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(*E*)-4-(Isonicotinoylhydrazono)pentanoic acid monohydrate

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Key indicators

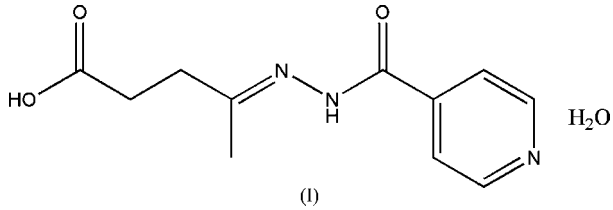
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.080
wR factor = 0.185
Data-to-parameter ratio = 13.9

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

The title compound, $C_{11}H_{13}N_3O_3\cdot H_2O$, was synthesized by the reaction of acetopropanoic acid and isonicotinoylhydrazide in ethanol. A three-dimensional network of intermolecular O—H···O, O—H···N and N—H···O hydrogen bonds stabilizes the crystal structure.

Comment

Hydrazine and its derivatives exhibit biological and pharmacological activities (Vicini *et al.*, 2002; Maccari *et al.*, 2005). These compounds are also used as synthetic intermediates (Rollas *et al.*, 2002). The title compound, (I), was synthesized in our laboratory by the reaction of acetopropanoic acid and isonicotinoylhydrazide in an ethanol medium. We present here its crystal structure (Fig. 1).



(I)

The bond lengths (Table 1) and angles show normal values. The uncoordinated water molecule plays an important role in the formation of a three-dimensional network of intermolecular O—H···O and N—H···O hydrogen bonds (Table 2), which stabilize the crystal structure (Fig. 2). O—H···N interactions are also present.

Experimental

A mixture of acetopropanoic acid (1.16 mg, 10 mmol) and isonicotinoylhydrazide (1.37 mg, 10 mmol) was refluxed in ethanol for 3 h. After cooling, the mixture was filtered and dried. The title compound was recrystallized from a mixed solvent of methanol and water (1:5) in 85% yield (200 mg). Block-shaped colourless single crystals suitable for X-ray diffraction were obtained. Analysis found (%): C 52.03, H 5.96, N 16.66; $C_{11}H_{15}N_3O_4$ requires (%): C 52.17, H 5.97, N 16.59.

Crystal data

$C_{11}H_{13}N_3O_3\cdot H_2O$
M_r = 253.26
Monoclinic, $P2_1/c$
a = 9.7834 (14) Å
b = 12.4798 (17) Å
c = 10.0924 (15) Å
β = 92.447 (3)°
V = 1231.1 (3) Å^3
Z = 4
D_x = 1.366 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6637 reflections
θ = 2–52°
μ = 0.11 mm^{-1}
T = 293 (2) K
Block, colourless
0.30 × 0.30 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.969, T_{max} = 0.979
6637 measured reflections
2425 independent reflections
1674 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 26.0°
$h = -5 \rightarrow 12$
$k = -15 \rightarrow 14$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.080
$wR(F^2)$ = 0.185
S = 1.23
2425 reflections
175 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0716P)^2 + 0.1162P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Table 1
Selected bond lengths (Å).

O1—C6	1.224 (3)	N1—C5	1.331 (4)
O2—C11	1.321 (4)	N2—C6	1.342 (3)
O3—C11	1.204 (3)	N2—N3	1.396 (3)
N1—C1	1.325 (4)	N3—C7	1.273 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1*W*—H1*WA*···O1^{i}	0.86 (5)	1.92 (5)	2.769 (4)	171 (4)
N2—H2···O1*W*	0.86	2.09	2.869 (3)	151
O1*W*—H1*WB*···O2^{ii}	0.86 (5)	2.04 (5)	2.887 (3)	168 (4)
O2—H2*C*···N1^{iii}	0.85 (4)	1.81 (4)	2.647 (4)	170 (4)

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

All H atoms were initially located in a difference Fourier map. The O-bound H atoms were refined isotropically. All other H atoms were placed in geometrically idealized positions and refined as riding, with N—H = 0.86 Å, C—H = 0.93–0.97 Å and U_{iso}(H) = 1.2U_{eq} of the parent atom.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); 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, 1999); software used to prepare material for publication: *SHELXTL*.

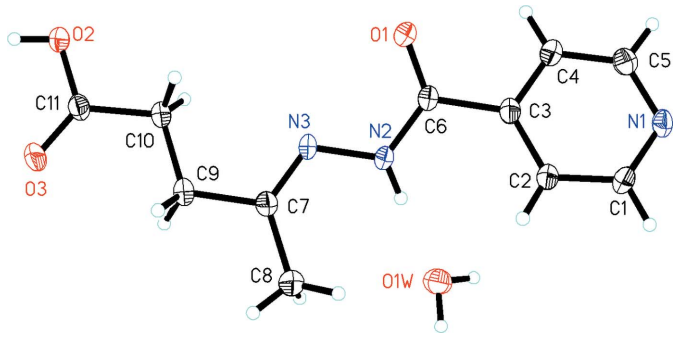


Figure 1
View of (I), showing displacement ellipsoids drawn at the 30% probability level. H atoms are represented by circles of arbitrary size.

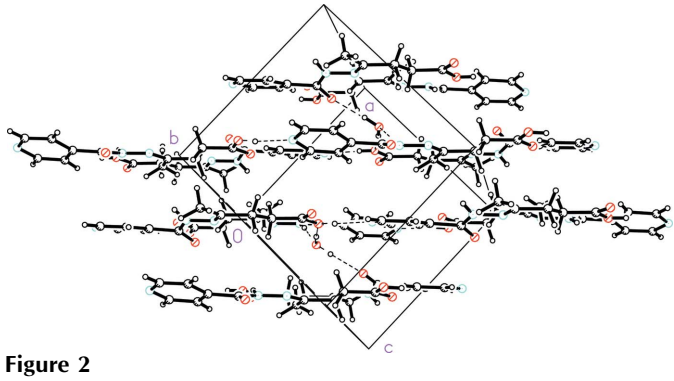


Figure 2
Packing diagram, showing the intermolecular hydrogen bonds as dashed lines.

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