixture was stirred at room temperature for about 10 min. to give a clear yellow solution. The solution was set aside for 8 d to allow slow evaporation of the solvent. Large colourless plate-shaped crystals separated from the solution; these were collected and washed three times with water.

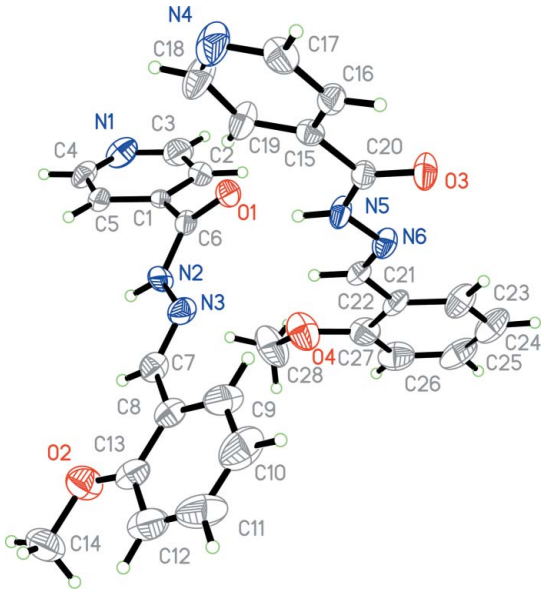


Figure 1
The structure of the asymmetric unit of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

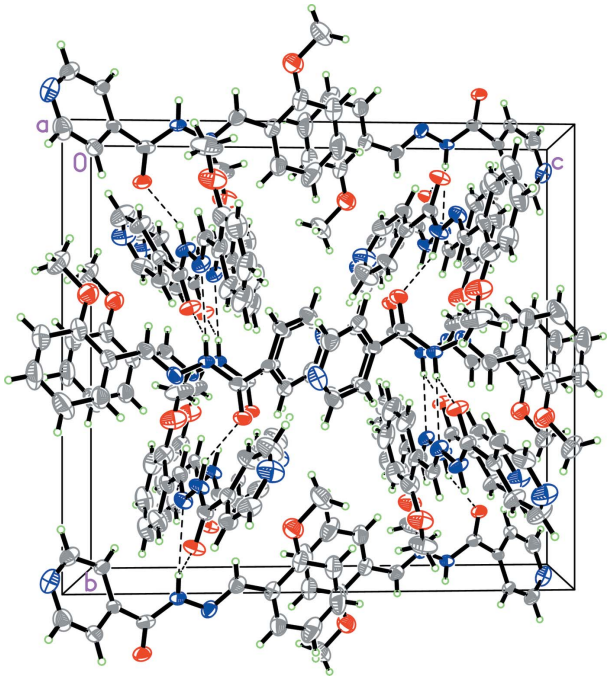


Figure 2
The crystal packing of (I), viewed along the *a* axis. Dashed lines indicate intermolecular hydrogen bonds.

Crystal data

$C_{14}H_{13}N_3O_2$
M_r = 255.27
Monoclinic, $P2_1/c$
a = 9.766 (2) Å
b = 15.935 (4) Å
c = 17.428 (4) Å
β = 95.404 (4)°
V = 2700.1 (10) Å^3
Z = 8
D_x = 1.256 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1047 reflections
θ = 4.9–35.8°
μ = 0.09 mm^{-1}
T = 298 (2) K
Plate, colourless
0.40 × 0.20 × 0.10 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.966, T_{max} = 0.991
13018 measured reflections
4742 independent reflections
2711 reflections with $I > 2\sigma(I)$
R_{int} = 0.090
θ_{max} = 25.0°
$h = -11 \rightarrow 11$
$k = -17 \rightarrow 18$
$l = -20 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.144
$wR(F^2)$ = 0.278
S = 1.24
4742 reflections
345 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0652P)^2 + 1.9118P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.007
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C6	1.221 (6)	N5—C20	1.327 (7)
O3—C20	1.216 (6)	N5—N6	1.364 (6)
N2—C6	1.340 (8)	N6—C21	1.269 (7)
N2—N3	1.366 (7)		
C6—N2—N3	120.8 (5)	C21—N6—N5	115.1 (5)
C7—N3—N2	113.8 (5)	O1—C6—N2	124.1 (7)
C20—N5—N6	119.6 (5)	O3—C20—N5	122.9 (6)
C6—N2—N3—C7	−178.0 (6)	C7—C8—C13—O2	−0.3 (10)
C20—N5—N6—C21	−178.3 (6)	C21—C22—C27—O4	−1.0 (10)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O3^{i}	0.86	2.09	2.825 (6)	143
N2—H2A···N6^{i}	0.86	2.55	3.294 (7)	146
N5—H5A···O1	0.86	2.07	2.800 (6)	142

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

All H atoms were placed in geometrically idealized positions [C—H = 0.93 (aromatic H atoms) or 0.96 Å (methyl H atoms); N—H 0.86 Å], and refined as riding atoms, with U_{iso}(H) = 1.2U_{eq}(aromatic C,N) or 1.5U_{eq}(methyl C).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); 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, 2000); software used to prepare material for publication: *SHELXL97*.

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References

Allen, F. H., Kennard, O.,Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (2000). *SHELXTL*. Version 6.1. Bruker AXS Inc., Madison, Wisconsin, USA.

Bruker (2002). *SADABS*, *SAINT* and *SMART*. Bruker AXS Inc., Madison, Winconsin.
Fun, H. K., Lu, Z. L., Duan, C. Y., Tian, Y. P., You, X. Z., Guo, Y. M., Gong, X. Y. (1997). *Acta Cryst.* C**53**, 1452–1454.
Sheldrick G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Xie, Y.-H., Sheng, L.-Q., Zhu, W. & Shao, S.-C. (2006). *Acta Cryst.* E**62**, o26–o27.