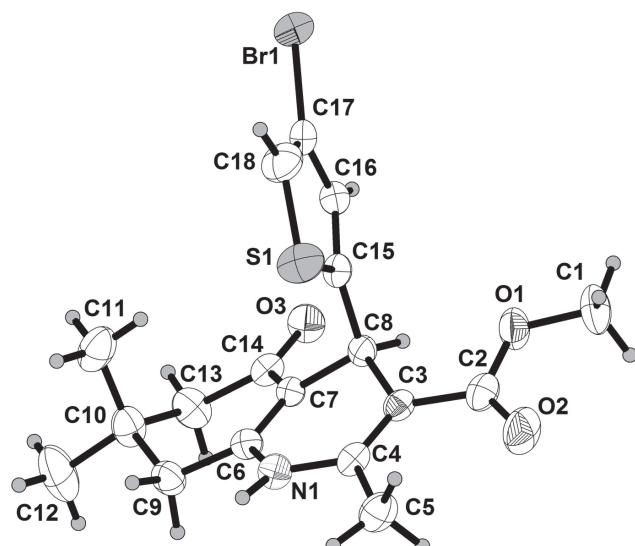


Bo Tang*

Crystal structure of methyl 4-(4-bromothiophen-2-yl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate, C₁₈H₂₀BrNO₃S



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Abstract

C₁₈H₂₀BrNO₃S, monoclinic, P2₁/n (no. 14), $a = 11.558(5)$ Å, $b = 13.098(5)$ Å, $c = 12.250(5)$ Å, $\beta = 106.923(17)^\circ$, $V = 1774.2(12)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0528$, $wR_{\text{ref}}(F^2) = 0.1526$, $T = 293(2)$ 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

A mixture of 5,5-dimethyl-cyclohexane-1,3-dione (10 mmol), 4-bromothiophene-2-carboxaldehyde (10 mmol), 3-amino-2-butenoic acid methyl ester (10 mmol) in ethanol (100 mL)

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.29 × 0.24 × 0.21 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	2.45 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max} , completeness:	27.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	10627, 4042, 0.072
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2226
$N(\text{param})_{\text{refined}}$:	217
Programs:	OLEX2 [1], SHELX [2], Bruker [3]

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.4742(3)	0.3063(2)	-0.0587(2)	0.0349(8)
H1	0.409691	0.278705	-0.102250	0.042*
Br1	1.10012(4)	0.11967(4)	0.05825(5)	0.0679(2)
S1	0.74515(12)	0.23088(9)	-0.12100(10)	0.0535(3)
O1	0.8040(3)	0.5173(2)	-0.0242(2)	0.0461(7)
O2	0.6372(3)	0.5684(2)	-0.1523(3)	0.0594(9)
O3	0.7830(3)	0.2909(2)	0.2806(2)	0.0411(7)
C1	0.8642(5)	0.6008(3)	-0.0579(4)	0.0545(13)
H1A	0.947562	0.601302	-0.012982	0.082*
H1B	0.859418	0.593983	-0.137114	0.082*
H1C	0.826435	0.663505	-0.046350	0.082*
C2	0.6869(4)	0.5080(3)	-0.0805(3)	0.0368(10)
C3	0.6339(4)	0.4195(3)	-0.0440(3)	0.0299(8)
C4	0.5225(4)	0.3894(3)	-0.0979(3)	0.0328(9)
C5	0.4375(4)	0.4380(3)	-0.1999(3)	0.0455(11)
H5A	0.362786	0.400732	-0.221363	0.068*
H5B	0.422301	0.507239	-0.182157	0.068*
H5C	0.472633	0.437514	-0.261900	0.068*
C6	0.5246(3)	0.2664(3)	0.0461(3)	0.0304(9)
C7	0.6349(3)	0.2956(3)	0.1063(3)	0.0261(8)
C8	0.7130(3)	0.3558(3)	0.0513(3)	0.0282(8)
H8	0.763925	0.401745	0.108546	0.034*
C9	0.4485(4)	0.1944(3)	0.0879(3)	0.0394(10)
H9A	0.404918	0.150758	0.025479	0.047*
H9B	0.389440	0.232701	0.113493	0.047*
C10	0.5221(4)	0.1285(3)	0.1850(3)	0.0428(10)
C11	0.5950(5)	0.0513(3)	0.1439(4)	0.0559(13)
H11A	0.541854	0.007224	0.089090	0.084*

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Table 2 (continued)

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
H11B	0.648548	0.085549	0.108981	0.084*
H11C	0.641549	0.011439	0.207344	0.084*
C12	0.4375(5)	0.0732(4)	0.2395(4)	0.0761(17)
H12A	0.390210	0.122189	0.266181	0.114*
H12B	0.384726	0.029277	0.184160	0.114*
H12C	0.484104	0.033068	0.302631	0.114*
C13	0.6056(4)	0.1981(3)	0.2715(3)	0.0444(11)
H13A	0.557250	0.241339	0.305449	0.053*
H13B	0.657239	0.156660	0.331842	0.053*
C14	0.6838(4)	0.2647(3)	0.2224(3)	0.0314(9)
C15	0.7931(3)	0.2852(3)	0.0108(3)	0.0302(9)
C16	0.9033(4)	0.2502(3)	0.0682(3)	0.0350(9)
H16	0.945269	0.270438	0.141858	0.042*
C17	0.9477(4)	0.1799(3)	0.0044(4)	0.0421(10)
C18	0.8739(4)	0.1616(4)	-0.0979(4)	0.0522(12)
H18	0.890187	0.116624	-0.150337	0.063*

was refluxed for 2–3 h and then cooled to room temperature. After filtering the precipitates, they were sequentially washed with ice-cooled water and ethanol and then dried under a vacuum. Crystals were obtained by slow evaporation from ethanol.

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 $U_{\text{iso}}(\text{H})$ = 1.2 times $U_{\text{eq}}(\text{C})$ and 1.2 times $U_{\text{eq}}(\text{N})$.

Comment

4-Arylpolyhydroquinoline derivatives possess several types of pharmacological properties such as anticancer, anti-coagulant, spasmolytic, and antibacterial activity [4–6]. Recent research has also indicated that these heterocycles are effective oral anticoagulants for patients undergoing mechanical valve replacement.

The molecular structure of the title compound is shown in the figure. Within its structure unit, the angle between the mean plane of six-membered ring constructed by C3, C4, C6-C8 and N1 atoms and the five membered ring constructed by C15-C18 and S1 atoms is 86.7°. The bond lengths and angles presented in the title compound are all in their normal scopes. The features of the title compound are similar with those in the structure of methyl-2,7,7-trimethyl-5-oxo-4-(3-phenoxyphenyl)-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate [6] and ethyl 4-[4-(dimethylamino)phenyl]-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylateethyl 4-[4-(dimethylamino)phenyl]-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate [7]; and there are many more related ones. The major differences are the functions located at C14 (*cf.* the figure).

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