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Crystal structure of 2′-hydroxyacetophenone nicotinoylhydrazone monohydrate, $C_{14}H_{15}N_3O_3 \cdot H_2O$

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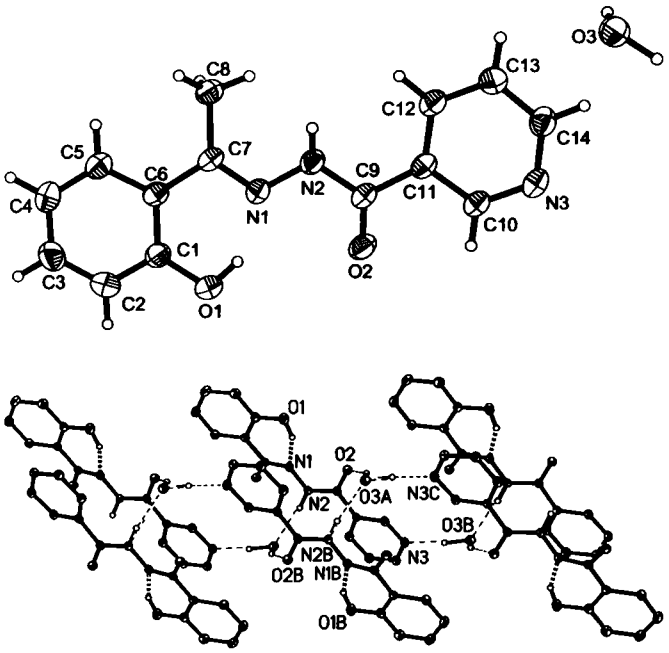

Abstract

$C_{14}H_{15}N_3O_3$, triclinic, $P\bar{1}$ (no. 2), a = 8.680(2) Å, b = 8.877(2) Å, c = 9.273(2) Å, α = 81.28(3)°, β = 86.26(3)°, γ = 70.48(3)°, V = 665.5 Å^3, Z = 2, $R_{gt}(F)$ = 0.064, $wR_{ref}(F^2)$ = 0.177, T = 291 K.

Source of material

All commercially available reagents were used as supplied. The title compound was prepared by a method similar to that of [1]. A solution of 2′-hydroxyacetophenone (10 mmol) in ethanol (10 ml) was added slowly to a solution of nicotinic acid hydrazide (10 mmol) in ethanol. The reaction mixture was refluxed for 4 h with stirring, then the resulting precipitate was collected by filtration, washed several times with ethanol and dried in vacuo (yield 62 %). A ethanol/methanol (1:1, *v*/*v*) solution of the title compound was slowly evaporated and colorless prismatic crystals were obtained after a week.

Elemental analysis – found: C, 65.67 %; H, 5.38 %; N, 16.41%; calc. for $C_{14}H_{15}N_3O_3$: C, 65.88 %; H, 5.10 %; N, 16.47 %. Analytical data are available in the CIF.

Experimental details

The H atoms bonded to C atoms were added geometrically and refined under using of rigid body approximation. The H atoms attached to N or O atoms were refined freely due to their role in the formation of hydrogen bonds.

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Discussion

Aroylhydrazones ligands and their complexes with metal ions have received considerable attention over the past two decades [2,3], This may be attributed to unusual structural features in the resultant metal complexes and their biological activity [4,5]. The chemical and pharmacological properties of aroylhydrazones have been investigated extensively, owing to their chelating ability with metal ions and to their potentially beneficial properties, such as magnetic, antitumor, antineoplastic and antibacterial [6]. The crystal structure of the title compound is built up by the 2′-hydroxyacetophenone nicotinic acid hydrazone and H_2O molecules. In the title compound, the C=O bond length is 1.231(3) Å, which suggests that the title compound is in the *keto* form (figure, top). The bonds distances and angles in this structure agree with that reported in [7]. The molecules show one strong intramolecular hydrogen bond with d(O1···N1) = 2.551(3) Å. Through the intermolecular hydrogen bonds (d(O···O) = 2.876(4) Å, d(O···N) = 2.902(4) Å and d(N···O) = 2.922(4) Å) present between the water O atom and the O and N atoms of 2′-hydroxyacetophenone nicotinic acid hydrazone, the molecules are assembled into 2D layers stacked along the a axis (figure, bottom).

Table 1. Data collection and handling.

