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Crystal structure of 4-(8-chloro-[1,2,4]triazolo[4,3-α]pyridin-3-yl) phenol

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ABSTRACT

The title compound 4-(8-chloro-[1,2,4]triazolo[4,3-α]pyridin-3-yl)phenol was structurally characterized by X-ray crystal structural analysis. The crystals are triclinic, space group P-1 with a = 7.307(3), b = 11.792(4), c = 12.474(5) Å, $\alpha = 83.850(10)$, $\beta = 87.148(13)$, $\gamma = 77.691(11)°$, V = 1043.6(6) Å^3, Z = 2, F(000) = 504, $D_c = 1.564$ g/cm^3, $\mu = 0.350$ mm^{-1}, the final R = 0.0433 and wR = 0.1232. A total of 10,746 reflections were collected, of which 4920 were independent ($R_{int} = 0.0357$). In the crystal packing diagram, intermolecular O-H …Cl and C-H …O hydrogen bonds stabilize the solid state of the title compound.

KEYWORDS

Crystal structure; heterocyclic compound; triazole

Introduction

In recent years, nitrogen-linked heterocyclic compounds have attracted much attention due to their wide range of applications [1–6]. 1,2,4-Triazoles are widely used in industry, medicine, and agriculture because they always have some outstanding activity, such as antitumor [7] and antitubercular [8]. Synthesis and characterization of 1,2,4-triazoles has become an interesting research field. In view of the reasons mentioned above and as a part of our work on the synthesis and characterization of heterocyclic compounds [9,10], the title compound 4-(8-chloro-[1,2,4]triazolo[4,3-α]pyridin-3-yl)phenol was synthesized and characterized by X-ray diffraction technique.

Experimental

Crystal structure determination

The crystal of the title compound with dimensions of 0.26 mm × 0.10 mm × 0.06 mm was mounted on a Rigaku Saturn CCD area detector diffractometer with a graphite-monochromated MoKα radiation (λ = 0.71073Å) by using a phi and scan modes at 113(2) K in the range of $1.64° \le \theta \le 27.89°$. The crystal belongs to triclinic system with space group *P*-1 and crystal parameters of $a = 7.307(3)$ Å, $b = 11.792(4)$ Å, $c = 12.474(5)$ Å, $\alpha = 83.850(10)°$, $\beta = 87.148(13)°$, $\gamma = 77.691(11)°$, $V = 1043.6(6)$ A^3, $D_c = 1.564$ g/cm^3, The absorption coefficient $\mu = 0.350$ mm^{-1}, and $Z = 2$. A summary of crystal data is presented in Table 1.

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Table 1. Crystal data and structure refinement.

Empirical formula	$C_{24}H_{16}Cl_2N_6O_2$
Formula weight	491.33
Crystal system	Triclinic
Unit cell dimensions	
a (*SÅ*)	7.307(3)
b (*SÅ*)	11.792(4)
c (*SÅ*)	12.474(5)
Unit cell angles (•)	
α	83.850(10)
β	87.148(13)
γ	77.691(11)
Volume ($SÅ^3$)	1043.6(6)
Z	2
Temperature (K)	113(2)
space group	*P*-1
Wavelength (*SÅ*)	0.71073
Calculated density (g/cm^3)	1.564
Absorption coefficient (mm^{-1})	0.350
F(000)	504
Crystal size (mm)	0.26 × 0.10 × 0.06
Theta range for data collection (º)	1.64–27.89
Reflections collected	10746
Independent reflections	4920 [$R_{(int)} = 0.0357$]
Final *R* indices [I>2σ(I)]	$R_1 = 0.0433$, $wR_2 = 0.1232$

The structure was solved by direct methods with SHELXS-97 [11] and refined by the full-matrix least squares method on F^2 data using SHELXL-97 [12]. The empirical absorption corrections were applied to all intensity data. H atom of N-H was initially located in a difference Fourier map and were refined with the restraint Uiso(H) = 1.2Ueq(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) = 1.2Ueq(C) or 1.5Ueq(Cmethyl). The final full-matrix least squares refinement gave $R = 0.0433$ and $wR = 0.1232$.

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.

Table 2. Selected bond lengths (*SÅ*) and bond angles (°).

Bond lengths			
Cl(1)-C(2)	1.7262(15)	Cl(2)-C(14)	1.7159(14)
O(1)-C(10)	1.3575(16)	O(2)-C(22)	1.3531(16)
N(1)-C(5)	1.3830(16)	N(1)-C(6)	1.3886(17)
N(1)-C(1)	1.3886(16)	N(2)-C(6)	1.3121(17)
N(2)-N(3)	1.3779(17)	N(3)-C(1)	1.3188(17)
N(5)-N(6)	1.3791(16)	N(6)-C(18)	1.3172(17)
C(1)-C(2)	1.422(2)	C(7)-C(12)	1.4048(18)
Bond angles			
C(5)-N(1)-C(6)	133.22(12)	C(5)-N(1)-C(1)	122.62(12)
C(6)-N(2)-N(3)	109.38(11)	C(1)-N(3)-N(2)	106.59(11)
C(13)-N(5)-N(6)	106.70(10)	C(18)-N(6)-N(5)	109.22(10)
N(3)-C(1)-N(1)	110.68(12)	N(3)-C(1)-C(2)	131.38(12)
N(1)-C(1)-C(2)	117.86(12)	C(4)-C(5)-N(1)	118.37(12)
N(2)-C(6)-N(1)	109.25(12)	N(2)-C(6)-C(7)	123.76(12)
N(1)-C(6)-C(7)	126.97(12)	C(12)-C(7)-C(8)	118.73(12)

