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X-ray Structural Analysis of 5-(4-pyridyl)-1,3,4-oxadiazole-2(3H)-thione

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The title compound 5-(4-pyridyl)-1,3,4-oxadiazole-2(3H)-thione was characterized by single-crystal X-ray diffraction analysis. The crystals are monoclinic, space group P2*(1)/n with* a = *4.7946(15),* b = *13.775(4),* c = *11.722(4) Å,* $\alpha = 90$, $\beta = 96.557(5)$, $\gamma = 90^\circ$, V = *769.1(4)* $Å^3$, Z = *4,* F(000) = *368,* $D_c = 1.548$ *g/cm*^{3}, $\mu = 0.368$ *mm*$^{-1}$, *the final* R = *0.0301, and* wR = *0.0838. A total of 6203 reflections were collected, of which 1358 were independent (*$R_{int} = 0.0348$*). In the crystal packing diagram, intermolecular C–H···S and N–H···N hydrogen bonds stabilize the solid state of the title compound.*

Keywords Crystal structure; oxadiazole; pyridyl

Introduction

In recent years, heterocyclic compounds containing nitrogen and oxygen have received great attention due to their widely applications [1–5]. 1,3,4-oxadiazole compounds are utilized in industry, medicinal, and agricultural applications [6–8]. In addition, pyridyl-based ligands are used often in the coordination chemistry [9–12]. As a continuation of our previous research work [13–15], and an oxadiazole compound containing pyridyl group have been successfully synthesized and its structure was further characterized by single-crystal X-ray diffraction analysis. In this paper, we report the crystal structure of 5-(4-pyridyl)-1,3,4-oxadiazole-2(3H)-thione.

Experimental

Crystal Structure Determination

The crystal of the title compound with dimensions of 0.16 mm × 0.06 mm × 0.06 mm was mounted on a Rigaku Saturn CCD area detector diffractometer with a graphite-monochromated MoKα radiation ($\lambda = 0.71073$Å) by using a phi and scan modes at 113(2) K in the range of $2.29^\circ \leq \theta \leq 25.02^\circ$. The crystal belongs to Monoclinic system with space group *P*2(1)/n and crystal parameters of $a = 4.7946(15)$ Å, $b = 13.775(4)$ Å, $c = 11.722(4)$ Å, $\alpha = 90^\circ$, $\beta = 96.557(5)^\circ$, $\gamma = 90^\circ$, $V = 769.1(4)$ A^3, $D_c = 1.548$ g/cm^3, the absorption coefficient $\mu = 0.368$ mm^{-1}, and $Z = 4$. A summary of crystal data is presented in Table 1.

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Table 1. Crystal data and structure refinement

Empirical formula	$C_7H_5N_3OS$
Formula weight	179.20
Crystal system	Monoclinic
Unit cell dimensions	
a (Å)	4.7946(15)
b (Å)	13.775(4)
c (Å)	11.722(4)
Unit cell angles (°)	
α	90
β	96.557(5)
γ	90
Volume (Å^3)	769.1(4)
Z	4
Temperature (K)	113(2)
space group	$P2(1)/n$
Wavelength (Å)	0.71073
Calculated density (g/cm^3)	1.548
Absorption coefficient (mm^{-1})	0.368
$F(000)$	368
Crystal size (mm)	0.16 × 0.06 × 0.06
Theta range for data collection (°)	2.29 –25.02
Reflections collected	6203
Independent reflections	1358 [$R_{(int)} = 0.0348$]
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0301$, w$R_2 = 0.0838$

The structure was solved by direct methods with SHELXS-97 [16] and refined by the full-matrix least squares method on F^2 data using SHELXL-97 [17]. 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 $R = 0.0301$ and $wR = 0.0838$.

Results and Discussion

The title compound 5-(4-pyridyl)-1,3,4-oxadiazole-2(3H)-thione was confirmed by single-crystal X-ray diffraction analysis. The selected bond lengths and bond angles listed in Table 2. 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 monoclinic space group $P2(1)/n$ with four molecules in the unit cell and one molecule in the asymmetric unit. As shown in Fig. 1, the molecular structure contains a pyridyl and an oxadiazole units linking

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

Bond lengths			
S(1)-C(1)	1.6417(17)	N(1)-C(1)	1.335(2)
N(1)-N(2)	1.3761(18)	N(2)-C(2)	1.286(2)
N(3)-C(5)	1.339(2)	N(3)-C(6)	1.342(2)
O(1)-C(2)	1.3766(19)	O(1)-C(1)	1.387(2)
C(2)-C(3)	1.448(2)	C(3)-C(4)	1.392(2)
C(3)-C(7)	1.395(2)	C(4)-C(5)	1.381(2)
Bond angles			
C(1)-N(1)-N(2)	112.59(13)	C(2)-N(2)-N(1)	104.14(12)
C(5)-N(3)-C(6)	118.30(14)	C(2)-O(1)-C(1)	105.76(12)
N(1)-C(1)-O(1)	104.73(13)	N(1)-C(1)-S(1)	131.04(13)
O(1)-C(1)-S(1)	124.22(12)	N(2)-C(2)-O(1)	112.78(14)
N(2)-C(2)-C(3)	127.98(14)	O(1)-C(2)-C(3)	119.23(13)
C(4)-C(3)-C(7)	118.66(14)	C(4)-C(3)-C(2)	120.76(13)
C(7)-C(3)-C(2)	120.58(13)	C(5)-C(4)-C(3)	118.95(14)
N(3)-C(5)-C(4)	122.62(14)	N(3)-C(6)-C(7)	123.02(15)

