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Crystal structure of methyl 2,7,7-trimethyl-4-(3-methylthiophen-2-yl)-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate, $C_{19}H_{23}NO_3S$

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.26 × 0.21 × 0.18 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.20 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
$\theta_{\max}$, completeness:	27.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	10596, 3991, 0.033
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3051
$N(\text{param})_{\text{refined}}$:	218
Programs:	Bruker programs [1], SHELX [2], OLEX2 [3]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.32341(19)	0.30614(8)	1.07671(14)	0.0338(4)
H1	0.388221	0.315130	1.149540	0.041*
S1	0.28177(6)	0.17542(3)	0.64855(5)	0.0460(2)
O1	0.2427(2)	0.45008(8)	0.77028(14)	0.0470(4)
O2	0.1718(2)	0.35403(9)	0.64426(14)	0.0539(4)
O3	−0.02911(17)	0.13850(8)	0.82432(13)	0.0432(4)
C1	0.2210(3)	0.49559(12)	0.6610(2)	0.0551(6)
H1A	0.242872	0.545771	0.686637	0.083*
H1B	0.290088	0.479838	0.615968	0.083*
H1C	0.115383	0.491517	0.607640	0.083*
C2	0.2162(2)	0.37880(10)	0.74868(18)	0.0328(4)
C3	0.2425(2)	0.33292(9)	0.86082(17)	0.0294(4)
C4	0.3149(2)	0.35428(10)	0.97952(17)	0.0313(4)
C5	0.3927(3)	0.42595(11)	1.02341(19)	0.0418(5)
H5A	0.434374	0.425673	1.113392	0.063*
H5B	0.475356	0.433307	0.987648	0.063*
H5C	0.318276	0.464993	0.997443	0.063*
C6	0.2330(2)	0.24497(10)	1.06150(17)	0.0309(4)
C7	0.1565(2)	0.22066(9)	0.94458(16)	0.0296(4)
C8	0.1911(2)	0.25359(9)	0.83230(16)	0.0288(4)
H8	0.095289	0.253068	0.760976	0.035*
C9	0.0476(2)	0.16142(10)	0.92871(17)	0.0321(4)
C10	0.0236(2)	0.12802(12)	1.04415(18)	0.0408(5)
H10A	−0.007065	0.077136	1.026463	0.049*
H10B	−0.061510	0.153368	1.062105	0.049*
C11	0.1658(2)	0.13083(11)	1.16093(17)	0.0351(4)
C12	0.2935(3)	0.08097(13)	1.1446(2)	0.0522(6)
H12A	0.321465	0.096336	1.072399	0.078*
H12B	0.382873	0.083912	1.218063	0.078*

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Abstract

$C_{19}H_{23}NO_3S$, monoclinic, $P2_1/n$ (no. 14), $a = 9.0403(15)$ Å, $b = 18.213(3)$ Å, $c = 11.1889(19)$ Å, $\beta = 107.964(2)^\circ$, $V = 1752.4(5)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0498$, $wR_{\text{ref}}(F^2) = 0.1536$, $T = 296(2)$ 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.

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Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
H12C	0.256546	0.031250	1.133143	0.078*
C13	0.1213(3)	0.10645(15)	1.2756(2)	0.0550(6)
H13A	0.040821	0.137875	1.285894	0.083*
H13B	0.084365	0.056725	1.264153	0.083*
H13C	0.210691	0.109387	1.349073	0.083*
C14	0.2210(2)	0.21038(11)	1.17921(17)	0.0369(4)
H14A	0.149186	0.238682	1.209758	0.044*
H14B	0.322045	0.212140	1.242817	0.044*
C15	0.3126(2)	0.20888(10)	0.79773(16)	0.0315(4)
C16	0.4540(2)	0.18702(12)	0.8728(2)	0.0419(5)
H16	0.493344	0.199826	0.957114	0.050*
C17	0.5355(3)	0.14309(12)	0.8109(2)	0.0467(5)
H17	0.634155	0.124362	0.850417	0.056*
C18	0.4577(3)	0.13066(12)	0.6894(2)	0.0439(5)
C19	0.5052(3)	0.08490(16)	0.5967(3)	0.0699(8)
H19A	0.425340	0.086516	0.516955	0.105*
H19B	0.600445	0.103671	0.587763	0.105*
H19C	0.520431	0.035068	0.625951	0.105*

Source of material

A mixture of 5,5-dimethyl-cyclohexane-1,3-dione (10 mmol), 3-methylthiophene-2-carboxaldehyde (10 mmol), 3-amino-2-butenic acid methyl ester (10 mmol) in ethanol (100 mL) was refluxed for 2–3 h and then cooled to room temperature. After filtering of the precipitates, they were sequentially washed with ice-cooled water and ethanol and then dried under a vacuum.

Experimental details

H atoms bonded to C and N atoms were positioned geometrically and refined using a riding model, with C–H = 0.93/0.96/0.97 Å and N–H = 0.86 Å with *U*_{iso}(H) = 1.2 times *U*_{eq}(C) and 1.2 times *U*_{eq}(N).

Discussion

4-Arylpolyhydroquinoline derivatives possess several types of pharmacological properties such as anticancer, anti-HIV, anticoagulant, spasmolytic, and antibacterial activity [4]. Recent research has also indicated that these heterocycles

are an effective treatment to shrink cancer and control symptoms [5].

In the crystal structure of the title compound (*cf.* the figure), the six-membered ring containing nitrogen atom and the adjacent ring form the core of this compound. The angle between the five membered ring constructed by C15–C18 and S1 and the adjacent ring built by C3, C4, C6–C8 and N1 is 84.8°. Similar structures have also been reported. The bond lengths of this compound are in agreement with those in previously reported structure compound [6, 7]. There are also some structures that are directly related [8, 9].

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