Crystal:	colorless, prismatic, size 0.17 × 0.18 × 0.20 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.98 cm^{-1}
Diffractometer, scan mode:	Rigaku R-AXIS IV, ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1937, 1937
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1420
$N(param)_{refined}$:	193
Program:	SHELXTL [8]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(2E)	2*i*	0.582(4)	0.687(4)	0.359(3)	0.059(9)
O(3)	2*i*	0.2203(3)	0.4627(3)	−0.2600(3)	0.0659(7)
H(2A)	2*i*	0.6527	0.8647	1.0220	0.086
H(3A)	2*i*	0.8943	0.9153	1.0198	0.093
H(4A)	2*i*	1.0451	0.9225	0.8071	0.091
H(5A)	2*i*	0.9496	0.8875	0.5942	0.074
H(8A)	2*i*	0.7081	0.8459	0.3180	0.091
H(8B)	2*i*	0.8739	0.8103	0.3962	0.091
H(8C)	2*i*	0.7440	0.9838	0.3827	0.091
H(10A)	2*i*	0.1292	0.5952	0.4295	0.067
H(12A)	2*i*	0.4624	0.7043	0.1553	0.077
H(13A)	2*i*	0.3470	0.6302	−0.0281	0.084
H(14A)	2*i*	0.1288	0.5425	0.0224	0.076
H(1E)	2*i*	0.501(5)	0.782(5)	0.729(5)	0.11(2)
H(3E)	2*i*	0.200(5)	0.555(5)	−0.306(4)	0.09(1)
H(3F)	2*i*	0.088(8)	0.472(8)	−0.257(6)	0.19(2)

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	2*i*	0.5665(3)	0.7665(3)	0.5528(3)	0.044(1)	0.057(2)	0.056(1)	−0.020(1)	0.000(1)	−0.008(1)
N(2)	2*i*	0.5104(3)	0.7310(3)	0.4293(3)	0.041(2)	0.061(2)	0.058(2)	−0.019(1)	0.006(1)	−0.014(1)
N(3)	2*i*	0.1055(3)	0.5624(3)	0.2302(3)	0.046(2)	0.065(2)	0.076(2)	−0.025(1)	0.000(1)	−0.008(1)
O(1)	2*i*	0.5124(3)	0.8071(4)	0.8202(3)	0.064(2)	0.113(2)	0.060(2)	−0.044(2)	0.010(1)	−0.007(1)
O(2)	2*i*	0.2787(3)	0.7294(3)	0.5547(2)	0.059(1)	0.079(2)	0.069(1)	−0.035(1)	0.018(1)	−0.023(1)
C(1)	2*i*	0.6527(4)	0.8427(4)	0.8082(3)	0.050(2)	0.056(2)	0.060(2)	−0.019(2)	0.001(1)	−0.002(2)
C(2)	2*i*	0.7122(5)	0.8682(5)	0.9352(4)	0.079(3)	0.082(3)	0.055(2)	−0.030(2)	0.002(2)	−0.003(2)
C(3)	2*i*	0.8565(5)	0.8985(5)	0.9341(4)	0.091(3)	0.089(3)	0.065(2)	−0.045(2)	−0.015(2)	−0.010(2)
C(4)	2*i*	0.9457(5)	0.9040(5)	0.8071(4)	0.065(2)	0.090(3)	0.084(2)	−0.040(2)	−0.007(2)	−0.013(2)
C(5)	2*i*	0.8885(4)	0.8823(4)	0.6799(4)	0.056(2)	0.066(2)	0.069(2)	−0.028(2)	0.005(2)	−0.010(2)
C(6)	2*i*	0.7401(4)	0.8527(3)	0.6763(3)	0.048(2)	0.043(2)	0.056(2)	−0.016(1)	0.004(1)	−0.006(1)
C(7)	2*i*	0.6832(3)	0.8257(3)	0.5385(3)	0.041(2)	0.042(2)	0.059(2)	−0.014(1)	0.008(1)	−0.007(1)
C(8)	2*i*	0.7591(4)	0.8705(4)	0.3961(3)	0.062(2)	0.064(2)	0.063(2)	−0.030(2)	0.012(2)	−0.015(2)
C(9)	2*i*	0.3643(4)	0.7061(3)	0.4437(3)	0.041(2)	0.050(2)	0.063(2)	−0.018(1)	0.005(1)	−0.007(2)
C(10)	2*i*	0.1737(4)	0.6034(4)	0.3358(3)	0.044(2)	0.061(2)	0.065(2)	−0.022(2)	0.004(2)	−0.007(2)
C(11)	2*i*	0.3078(3)	0.6578(3)	0.3148(3)	0.041(2)	0.047(2)	0.061(2)	−0.015(1)	0.005(1)	−0.008(1)
C(12)	2*i*	0.3732(4)	0.6682(4)	0.1751(4)	0.053(2)	0.080(2)	0.067(2)	−0.032(2)	0.010(2)	−0.016(2)
C(13)	2*i*	0.3045(4)	0.6245(5)	0.0664(4)	0.059(2)	0.094(3)	0.062(2)	−0.029(2)	0.008(2)	−0.019(2)
C(14)	2*i*	0.1732(4)	0.5724(4)	0.0976(4)	0.051(2)	0.068(2)	0.073(2)	−0.020(2)	−0.001(2)	−0.016(2)

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