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together through C2 and C3 atoms. The H atom of the thiol group is transferred to the N atom of the 1,3,4-oxadiazole to form thioamide group. The five-membered ring C1O1C2N2N1 and the six-membered ring C3C4C5N3C6C7 are almost coplanar with the mean deviations of 0.0005 and 0.0032 Å, respectively. The dihedral angle between the pyridyl plane and oxadiazole plane is 9.6°, indicating that the two planes are nearly coplanar. The bond distances [S1-C1 = 1.6417(17) Å, N1-C1 = 1.335(2) Å, N1-N2 = 1.3761(18) Å, N2-C2 = 1.286(2) Å, N3-C5 = 1.339(2) Å, O1-C1 = 1.387(2) Å and C2-C3 = 1.448(2) Å] and bond angles [C1-N1-N2 = 112.59(13)°, C2-N2-N1 = 104.14(12)°, C5-N3-C6 = 118.30(14)°, C2-O1-C1 = 105.76(12)°, N1-C1-O1 = 104.73(13)°, N1-C1-S1 = 131.04(13)°, N2-C2-O1 = 112.78(14)° and N2-C2-C3 = 127.98(14)°] are comparable to the analogous compounds [18–25].

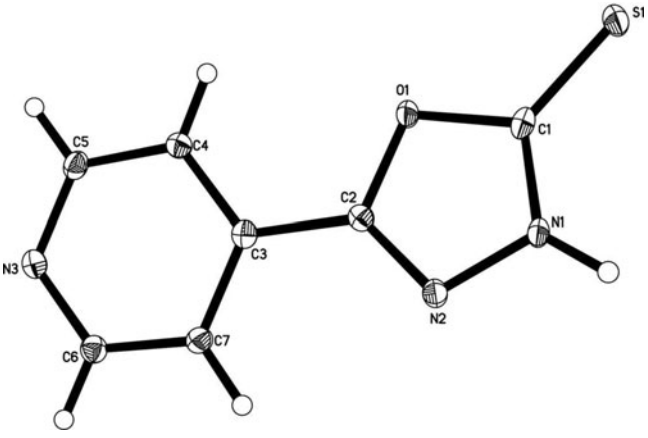

Figure 1. Molecular structure of the title compound.

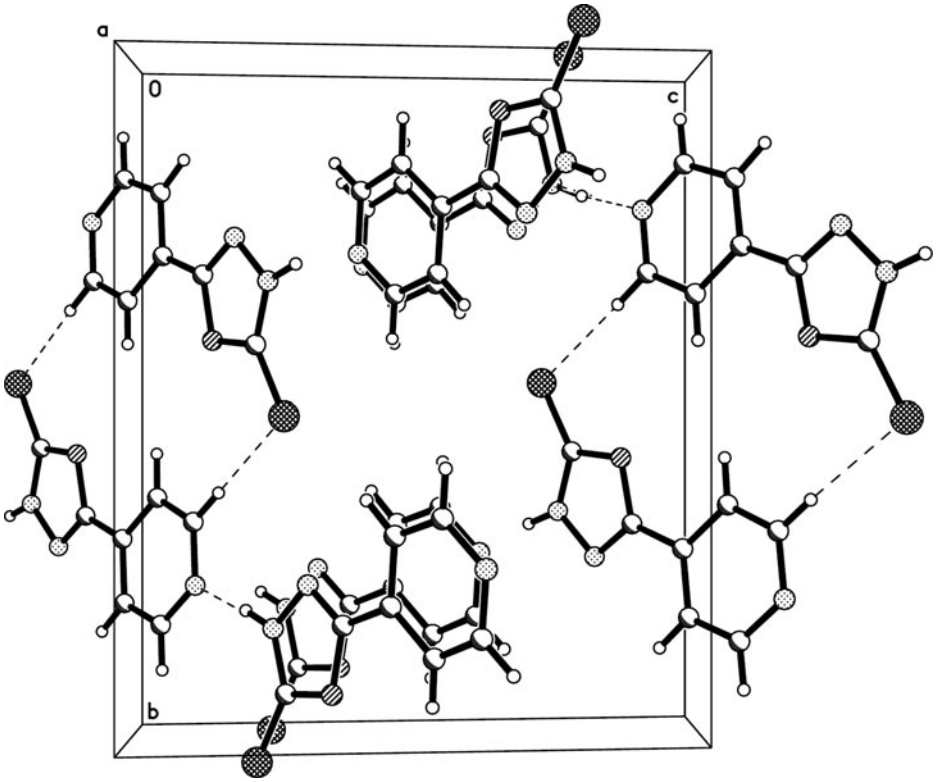

Figure 2. The crystal packing view of the title compound.

As shown in Fig. 2, the crystal packing diagram of the title compound along the *a*-axis reveals that there are intermolecular C-H···S and N-H···N hydrogen bonds existing between CH of the phenyl group and sulfur atom and NH of the oxadiazole ring and N atom of pyridine. In addition, face-to-face π–π stacking interactions are also observed between the pyridyl and oxadiazole rings in the crystal packing diagram. These interactions stabilize the solid-state of the title compound to form a one- dimentional chain.

Conclusions

In summary, the title compound 5-(4-pyridyl)-1,3,4-oxadiazole-2(3H)-thione has been structurally characterized by X-ray crystallography.

Acknowledgments

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Supplemental Materials

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication number CCDC 1021198

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for the title compound. Copies of the data can be obtained free of charge at http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; e-mail: deposit@ccdc.cam.ac.uk.

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