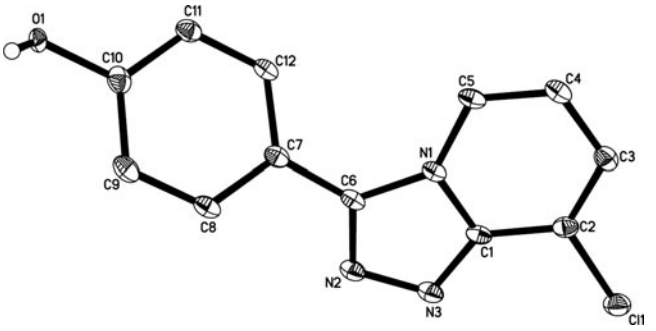

Figure 1. Molecular structure of the title compound.

The structure was solved by direct methods. Anisotropic displacement parameters were applied to all nonhydrogen 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 shown in Figs. 1 and 2, respectively. The title compound crystallizes in triclinic space group *P*-1 with two molecules in the unit cell and two molecules in the asymmetric unit. As shown in Fig. 1, the molecular structure is comprised of two phenyl groups and a 1,2,4-triazole with a chloro and a hydroxy groups. The five-membered ring C1N1C6N2N3 and the six-membered ring C1C2C3C4C5N1 are almost coplanar with the mean deviations of 0.0032 and 0.0049 Å,

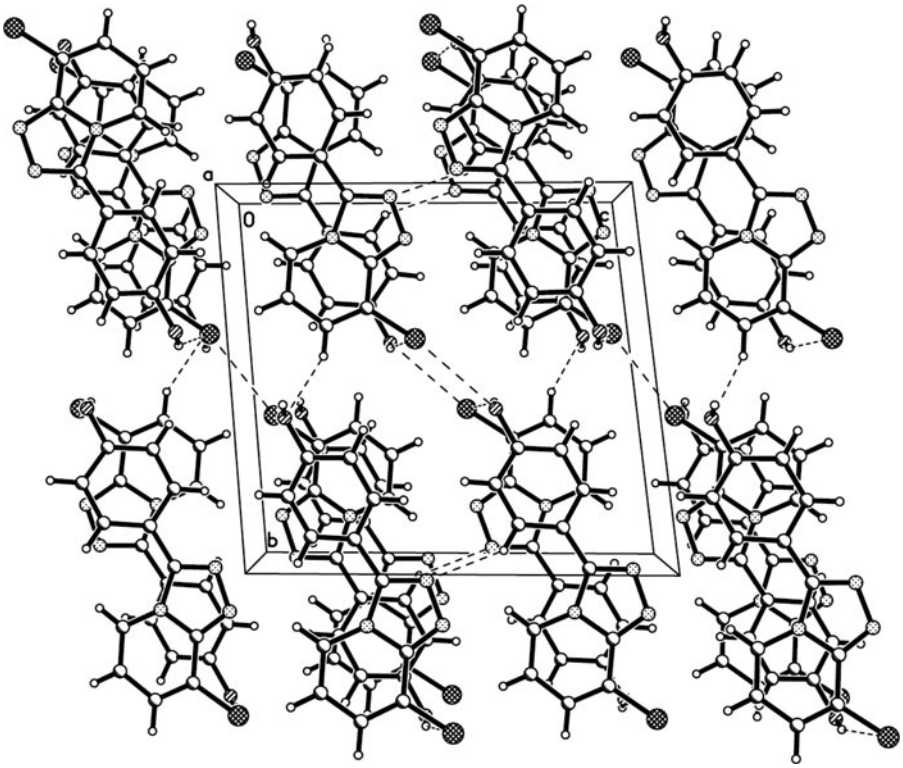

Figure 2. The crystal packing view of the title compound.

respectively. The dihedral angle between C1N1C6N2N3 and C1C2C3C4C5N1 is 2.5°, suggesting that the two plans are nearly coplanar. The dihedral angle between C1N1C6N2N3 and C7C8C9C10C11C12 is 38°. The bond distances [Cl(1)-C(2) = 1.7262(15) Å, Cl(2)-C(14) = 1.7159(14) Å, O(1)-C(10) = 1.3575(16) Å, O(2)-C(22) = 1.3531(16) Å, N(1)-C(5) = 1.3830(16) Å, N(2)-N(3) = 1.3779(17) Å, and N(5)-N(6) = 1.3791(16) Å] and bond angles [C(5)-N(1)-C(6) = 133.22(12)°, C(5)-N(1)-C(1) = 122.62(12)°, C(6)-N(2)-N(3) = 109.38(11)°, C(1)-N(3)-N(2) = 106.59(11)°, C(13)-N(5)-N(6) = 106.70(10)°, C(18)-N(6)-N(5) = 109.22(10)°, N(3)-C(1)-N(1) = 110.68(12)°, N(2)-C(6)-N(1) = 109.25(12)°, and C(1)-C(2)-Cl(1) = 117.76(10)°] are consistent with analogous compounds [13–16].

As shown in Fig. 2, intermolecular O-H …Cl and C-H …O hydrogen bonds stabilize the solid-state of the title compound in the crystal packing diagram.

Conclusions

In summary, the title compound 4-(8-chloro-[1,2,4]triazolo[4,3-α]pyridin-3-yl)phenol has been structurally characterized by X-ray crystallography.

Acknowledgment

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