N′-(3-Ethoxy-4-hydroxybenzylidene)-isonicotinohydrazide

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Key indicators

Single-crystal X-ray study

$T = 298$ K

Mean σ(C–C) = 0.002 Å

R factor = 0.035

wR factor = 0.102

Data-to-parameter ratio = 15.0

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

The molecule of the title Schiff base compound, $C_{15}H_{15}N_3O_3$, is non-planar. The pyridine ring is twisted with respect to the rest of the molecule with a dihedral angle of 24.48 (4)°. Besides N—H···O and O—H···N hydrogen bonding, C—H···π interactions occur in the crystal structure.

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 study of the coordination chemistry of Schiff bases, we have synthesized the title compound, (I), and determined its crystal structure.

(I)
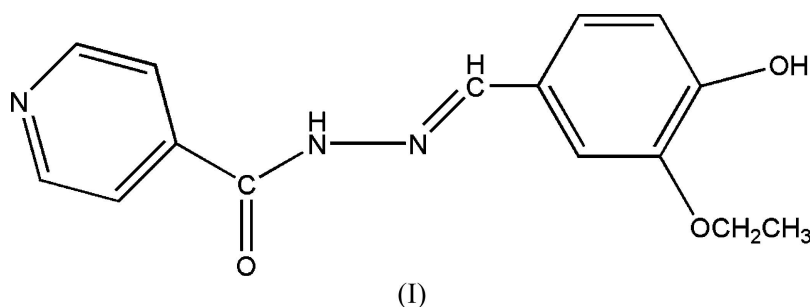
The molecular structure of (I) is shown in Fig. 1. The molecule is non-planar, the pyridine ring being twisted with respect to the rest of the molecule with a dihedral angle of 24.48 (4)°. The C7—N2 bond distance of 1.2755 (16) Å is typical for a C=N double bond.
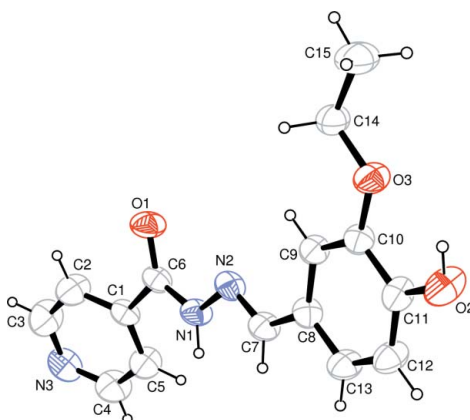

Figure 1
The molecular structure of (I) with 50% probability displacement ellipsoids (H atoms shown as small spheres of arbitrary radii).

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Classical N—H···O and O—H···N hydrogen bonding (Table 1) occurs between neighboring molecules. A C—H···π interaction is also observed. H14A···Cg^i = 2.69 Å, C14···Cg^i = 3.4713 (16) Å and C14—H14A···Cg^i = 138° [Cg is the centroid of the C8-containing benzene ring; symmetry code: (i) $x, \frac{1}{2} - y, \frac{1}{2} + z$].

Experimental

Pyridine-4-carboxylic acid hydrazide (1 mmol, 0.137 g) was dissolved in anhydrous ethanol (5 ml) and the mixture was stirred for several minutes at 351 K. 3-Ethoxy-4-hydroxybenzaldehyde (1 mmol, 0.166 g) in ethanol (5 ml) was added dropwise and the mixture was refluxed for 3 h. The solid product was filtered off and recrystallized from a methanol solution. Single crystals of (I) were obtained after 2 d.

Crystal data

$C_{15}H_{15}N_3O_3$	$Z = 4$
$M_r = 285.30$	$D_x = 1.321$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 13.631$ (3) Å	$\mu = 0.09$ mm^{-1}
$b = 12.075$ (2) Å	$T = 298$ (2) K
$c = 8.777$ (2) Å	Block, yellow
$\beta = 96.70$ (3)°	$0.42 \times 0.35 \times 0.27$ mm
$V = 1434.8$ (5) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	2970 independent reflections
	2304 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.042$
Absorption correction: none	$\theta_{max} = 26.5°$
11261 measured reflections	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0488P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.035$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.102$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.08$	$\Delta\rho_{max} = 0.23$ e Å^{-3}
2970 reflections	$\Delta\rho_{min} = -0.14$ e Å^{-3}
198 parameters	Extinction correction: SHELXL97
H atoms treated by a mixture of independent and constrained refinement	Extinction coefficient: 0.013 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^i	0.903 (9)	2.134 (12)	2.9582 (15)	151.3 (15)
O2—H2A···N3^{ii}	0.868 (9)	1.927 (11)	2.7512 (16)	158.0 (16)

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

Methyl H atoms were placed in calculated positions, with C—H = 0.96 Å, and the torsion angle was refined with U_{iso}(H) = 1.5U_{eq}(C). H atoms attached to the N and O atoms were located in a difference Fourier map and refined freely with fixed U_{iso}(H) = 0.08 Å^2. Other H atoms were placed in calculated positions with C—H = 0.93 (aromatic) and 0.97 Å (methylene), and refined in riding mode with U_{iso}(H) = 1.2U_{eq}(C).

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 (Sheldrick, 2000); software used to prepare material for publication: SHELXTL.

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