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(*E*)-*N*′-(3-Hydroxy-4-methoxybenzylidene)isonicotinohydrazide

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.044
wR factor = 0.085
Data-to-parameter ratio = 14.9

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

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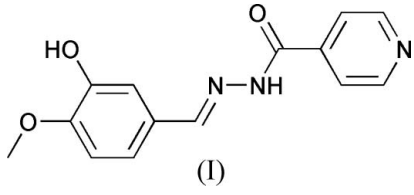
The molecule of the title compound, $C_{14}H_{13}N_3O_3$, is non-planar. The isovanillin group is almost perpendicular to the pyridine ring, the dihedral angle being 85.26 (7)°. An intermolecular N—H···O hydrogen bond and a bifurcated intra/intermolecular O—H···O,N hydrogen-bond system help to establish the molecular conformation and consolidate the crystal packing.

Comment

Metal complexes based on Schiff bases have attracted much attention because of their biological activity (Kahwa *et al.*, 1986). One of the aims of investigating the structural chemistry of Schiff bases is to develop protein and enzyme mimics (Santos *et al.*, 2001). As part of an investigation of the coordination properties of Schiff bases functioning as ligands, we report the synthesis and structure of the title compound, (I) (Fig. 1). In (I), the isovanillin group (C8–C13/O1/O2) is planar, with an r.m.s. deviation from the mean plane of 0.0048 Å, and is almost perpendicular to the pyridine ring (C1–C5/N3), the dihedral angle being 85.26 (7)°. All bond lengths and angles for (I) (Table 1) are within normal ranges. An intermolecular N—H···O hydrogen bond and a bifurcated intra/intermolecular O—H···O,N hydrogen-bond system are found in (I) (Table 2). The intramolecular bond stabilizes the conformation of the molecule, while the intermolecular bonds help to consolidate the crystal packing (Fig. 2).



(I)

Experimental

An anhydrous ethanol solution of 3-hydroxy-4-methoxybenzaldehyde (1.52 g, 10 mmol) was added to an anhydrous ethanol solution of isonicotinohydrazide (1.37 g, 10 mmol) and the mixture

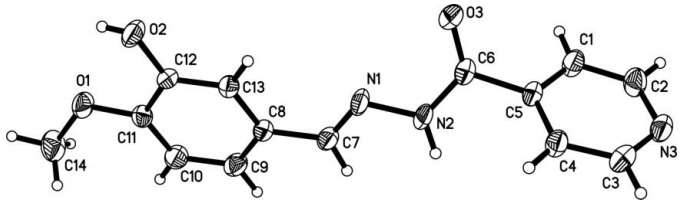


Figure 1
The structure of (I), with displacement ellipsoids for non-H atoms drawn at the 30% probability level.

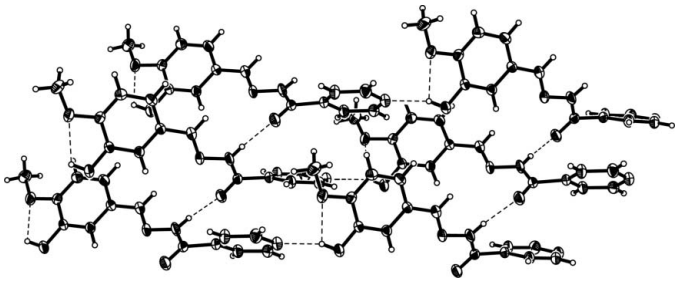


Figure 2
Intermolecular hydrogen-bonding interactions (dashed lines) in (I).

was stirred at 350 K for 5 h under nitrogen. A pale-yellow product precipitated, and was then isolated, recrystallized from ethanol and dried in a vacuum to give the pure compound in 81% yield. Colorless single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an acetonitrile solution.

Crystal data

$C_{14}H_{13}N_3O_3$
M_r = 271.27
Monoclinic, $P2_1/c$
a = 11.516 (3) Å
b = 15.440 (4) Å
c = 7.977 (2) Å
β = 109.548 (5)°
V = 1336.5 (6) Å^3
Z = 4
D_x = 1.348 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1034 reflections
θ = 2.6–21.8°
μ = 0.10 mm^{-1}
T = 293 (2) K
Block, colorless
0.22 × 0.14 × 0.10 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.972, T_{max} = 0.990
7497 measured reflections
2738 independent reflections
1222 reflections with $I > 2\sigma(I)$
R_{int} = 0.072
θ_{max} = 26.4°
$h = -8 \rightarrow 14$
$k = -19 \rightarrow 19$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.085
S = 1.02
2738 reflections
184 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.02P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0047 (8)

Table 1
Selected geometric parameters (Å, °).

O1—C11	1.366 (2)	N1—N2	1.396 (2)
O1—C14	1.429 (2)	N2—C6	1.346 (3)
O2—C12	1.359 (2)	N3—C3	1.322 (3)
O3—C6	1.220 (2)	N3—C2	1.330 (3)
N1—C7	1.274 (3)		
C11—O1—C14	117.99 (17)	N3—C3—C4	123.6 (2)
C7—N1—N2	114.52 (19)	N2—C6—C5	113.5 (2)
C6—N2—N1	118.69 (18)	N1—C7—C8	122.0 (2)
N3—C2—C1	123.7 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···N3^{i}	0.82	1.96	2.744 (3)	159
O2—H2···O1	0.82	2.28	2.686 (2)	111
N2—H2A···O3^{ii}	0.86	2.11	2.963 (3)	174

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

H atoms were included in calculated positions and refined using a riding model approximation [C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C) for aromatic CH; C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C) for methyl CH_3; O—H = 0.82 Å and U_{iso}(H) = 1.5U_{eq}(O) for OH; and N—H = 0.86 Å and U_{iso}(H) = 1.2U_{eq}(N) for NH].

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

References

Bruker (1999). *SMART* (Version 5.0) and *SAINT* (Version 4.0) for Windows NT. Bruker AXS Inc., Madison, Wisconsin, USA.
Kahwa, I. A., Selbin, J., Hsieh, T. C.-Y. & Laine, R. A. (1986). *Inorg. Chim. Acta*, **118**, 179–185.
Santos, M. L. P., Bagatin, I. A., Pereira, E. M. & Ferreira, A. M. D. C. (2001). *J. Chem. Soc. Dalton Trans.* pp. 838–844.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*a*). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Version 5.10 for Windows NT. Bruker AXS Inc., Madison, Wisconsin, USA.