Hao Wu and Hai-Tao Zong*

Crystal structure of 3-methyl-2,3-dihydro-2-thioxoquinazolin-4(1*H*)-one, C₉H₈N₂OS

<https://doi.org/10.1515/ncrs-2018-0013>

Received January 25, 2018; accepted May 3, 2018; available online May 18, 2018

Abstract

C₉H₈N₂OS, monoclinic, *C*2/*c* (no. 15), *a* = 24.273(5) Å, *b* = 9.026(2) Å, *c* = 8.2852(19) Å, β = 108.818(4)°, *V* = 1718.2(7) Å³, *Z* = 8, *R*_{gt}(*F*) = 0.0438, *wR*_{ref}(*F*²) = 0.1190, *T* = 296(2) K.

CCDC no.: 1841237

The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The title compound was synthesized according to procedures described in the literature [3]. The crystal for single-crystal X-ray diffraction was obtained by recrystallization from acetonitrile/water (1:1) solution.

Experimental details

The structure was solved by Direct Methods and refined with the SHELX crystallographic software package [6].

*Corresponding author: Hai-Tao Zong, School of Physics and Electronic Information Engineering, Henan Polytechnic University, Jiaozuo, 454000, P.R. China, e-mail: haitaozong@163.com

Hao Wu: College of Chemistry and Chemical Engineering, Henan Polytechnic University, Jiaozuo, 454000, P.R. China

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.12 × 0.10 × 0.08 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.33 mm ^{−1}
Diffractometer, scan mode:	Bruker SMART, φ and ω -scans
$\theta_{\max}$, completeness:	25°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	4292, 1521, 0.054
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ (<i>I</i> _{obs}), 972
<i>N</i> (<i>param</i>) _{refined} :	118
Programs:	Bruker programs [1], SHELX [2]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
S1	0.00345(3)	0.23770(8)	0.50042(10)	0.0506(3)
N1	0.07239(10)	0.0494(3)	0.7138(3)	0.0402(6)
H1A	0.049342	−0.016256	0.652344	0.048*
N2	0.09650(10)	0.2956(2)	0.7733(3)	0.0401(6)
O1	0.17137(9)	0.3590(3)	1.0094(3)	0.0642(7)
C1	0.11906(11)	−0.0005(3)	0.8480(3)	0.0346(7)
C2	0.12996(12)	−0.1515(3)	0.8753(4)	0.0449(8)
H2B	0.105378	−0.220816	0.804626	0.054*
C3	0.17737(13)	−0.1965(4)	1.0075(4)	0.0508(9)
H3A	0.184859	−0.297194	1.026413	0.061*
C4	0.21444(13)	−0.0939(4)	1.1139(4)	0.0524(9)
H4A	0.246395	−0.126055	1.203486	0.063*
C5	0.20375(13)	0.0552(4)	1.0865(4)	0.0487(8)
H5A	0.228695	0.123861	1.157116	0.058*
C6	0.15574(11)	0.1036(3)	0.9533(3)	0.0373(7)
C7	0.14351(13)	0.2598(3)	0.9208(4)	0.0426(8)
C8	0.06008(12)	0.1923(3)	0.6716(4)	0.0375(7)
C9	0.08540(15)	0.4535(3)	0.7362(4)	0.0583(10)
H9A	0.114528	0.510979	0.818375	0.087*
H9B	0.086757	0.474469	0.623938	0.087*
H9C	0.047656	0.478669	0.741983	0.087*

The hydrogen atoms were placed at calculated positions and refined as riding atoms with isotropic displacement parameters.

Discussion

Thioquinazoline derivatives have been paid much attention, not only due to their pharmacological activities, but also to

their applications in the synthesis of a variety of heterocyclic compounds [4, 5]. In the course of a search for bioactive compounds, the title compound was synthesized and its crystal structure is presented here.

In the title compound, the bond lengths of C=S and C=O double bonds are 1.676(3) and 1.216(3) Å, comparable with the parameters reported in the literature [6, 7]. In the crystal, pairs of intermolecular N—H···S hydrogen bonds link two molecules into a centrosymmetric dimer, forming a R₂²(8) ring motif according to the Etter nomenclature. All geometric parameters are in the expected ranges.

References

1. Bruker. SMART and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA (2007).
2. Sheldrick, G. M.: A short history of SHELX. *Acta Crystallogr.* **A64** (2008) 112–122.
3. Peng, H.-D.; Yang, J.-H.; Yang, G.-C.; Chen, Z.-X.: Synthesis of 2-thioquinazolin-4-ones. *J. Hubei Univ. (Nat. Sci.)* **28** (2006) 282–284, 292.
4. Kadi, A. A.: Synthesis and antimicrobial activity of some new quinazolin-4(3*H*)-one derivatives. *J. Saudi Chem. Soc.* **15** (2011) 95–100.
5. Abdel-Megeed, M. F.; Azaam, M. M.; El-Hiti, G. A.: A simple procedure for synthesis of 3*H*-quinazolin-4-one hydrazones under mild conditions. *J. Saudi Chem. Soc.* **18** (2014) 1022–1027.
6. Cai, H.-X.; Yan, L.-L.: Crystal structure of 3-benzyl-2,3-dihydro-2-thioxoquinazolin-4(1*H*)-one, C₁₅H₁₂N₂OS. *Z. Kristallogr. NCS* **232** (2017) 811–812.
7. Yang, P.; Yan, L.-L.; Cai, H.-X.: Crystal structure of 3-(3-dimethylaminopropyl)-2,3-dihydro-2-thioxoquinazolin-4(1*H*)-one, C₁₃H₁₇N₃OS. *Z. Kristallogr. NCS* **233** (2018) 117–118.