Crystal structure of 1-(2-pyridyl)-3-(4-pyridyl)1*H*-pyrazol-5-ol, $C_{13}H_{10}N_4O$

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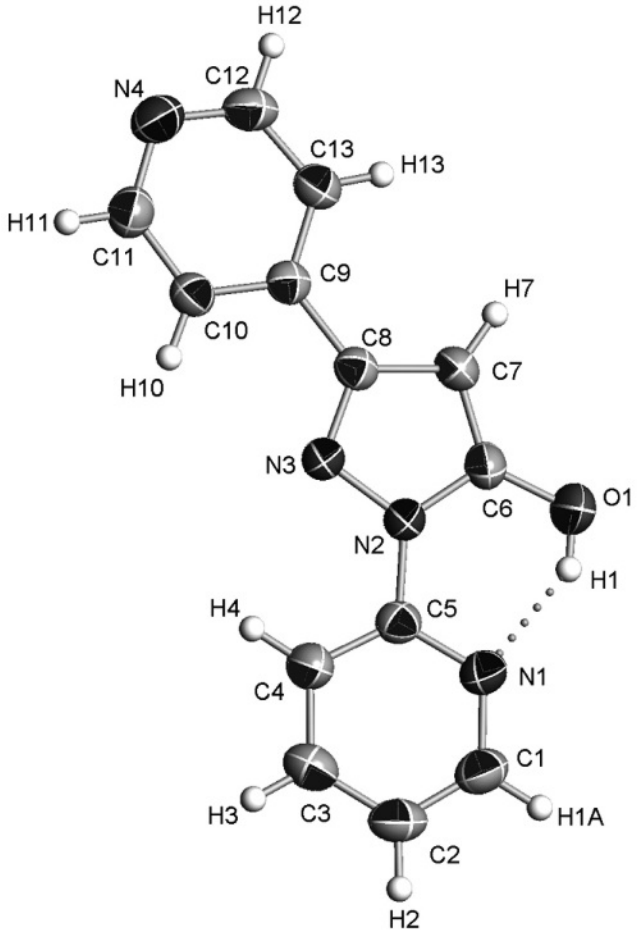
Received September 17, 2014, accepted October 13, 2014, available online October 24, 2014, CCDC no. 1020942



Abstract

$C_{13}H_{10}N_4O$, orthorhombic, $P2_12_12_1$ (no. 19), a = 3.857(2) Å, b = 11.159(4) Å, c = 25.58(1) Å, V = 1100.8 Å^3, Z = 4, $R_{gt}(F)$ = 0.0284, $wR_{ref}(F^2)$ = 0.0761, T = 296 K.

Source of material

The title compound was prepared following the reported procedure [1, 2]. 2-hydrazinylpyridine (1.09 g, 0.010 mol) was added to 20 mL of an anhydrous ethanol solution of ethyl isonicotinoylacetate (1.93 g, 0.010 mol) in a 100 mL flask. The mixture was heated at reflux and stirred for 2 h, the product washed with anhydrous ether (20 mL). After filtration, the yellow powder was dried in vacuo to constant weight and shown to be 3-phenyl-1-(pyridin-2-yl)-1*H*-pyrazol-5-ol (yield 91%). The single crystals were obtained by slow evaporation form an acetone solution at room temperature after several days.

Table 1. Data collection and handling.

Crystal:	yellow blocks, size 0.22×0.25×0.36 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.97 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6015, 2060
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1927
$N(param)_{refined}$:	165
Programs:	SHELX [3]

Experimental details

The structure was solved by direct methods with the SHELXS-97 program [3]. All H-atoms were positioned with idealized geometry and refined isotropic (U_{iso}(H) = 1.5U_{eq}(C) for hydroxy H atoms and U_{iso}(H) = 1.2U_{eq}(C) for all other H atoms) using a riding model with C–H = 0.93 Å, O–H = 0.82 Å.

Discussion

The pyrazole compounds are important targets in medical chemistry and coordination chemistry because this fragment is a key moiety in numerous biologically active compounds and extracting reagents for many metal ions. For example, they exhibit anti-anxiety, antipyretic, analgesic, anti-inflammatory properties as medicine. They have been applied in fungicides, pesticides, insecticides and dyestuffs [4–6]. Furthermore, their metal complexes are used as NMR shift-reagents and molecular precursors for supercritical fluid transport (SFT) CVD [7, 8]. In recent years, we have been engaged in the preparation of the derivatives of heterocycles for designing new heterocycles with potential biological interest. Herein we report the crystal structure of a compound which may be used as a new precursor for obtaining bioactive molecules. The title crystal structure is built by the $C_{13}H_{10}N_4O$ molecules, in which all bond lengths and angles are in normal ranges. The pyridine (N1, C1-C5) and pyridine (N4, C9-C13) rings of the title compound make dihedral angles of 2.64(8) and 3.03(8)° with the pyrazolone moiety, respectively. The clear evidence of the hydroxyl H atom in the difference Fourier synthesis and the absence of any residual electron density in the vicinity of C7 confirm that $C_{13}H_{10}N_4O$ crystallizes as a pure enol tautomer and that no desmotropism is present. In addition, there is one intramolecular hydrogen bond: O1–H1···N1, [O···N = 2.6178(18) Å, O–H···N = 144.3°].

