Molecular Crystals and Liquid Crystals

ISSN: 1542-1406 (Print) 1563-5287 (Online) Journal homepage: http://www.tandfonline.com/loi/gmcl20

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To cite this article: Wei Gao (2016) X-ray crystal structural analysis of 1,2-dihydro-3-methylpyrazole-5-one, Molecular Crystals and Liquid Crystals, 625:1, 221-224, DOI: 10.1080/15421406.2015.1069467

To link to this article: http://dx.doi.org/10.1080/15421406.2015.1069467

Published online: 19 Feb 2016.

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MOL. CRYST. LIQ. CRYST.
2016, VOL. 625, 221–224
http://dx.doi.org/10.1080/15421406.2015.1069467

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X-ray crystal structural analysis of 1,2-dihydro-3-methylpyrazole-5-one

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ABSTRACT

The title compound 1,2-dihydro-3-methylpyrazole-5-one was determined by X-ray crystal structural analysis. The crystals are monoclinic, with space group P2(1)/n with a = 7.968(7), b = 6.502(6), c = 9.986(10) Å, $\alpha = 90°$, $\beta = 109.995(15)°$, $\gamma = 90°$, V = 486.1(8) Å^3, Z = 4, F(000) = 208, D_c = 1.340 g cm^{-3}, $\mu = 0.100$ mm^{-1}, and the final R = 0.0373 and wR = 0.0961. A total of 4776 reflections were collected, of which 1148 were independent (R_{int} = 0.0589). In the crystal packing diagram, intermolecular N–H···O hydrogen bonds and π–π stacking interactions stabilize the solid state of the title compound.

KEYWORDS

Crystal structure; heterocyclic compound; pyrazole

Introduction

Recently, heterocyclic compounds comprising nitrogen, because of their wide applications in industry, medicine, and agriculture, have attracted much interest of synthetic chemists [1–8]. In continuation of our previous study on heterocyclic compounds [9–11], a heterocyclic compound containing pyrazole unit was prepared and its structure was confirmed by X-ray crystal structural analysis. In this contribution, we describe the X-ray crystal structural analysis of 1,2-dihydro-3-methylpyrazole-5-one.

Experimental

Crystal structure determination

The crystal of the title compound with dimensions of 0.20 × 0.18 × 0.10 mm was mounted on a Rigaku Saturn CCD area detector diffractometer with a graphite–monochromated MoKα radiation ($\lambda = 0.71073$ Å) by using Φ and scan modes at 113(2) K in the range of $3.81° \leq \theta \leq 27.83°$. The crystal belongs to monoclinic system with space group $P2(1)/n$ and crystal parameters of $a = 7.968(7)$ Å, $b = 6.502(6)$ Å, $c = 9.986(10)$ Å, $\alpha = 90°$, $\beta = 109.995(15)°$, $\gamma = 90°$, $V = 486.1(8)$ A^3, $D_c = 1.340$ g cm^{-3}, absorption coefficient $\mu = 0.100$ mm^{-1}, and $Z = 4$. A summary of crystal data is presented in Table 1.

The structure was solved by direct methods with SHELXS-97 [12], and refined by the full-matrix least squares method on F^2 data using SHELXL-97 [13]. The empirical absorption corrections were applied to all intensity data. H atom of N-H was initially located in a difference

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Table 1. Crystal data and structure refinement.

Empirical formula	$C_4H_6N_2O$
Formula weight	98.11
Crystal system	Monoclinic
Unit cell dimensions	
a (Å)	7.968(7)
b (Å)	6.502(6)
c (Å)	9.986(10)
Unit cell angles	
α	90°
β	109.995(15)
γ	90
Volume (Å^3)	486.1(8)
Z	4
Temperature (K)	113(2)
Space group	*P*2(1)/n
Wavelength (Å)	0.71073
Calculated density (g cm^{-3})	1.340
Absorption coefficient (mm^{-1})	0.100
F(000)	208
Crystal size	0.20 × 0.18 × 0.10 mm
θ range for data collection	3.81–27.83°
Reflections collected	4776
Independent reflections	1148 [$R_{(int)} = 0.0589$]
Final *R* indices [I > 2σ(I)]	$R_1 = 0.0373$, $wR_2 = 0.0961$

Fourier map and was refined with the restraint Uiso(H) = 1.2 Ueq(N). Other H atoms were positioned geometrically and refined using a riding model, with d(C—H) = 0.93–0.97 Å and Uiso(H) = 1.2 Ueq(C) or 1.5 Ueq(Cmethyl). The final full-matrix least squares refinement gave $R = 0.0373$ and $wR = 0.0961$.

Results and discussion

The title compound 1,2-dihydro-3-methylpyrazole-5-one has been confirmed by single crystal X-ray diffraction analysis. The selected bond lengths and bond angles are listed in Table 2. The structure was solved by direct methods. Anisotropic displacement parameters were applied to all non-hydrogen atoms in full-matrix least-square refinements based on F^2. The hydrogen atoms were set in calculated positions with a common fixed isotropic thermal parameter.

The molecular structure and the packing view of the title compound are displayed in Figs. 1 and 2, respectively. The title compound crystallizes in monoclinic space group *P*2(1)/n with four molecules in the unit cell and one molecule in the asymmetric unit. As shown in Fig. 1, the molecular structure comprises a five-membered ring with a methyl group.

