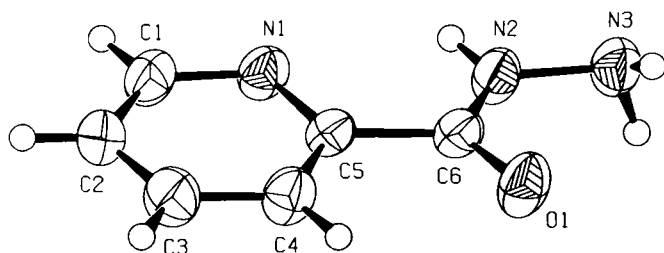


Crystal structure of picolinic acid hydrazide, C₆H₇N₃O

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Abstract

C₆H₇N₃O, monoclinic, C12/c1 (no. 15), $a = 19.222(2)$ Å, $b = 3.8987(4)$ Å, $c = 18.115(2)$ Å, $\beta = 107.095(2)^\circ$, $V = 1297.6$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.038$, $wR_{\text{ref}}(F^2) = 0.122$, $T = 293$ K.

Source of material

The title compound was synthesized by the reaction of ethyl ester of picolinic acid with hydrazine hydrate (80 %) in absolute ethanol. The whole reaction mixture was refluxed for 7 h. The excess of solvent was distilled off, the residue was filtered, washed with water and recrystallized from ethanol (60 %). Colorless thin needle-shaped single crystals suitable for X-ray analysis were obtained by slow evaporation at RT (m.p. 144–145 °C).

Experimental details

While the H atoms bound to N atoms were refined freely due to their importance in hydrogen bonding, the H atoms attached to C atoms were refined using conventional restraints.

Discussion

Aromatic hydrazides are a very important intermediate especially in the field of heterocyclic chemistry. Aromatic hydrazide have been used for the synthesis of various biologically active five membered heterocycles such as 2,5-disubstituted 1,3,4-oxadiazoles, 5-substituted 2-mercapto-1,3,4-oxadiazoles etc. Due to its versatilities in the synthesis of various heterocyclic compounds, we have synthesized the title compound and report its structure.

The r.m.s. deviation from plane in the molecular structure of the

title compound is 0.0171 Å. Within phenyl ring, the bond lengths lie between 1.374(2) Å and 1.388(2) Å which highlight the aromatic character. The valence angles N1–C1–C2 (123.7(1)°) and N1–C5–C4 (123.4(1)°) are larger than the standard value (120°). The opening of this angle is due to the presence of the nitrogen atom on 2-position, which involves a decrease of the ring angles of C2 (118.9(1)°), C3 (118.5(1)°) and C4 (118.9(1)°). The C6–O1 bond distance (1.235(1) Å) is compatible with respective distances in related structures [2] and smaller than those usually observed in carboxylic acids (1.365 Å). The structure is influenced by quite interesting intra- and intermolecular hydrogen bonding which stabilizes the whole crystal structure.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.18 × 0.20 × 0.28 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.02 cm ⁻¹
Diffractionmeter, scan mode:	Bruker SMART 1000 CCD, ω
$2\theta_{\text{max}}$:	56.46°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	3453, 1462
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1280
$N(\text{param})_{\text{refined}}$:	104
Program:	SHELXTL [3]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(2A)	8f	0.5174(8)	0.956(4)	0.178(1)	0.053(4)
H(3A)	8f	0.553(1)	0.584(6)	0.082(1)	0.082(6)
H(3B)	8f	0.5771(9)	0.911(5)	0.077(1)	0.064(5)
H(1A)	8f	0.3617	1.3371	0.2701	0.055
H(2)	8f	0.2409	1.3253	0.1991	0.055
H(3)	8f	0.2082	1.0940	0.0755	0.059
H(4)	8f	0.3002	0.8970	0.0258	0.053

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	8f	0.42436(4)	0.7346(3)	0.02353(5)	0.0453(5)	0.0707(6)	0.0357(5)	−0.0015(4)	0.0122(3)	−0.0147(4)
N(1)	8f	0.40259(5)	1.1190(3)	0.19451(5)	0.0389(5)	0.0532(6)	0.0305(5)	0.0003(4)	0.0082(3)	−0.0043(4)
N(2)	8f	0.50925(5)	0.8879(3)	0.13393(6)	0.0340(5)	0.0626(7)	0.0315(5)	0.0036(4)	0.0103(4)	−0.0044(4)
N(3)	8f	0.56853(5)	0.7600(4)	0.11009(6)	0.0384(5)	0.0701(8)	0.0417(6)	0.0100(5)	0.0148(4)	0.0007(5)
C(1)	8f	0.34908(7)	1.2431(3)	0.22071(7)	0.0512(6)	0.0559(7)	0.0334(5)	0.0043(5)	0.0155(5)	−0.0044(5)

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Table 3. Continued.

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
C(2)	8 <i>f</i>	0.27615(7)	1.2387(3)	0.17831(7)	0.0463(6)	0.0507(7)	0.0466(6)	0.0093(5)	0.0222(5)	0.0045(5)
C(3)	8 <i>f</i>	0.25680(6)	1.1040(4)	0.10495(7)	0.0338(5)	0.0645(8)	0.0476(6)	0.0031(5)	0.0090(4)	0.0004(5)
C(4)	8 <i>f</i>	0.31138(6)	0.9834(3)	0.07577(6)	0.0367(5)	0.0594(7)	0.0347(5)	−0.0024(5)	0.0078(4)	−0.0064(5)
C(5)	8 <i>f</i>	0.38284(5)	0.9939(3)	0.12239(5)	0.0340(5)	0.0383(6)	0.0287(5)	−0.0022(4)	0.0092(4)	0.0004(4)
C(6)	8 <i>f</i>	0.44071(5)	0.8601(3)	0.08897(6)	0.0370(5)	0.0410(6)	0.0302(5)	−0.0011(4)	0.0105(4)	−0.0003(4)

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