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(10)	4*a*	0.3647	–0.1695	0.0004	0.049
H(13)	4*a*	0.8453	0.1234	–0.0541	0.051
H(12)	4*a*	0.8156	0.0415	–0.1356	0.060
H(2)	4*a*	0.1536	–0.0788	0.2998	0.064
H(1)	4*a*	0.6946	0.2099	0.1707	0.099
H(7)	4*a*	0.8429	0.2073	0.0332	0.050
H(11)	4*a*	0.3483	–0.2409	–0.0832	0.059
H(4)	4*a*	0.2663	–0.1635	0.1484	0.055
H(1A)	4*a*	0.3973	0.1038	0.2828	0.065
H(3)	4*a*	0.0860	–0.2152	0.2321	0.062

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(3)	4*a*	0.4957(3)	–0.0302(1)	0.07624(4)	0.0471(7)	0.0361(6)	0.0342(6)	–0.0006(5)	–0.0010(5)	–0.0006(4)
C(9)	4*a*	0.6080(4)	–0.0134(1)	–0.01644(5)	0.0328(7)	0.0362(6)	0.0379(7)	0.0079(5)	–0.0008(5)	0.0029(5)
N(2)	4*a*	0.5395(3)	0.04184(9)	0.11936(4)	0.0508(7)	0.0347(5)	0.0334(6)	–0.0027(5)	–0.0025(5)	–0.0008(4)
C(10)	4*a*	0.4585(4)	–0.1243(1)	–0.02664(5)	0.0455(8)	0.0368(7)	0.0406(7)	0.0020(6)	0.0025(6)	0.0032(5)
N(1)	4*a*	0.4714(4)	0.0825(1)	0.20728(4)	0.0659(9)	0.0441(7)	0.0360(6)	0.0041(6)	–0.0036(6)	–0.0007(5)
N(4)	4*a*	0.5810(4)	–0.1081(1)	–0.11853(5)	0.0614(9)	0.0583(8)	0.0423(7)	0.0049(7)	0.0013(6)	–0.0039(6)
C(5)	4*a*	0.4288(4)	0.0029(1)	0.16890(5)	0.0422(7)	0.0400(7)	0.0348(7)	0.0050(6)	–0.0037(6)	0.0020(6)
C(13)	4*a*	0.7429(4)	0.0488(1)	–0.05875(5)	0.0428(8)	0.0413(7)	0.0434(7)	0.0021(7)	0.0027(7)	0.0054(6)
C(12)	4*a*	0.7231(5)	–0.0018(1)	–0.10787(6)	0.0528(9)	0.0564(8)	0.0404(7)	0.0045(8)	0.0070(7)	0.0082(7)
C(2)	4*a*	0.2238(5)	–0.0595(2)	0.26605(6)	0.058(1)	0.063(1)	0.0388(8)	0.0096(9)	0.0040(7)	0.0088(7)
C(8)	4*a*	0.6197(4)	0.0350(1)	0.03715(5)	0.0351(7)	0.0350(6)	0.0384(7)	0.0050(5)	–0.0003(5)	0.0035(5)
O(1)	4*a*	0.7569(4)	0.23340(9)	0.14181(4)	0.104(1)	0.0458(6)	0.0483(6)	–0.0207(7)	–0.0007(7)	–0.0070(5)
C(7)	4*a*	0.7443(4)	0.1474(1)	0.05372(5)	0.0473(8)	0.0363(7)	0.0418(7)	–0.0031(7)	0.0015(7)	0.0047(6)
C(11)	4*a*	0.4507(4)	–0.1667(1)	–0.07730(6)	0.054(1)	0.0439(8)	0.0490(9)	–0.0006(7)	–0.0014(7)	–0.0055(6)
C(4)	4*a*	0.2886(4)	–0.1101(1)	0.17611(6)	0.0523(9)	0.0441(7)	0.0422(7)	–0.0047(7)	–0.0016(7)	0.0011(6)
C(6)	4*a*	0.6888(4)	0.1493(1)	0.10614(5)	0.0529(9)	0.0336(6)	0.0432(7)	–0.0029(6)	–0.0038(7)	–0.0010(6)
C(1)	4*a*	0.3686(5)	0.0498(2)	0.25547(6)	0.069(1)	0.0564(9)	0.0361(7)	0.0116(9)	–0.0016(7)	–0.0017(7)
C(3)	4*a*	0.1836(4)	–0.1404(2)	0.22576(6)	0.052(1)	0.0527(8)	0.0504(8)	–0.0041(7)	0.0016(7)	0.0121(7)

Acknowledgments. The work was supported by the Henan Province Foundation and Advanced Technology Research Program (No. 122300410387), Scientific Research Starting Foundation for Doctor of Zhoukou Normal University (No. zksybscx201106), The Key Scientific and Technological Research Project of Education Department of Henan Province (No. 14B530008), and The opening laboratory project of Zhoukou Normal University (No. K201428).

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