Table 2. Selected bond lengths (Å) and bond angles (°).

Bond lengths			
O(1)-C(1)	1.2888(15)	N(1)-C(1)	1.3584(16)
N(1)-N(2)	1.3661(15)	N(2)-C(3)	1.3366(16)
C(1)-C(2)	1.4070(18)	C(2)-C(3)	1.3789(17)
Bond angles			
C(1)-N(1)-N(2)	109.24(9)	C(3)-N(2)-N(1)	108.63(10)
O(1)-C(1)-N(1)	121.31(10)	O(1)-C(1)-C(2)	132.33(10)
N(1)-C(1)-C(2)	106.35(10)	C(3)-C(2)-C(1)	107.11(10)
N(2)-C(3)-C(2)	108.64(11)	N(2)-C(3)-C(4)	120.32(10)

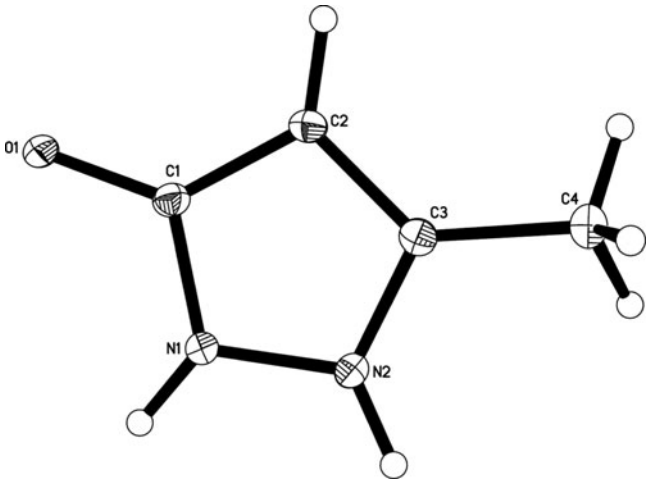

Figure 1. Molecular structure of the title compound.

The H atom of the hydroxy group is transferred to the N atom of pyrazole to form amide group. The five-membered ring C1C2C3N1N2 is almost coplanar with the mean deviation of 0.0059 Å. The bond distances [O(1)-C(1) = 1.2888(15) Å, N(1)-C(1) = 1.3584(16) Å, N(1)-N(2) = 1.3661(15) Å, N(2)-C(3) = 1.3366(16) Å, C(1)-C(2) = 1.4070(18) Å, C(2)-C(3) = 1.3789(17) Å, and C(3)-C(4) = 1.4893(18) Å] and bond angles [C(1)-N(1)-N(2) = 109.24(9)°, C(3)-N(2)-N(1) = 108.63(10)°, O(1)-C(1)-N(1) = 121.31(10)°, O(1)-C(1)-C(2) = 132.33(10)°, N(1)-C(1)-C(2) = 106.35(10)°, C(3)-C(2)-C(1) = 107.11(10)°, N(2)-C(3)-C(2) = 108.64(11)°, N(2)-C(3)-C(4) = 120.32(10)°, and C(2)-C(3)-C(4) = 131.03(10)°] are similar to other compounds [14–20].

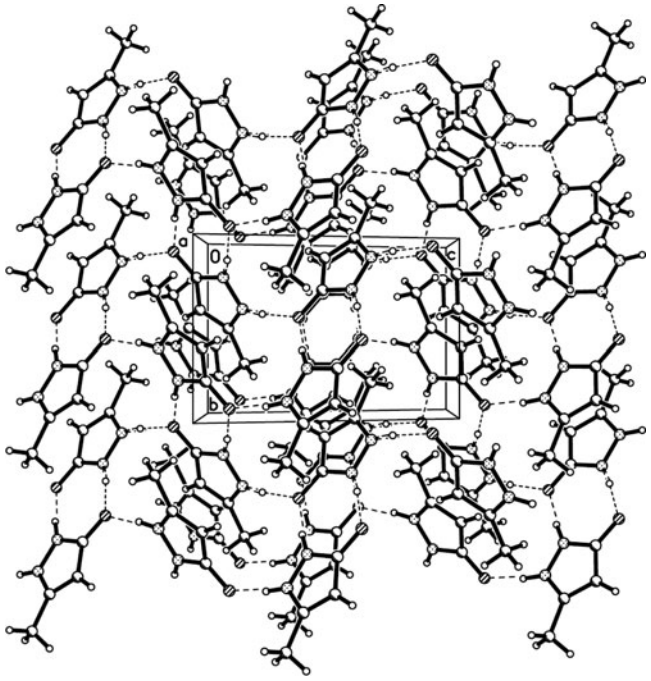

Figure 2. The crystal packing view of the title compound.

As shown in Fig. 2, the crystal packing diagram of the title compound shows that intermolecular N–H···O hydrogen bonds exist between NH of pyrazole and O atom of carbonyl. Furthermore, face-to-face π–π stacking interactions are also observed between pyrazole rings. These interactions stabilize the solid state of the title compound.

Conclusions

In summary, the title compound 1,2-dihydro-3-methylpyrazole-5-one has been structurally characterized by X-ray crystallography.

Funding

We gratefully acknowledge financial support from the Doctoral Research Fund of Henan University of Traditional Chinese Medicine.

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