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The crystal structure of (*E*)-*N'*-(butan-2-ylidene)isonicotinohydrazide 0.5 hydrate $C_{10}H_{13}N_3O \cdot 0.5H_2O$

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Abstract

$C_{10}H_{13}N_3O \cdot 0.5H_2O$, orthorhombic, *Aba2* (no. 41), $a = 19.9676(19)$ Å, $b = 8.4674(7)$ Å, $c = 12.4082(10)$ Å, $V = 2097.9(3)$ Å³, $Z = 8$, $R_{gt}(F) = 0.0299$, $wR_{ref}(F^2) = 0.0849$, $T = 173$ K.

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The asymmetric unit of the title structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

All reagents used were purchased and used without further purification. Isonicotinic acid hydrazide (isoniazid) (1.0 g; 7.29 mmol) and 15.0 mL of 2-butanone were added to a

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.91 × 0.79 × 0.14 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ:	0.09 mm ⁻¹
Diffractometer, scan mode:	Bruker D8 Venture Photon, ω
θ _{max} , completeness:	28.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	58009, 2526, 0.040
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 2474
$N(param)_{refined}$:	138
Programs:	Bruker [1], SHELX [2], WinGX [3], PLATON [4], ORTEP-III [5]

25 mL stainless steel jar containing 2 × 7 mm stainless steel balls. The jar was tightly sealed and the contents were shaken for 30 min at 25 Hz using the Retsch Mixer Mill MM 200. The mixture was then transferred into a 50 ml amber Schott bottle and left slightly open to allow slow evaporation at room temperature. Colourless block crystals were observed after three days.

Experimental details

The collection method involved ω-scans of width 0.5°. Data reduction was carried out using SAINT-Plus version 6.02.6 software, and SADABS was used to process empirical absorption corrections [1]. Hydrogen atoms were allowed to ride on their respective parent atoms with $d(C-H) = 0.95$ [2]. Hydrogen atoms were positioned geometrically Å and $U_{iso}(H) = 1.2U_{eq}(C)$. Diagrams and publication material were generated using WinGX [3], PLATON [4] and Ortep [5, 6].

Comment

Isoniazid, a synthetic derivative of nicotinic acid [7] has been used as a frontline drug for the treatment of Tuberculosis (TB) for decades [8]. Tuberculosis-causing mycobacteria have developed resistance against this drug, thus there is an urgent need for novel and effective drug design and

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C1	0.33289 (7)	0.36429 (17)	0.48805 (12)	0.0191 (3)
C2	0.30391 (9)	0.2671 (2)	0.41159 (14)	0.0277 (3)
H2	0.2566	0.2608	0.4052	0.033 [*]
C3	0.34505 (11)	0.1786 (2)	0.34421 (16)	0.0359 (4)
H3	0.3247	0.1136	0.2911	0.043 [*]
C4	0.43912 (9)	0.2735 (2)	0.42588 (16)	0.0329 (4)
H4	0.4865	0.2746	0.4321	0.039 [*]
C5	0.40252 (8)	0.3686 (2)	0.49557 (14)	0.0247 (3)
H5	0.4243	0.4344	0.5468	0.03 [*]
C6	0.29301 (7)	0.47594 (17)	0.55704 (12)	0.0196 (3)
C7	0.13160 (8)	0.50920 (19)	0.65346 (13)	0.0211 (3)
C8	0.09319 (8)	0.3983 (2)	0.58217 (16)	0.0295 (4)
H8A	0.118	0.3815	0.515	0.044 [*]
H8B	0.0872	0.297	0.6192	0.044 [*]
H8C	0.0493	0.4439	0.5657	0.044 [*]
C9	0.09022 (8)	0.6141 (2)	0.72541 (13)	0.0235 (3)
H9A	0.0624	0.5461	0.7725	0.028 [*]
H9B	0.0593	0.6761	0.6795	0.028 [*]
C10	0.12885 (9)	0.7277 (2)	0.79610 (16)	0.0309 (4)
H10A	0.1608	0.7871	0.7519	0.046 [*]
H10B	0.1532	0.6684	0.8515	0.046 [*]
H10C	0.0976	0.8011	0.8307	0.046 [*]
N1	0.23250 (6)	0.42502 (15)	0.58957 (12)	0.0237 (3)
H1A	0.2167	0.3325	0.5695	0.028 [*]
N2	0.41190 (9)	0.1807 (2)	0.35055 (14)	0.0373 (4)
N3	0.19548 (7)	0.52638 (16)	0.65723 (12)	0.0238 (3)
O1	0.31550 (6)	0.60805 (13)	0.57675 (11)	0.0276 (3)
O2	0.5	0	0.20287 (16)	0.0330 (4)
H2A	0.4739 (15)	0.050 (3)	0.244 (3)	0.059 (9) [*]

development. Modifying existing drugs to target resistant organisms intend to mitigate the deactivation process by the mycobacteria [9]. The crystal structure being reported in this article represents a covalent modification of isoniazid. The compound crystallizes in the orthorhombic *Aba2* space group. The asymmetric unit shown in the figure is a hemihydrate, containing one organic target molecule and half a water molecule. The water sits in a special position in the unit cell, with a twofold rotation axis passing through the oxygen atom of the water molecule. Bond lengths and angles are all in the expected ranges [10, 11]. The crystal packing contains bilayers of (*E*)-*N'*-(butan-2-ylidene)isonicotinohydrazide alternating with layers of water molecules. Each carbohydrazide moiety is hydrogen bonded by a O2–H2a⋯N2 hydrogen bond to a water molecule to

form the hemihydrate. Each carbohydrazide moiety is also hydrogen bonded via its N1–H donor to the carbonyl oxygen (O1) acceptor of an adjacent carbohydrazide moiety. The water present in the crystal is generated as a byproduct of the Schiff base reaction between isoniazid and butanone and is not added to the reaction mixture.

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