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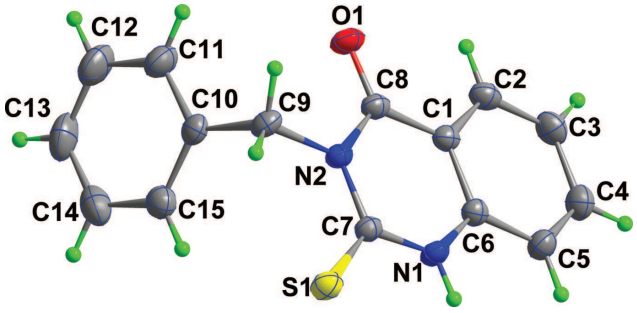
Crystal structure of 3-benzyl-2,3-dihydro-2-thioxoquinazolin-4(1*H*)-one, $C_{15}H_{12}N_2OS$



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Abstract

$C_{15}H_{12}N_2OS$, triclinic, $P\bar{1}$ (no. 2), $a = 6.4172(6)$ Å, $b = 9.7237(10)$ Å, $c = 10.5031(10)$ Å, $\alpha = 84.838(2)°$, $\beta = 87.561(2)°$, $\gamma = 78.557(2)°$, $V = 639.54(11)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0362$, $wR_{ref}(F^2) = 0.0934$, $T = 296$ K.

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

Source of materials

The title compound was synthesized according to the literature method [5]. The crystal suitable for single crystal X-ray diffraction was obtained by recrystallization from acetonitrile.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.25 × 0.23 × 0.20 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	2.5 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$2\theta_{max}$, completeness:	50°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	3328, 2255, 0.013
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1914
$N(param)_{refined}$:	173
Programs:	SHELX [1], Bruker programs [2]

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

Atom	x	y	z	U_{iso}*/U_{eq}
S1	0.20040(7)	1.04133(5)	0.33319(5)	0.04681(19)
N2	0.5610(2)	0.85199(14)	0.38262(13)	0.0355(3)
N1	0.2632(2)	0.82817(15)	0.50750(13)	0.0391(4)
H1A	0.1311	0.8575	0.5264	0.047*
O1	0.87772(19)	0.70547(14)	0.42301(13)	0.0521(4)
C7	0.3491(3)	0.90068(17)	0.41106(16)	0.0358(4)
C8	0.6891(3)	0.73697(18)	0.45076(17)	0.0384(4)
C6	0.3682(3)	0.71075(18)	0.57899(16)	0.0365(4)
C1	0.5829(3)	0.66224(18)	0.55210(16)	0.0360(4)
C2	0.6912(3)	0.54254(19)	0.62182(18)	0.0449(5)
H2A	0.8346	0.5088	0.6043	0.054*
C9	0.6700(3)	0.92533(19)	0.27768(17)	0.0420(4)
H9A	0.8056	0.9380	0.3073	0.050*
H9B	0.5845	1.0179	0.2561	0.050*
C5	0.2614(3)	0.6417(2)	0.67484(17)	0.0456(5)
H5A	0.1178	0.6746	0.6925	0.055*
C3	0.5854(3)	0.4747(2)	0.71638(19)	0.0513(5)
H3A	0.6576	0.3948	0.7630	0.062*
C4	0.3704(3)	0.5248(2)	0.74284(19)	0.0507(5)
H4A	0.3004	0.4783	0.8074	0.061*
C10	0.7074(3)	0.84590(19)	0.15964(17)	0.0427(4)
C15	0.5412(3)	0.8324(2)	0.08560(19)	0.0535(5)
H15A	0.4025	0.8738	0.1077	0.064*
C11	0.9117(3)	0.7851(3)	0.1226(2)	0.0632(6)
H11A	1.0256	0.7946	0.1708	0.076*
C14	0.5811(4)	0.7571(3)	−0.0220(2)	0.0692(7)
H14A	0.4687	0.7474	−0.0713	0.083*
C13	0.7853(5)	0.6970(3)	−0.0559(2)	0.0785(8)
H13A	0.8111	0.6468	−0.1281	0.094*
C12	0.9500(4)	0.7107(3)	0.0158(3)	0.0830(8)
H12A	1.0884	0.6698	−0.0072	0.100*

Experimental details

All the H atoms were located in the difference electron density map. The H atoms were situated into the idealized positions with the carrier atom-H distances = 0.93 Å for aryl, 0.97 Å for methylene and 0.86 Å for the secondary amine hydrogen atom. The U_{iso} values were constrained to be $1.2U_{eq}$ of the carrier atoms.

Comment

Quinazoline derivatives exhibit a wide variety of pharmacological activities [3–5], and they are important intermediates in the synthesis of a variety of valuable heterocyclic compounds [4]. The crystal structure of one thioquinazoline, namely 3-benzyl-2,3-dihydro-2-thioxoquinazolin-4(1*H*)-one, is reported in this work.

In the title crystal structure, the bond lengths of C=S and C=O are 1.6724(17) and 1.218(2) Å, confirming that both are double bonds (*cf*. the figure). In the crystal, pairs of intermolecular N—H···S hydrogen bonds link two molecules into a centrosymmetric dimer, forming a $R_2^2(8)$ ring motif. In addition, the dimers are further connected with each other into one dimensional chains *via* weak intermolecular C–H···O hydrogen bonds. Bond lengths and angles are in accord with directly related structures [6, 7].

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