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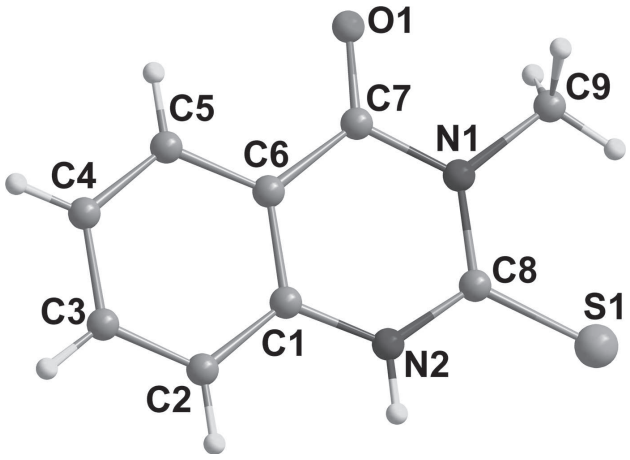
Crystal structure of 3-methyl-2,3-dihydro-2-thioxoquinazolin-4(1*H*)-one, $C_9H_8N_2OS$



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Abstract

$C_9H_8N_2OS$, monoclinic, $C2/c$ (no. 15), $a = 24.273(5)$ Å, $b = 9.026(2)$ Å, $c = 8.2852(19)$ Å, $\beta = 108.818(4)°$, $V = 1718.2(7)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0438$, $wR_{ref}(F^2) = 0.1190$, $T = 296(2)$ K.

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

Table 1: Data collection and handling.

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

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

Atom	x	y	z	U_{iso}*/U_{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*

Discussion

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

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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 lengthes 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, paris of intermolecular N—H···S hydrogen bonds link two molecules into a centrosymmetric dimer, forming a $R_2^2(8)$ ring motif according to the Etter nomenclature. All geometric parameters are in the expected ranges.

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