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3-Indolylformaldehyde isonicotinoylhydrazone methanol solvate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.107
Data-to-parameter ratio = 13.0

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

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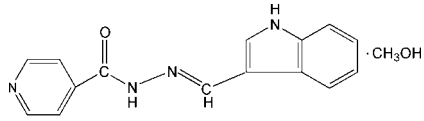
The title compound, $C_{15}H_{12}N_4O\cdot CH_4O$, an aroylhydrazone, has been synthesized. The C═O bond length is 1.2302 (18) Å, which suggests that the title compound is in the keto form. The C═N double bond has a length of 1.2789 (19) Å. In the crystal structure, the molecules are stabilized by N—H···N, N—H···O and O—H···O hydrogen bonds.

Comment

The chemical and pharmacological properties of aroylhydrazones have been investigated extensively, owing to their chelating ability with metal ions and to their potentially beneficial chemical and biological activities, such as magnetic (Bu *et al.*, 2001; Zhang *et al.*, 1996), antitumor, antineoplastic, antibacterial and antimalarial (Liao *et al.*, 2000; Fun *et al.*, 1996; Lu *et al.*, 1996). As part of our studies of the synthesis and characterization of these compounds, we report here the synthesis and crystal structure of 3-indolylformaldehyde isonicotinoylhydrazone methanol solvate, (I).



(I)

The C═O bond length is 1.2302 (18) Å, indicating that the molecule is in the keto form. The configuration of the N3—C7 bond is *E* (Fig. 1). The bonds distances and angles in this structure are normal and the molecule is practically planar. As the distances for C7═N3 and C6═O1 are 1.2789 (19) and 1.2302 (18) Å, respectively, typical for double bonds, this is a novel kind of aroylhydrazone. In the crystal structure, the molecules are stabilized by N—H···N, N—H···O and O—H···O hydrogen bonds (Table 1 and Fig. 2).

Experimental

A solution of 3-indolylformaldehyde (10 mmol) in 50 ml ethanol was added to a solution of isonicotinoyl hydrazine (10 mmol) in 10 ml ethanol. The reaction mixture was refluxed for 4 h with stirring, then the resulting pale yellow precipitate was obtained by filtration, washed several times with ethanol and dried *in vacuo* (yield 90%). Analysis calculated for the title compound ($C_{16}H_{16}N_4O_2$): C 64.86, H 5.41, N 18.92%; found: C 64.66, H 5.40, N 18.88%. IR (KBr, cm^{-1}): 3340 (–OH), 1657 (C═O), 1641 (C═N), 1599. A methanol solution of the title compound was slowly evaporated and pale yellow crystals were obtained after three weeks.

Crystal data

$C_{15}H_{12}N_4O\cdot CH_4O$	D_x = 1.321 Mg m^{-3}
M_r = 296.33	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 694 reflections
a = 10.520 (2) Å	θ = 2.5–23.6°
b = 14.020 (3) Å	μ = 0.09 mm^{-1}
c = 11.146 (3) Å	T = 293 (2) K
β = 115.02 (1)°	Plate, pale yellow
V = 1489.7 (6) Å^3	0.3 × 0.2 × 0.2 mm
Z = 4	

Data collection

Bruker SMART Apex CCD diffractometer	1836 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.048
Absorption correction: none	θ_{max} = 25.0°
7186 measured reflections	$h = -12 \rightarrow 12$
2621 independent reflections	$k = -16 \rightarrow 12$
	$l = -13 \rightarrow 11$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0567P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.040	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.107	$\Delta\rho_{max}$ = 0.14 e Å^{-3}
S = 0.96	$\Delta\rho_{min}$ = −0.15 e Å^{-3}
2621 reflections	Extinction correction: *SHELXL97*
202 parameters	Extinction coefficient: 0.017 (2)
H-atom parameters constrained	

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4A···N1^{i}	0.86	2.15	2.9491 (19)	155
N2—H2D···O2	0.86	2.15	2.934 (2)	152
O2—H2E···O1^{ii}	0.82	1.97	2.7751 (18)	168

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

The positions of all H atoms were fixed geometrically and refined isotropically using a riding model.The bond lengths for C—H are in the range 0.93–0.96 Å, The bond lengths for N—H and O—H are 0.86 and 0.82 Å, respectively.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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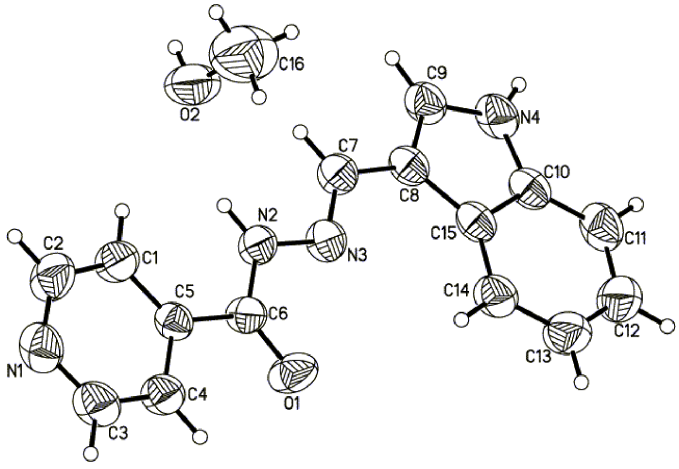

Figure 1
A view of the molecular structure, with labelling of the non-H atoms and 30% probability ellipsoids.

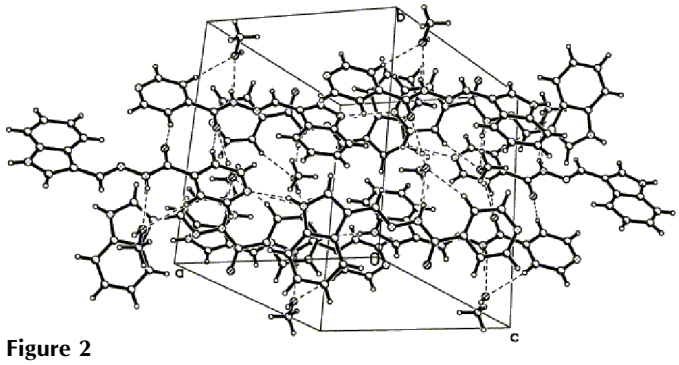

Figure 2
A view of the packing. Hydrogen bonds are indicated by dashed lines.

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