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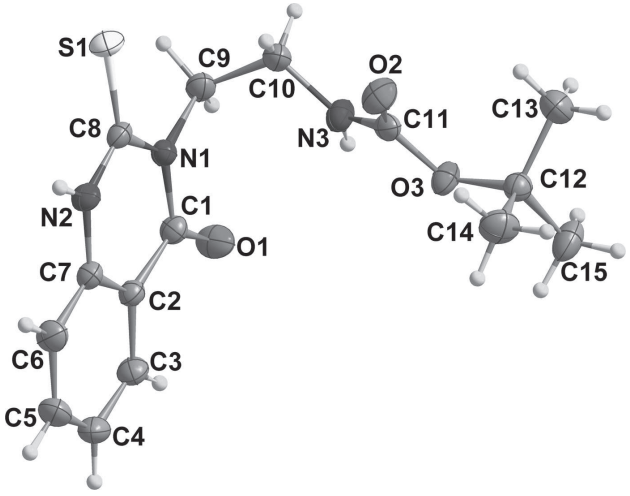
Crystal structure of *tert*-butyl (2-(4-oxo-2-thioxo-1,4-dihydroquinazolin-3(2*H*)-yl)ethyl)carbamate, $C_{15}H_{19}N_3O_3S$



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Abstract

$C_{15}H_{19}N_3O_3S$, triclinic, $P\bar{1}$ (no. 2), $a = 8.682(8)$ Å, $b = 9.700(8)$ Å, $c = 11.273(10)$ Å, $\alpha = 90.681(14)°$, $\beta = 112.624(13)°$, $\gamma = 112.632(13)°$, $V = 794.5(12)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0405$, $wR_{ref}(F^2) = 0.1171$, $T = 296(2)$ K.

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The 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 the literature [3]. The crystal suitable for single X-ray diffraction was obtained by recrystallization from acetonitrile solution.

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Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.20 × 0.15 × 0.10 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.22 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω-scans
θ_{max}, completeness:	25°, >98%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	4006, 2771, 0.013
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2172
$N(param)_{refined}$:	199
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	0.74468(9)	0.52939(7)	−0.04862(6)	0.0566(2)
O1	0.8053(2)	0.27450(19)	0.33814(14)	0.0640(5)
O2	0.7173(2)	0.76635(19)	0.30437(14)	0.0581(4)
O3	0.7563(2)	0.68216(17)	0.49665(13)	0.0485(4)
N1	0.7715(2)	0.38677(17)	0.15841(14)	0.0363(4)
N2	0.4993(2)	0.27750(18)	−0.03153(15)	0.0400(4)
H2A	0.432412	0.281001	−0.109878	0.048*
N3	0.8956(3)	0.6383(2)	0.38351(16)	0.0501(5)
H3B	0.927098	0.586817	0.442525	0.060*
C1	0.7099(3)	0.2702(2)	0.22396(18)	0.0400(5)
C2	0.5283(3)	0.1463(2)	0.14783(18)	0.0371(4)
C3	0.4587(3)	0.0192(2)	0.2001(2)	0.0484(5)
H3A	0.529062	0.011640	0.284034	0.058*
C4	0.2866(3)	−0.0948(3)	0.1281(2)	0.0559(6)
H4A	0.241070	−0.179773	0.162777	0.067*
C5	0.1812(3)	−0.0827(3)	0.0038(2)	0.0585(6)
H5A	0.063779	−0.159111	−0.043716	0.070*
C6	0.2468(3)	0.0401(3)	−0.0508(2)	0.0521(6)
H6A	0.174575	0.047020	−0.134443	0.062*
C7	0.4240(3)	0.1549(2)	0.02104(18)	0.0367(4)
C8	0.6687(3)	0.3921(2)	0.02965(18)	0.0373(5)
C9	0.9608(3)	0.5072(2)	0.2302(2)	0.0459(5)
H9A	1.021507	0.528678	0.171776	0.055*
H9B	1.029337	0.469985	0.301983	0.055*
C10	0.9653(3)	0.6533(2)	0.2841(2)	0.0496(5)
H10A	1.091248	0.730320	0.320942	0.060*
H10B	0.892978	0.688199	0.212480	0.060*
C11	0.7839(3)	0.7014(2)	0.38741(18)	0.0397(5)
C12	0.6819(3)	0.7721(2)	0.5460(2)	0.0445(5)

Table 2 (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C13	0.7968(4)	0.9399(3)	0.5616(3)	0.0690(7)
H13A	0.747816	0.997635	0.593682	0.104*
H13B	0.921477	0.965795	0.622616	0.104*
H13C	0.794129	0.962975	0.478455	0.104*
C14	0.4803(3)	0.7252(3)	0.4574(3)	0.0677(7)
H14A	0.434515	0.784697	0.490984	0.102*
H14B	0.467184	0.741830	0.370935	0.102*
H14C	0.411600	0.619406	0.454179	0.102*
C15	0.7044(4)	0.7260(3)	0.6779(2)	0.0725(8)
H15A	0.659878	0.778083	0.720414	0.109*
H15B	0.635320	0.618206	0.665267	0.109*
H15C	0.832139	0.752618	0.731220	0.109*

Experimental details

The structure was solved by Direct Methods and refined with the SHELX crystallographic software package [2]. The hydrogen atoms were placed at calculated positions and refined as riding atoms with isotropic displacement parameters.

Discussion

Quinazoline derivatives have attracted much attention due to their wide variety of pharmacological activities [3–6]. This contribution forms part of a study on the synthesis and the structures of 2,3-dihydro-2-thioxoquinazolin-4(1*H*)-ones [6–8]. In this paper, the structure of *tert*-butyl (2-(4-oxo-2-thioxo-1,4-dihydroquinazolin-3(2*H*)-yl)ethyl)carbamate is investigated by X-ray diffraction.

In the title compound, the bond lengths are comparable with those found in our previous reports [6–8]. The conformation about the C9–C10 bond leads to the hook shape of the title molecule (*cf*. the figure). In the crystal, pairs of intermolecular N—H···N hydrogen bonds link the title molecules into one dimentional chains.

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