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Crystal structure of ethyl 2-methyl-4-(5-methylthiophen-2-yl)-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate, $C_{18}H_{21}NO_3S$

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.26 × 0.21 × 0.17 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.21 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
$\theta_{\max}$, completeness:	25°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	4098, 2860, 0.027
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2594
$N(\text{param})_{\text{refined}}$:	218
Programs:	Bruker programmes [1], SHELX [2], OLEX2 [3]

filtering 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 atoms from organic ligands were positioned geometrically and refined using a riding model, with C–H = 0.93 Å, with $U_{\text{iso}}(\text{H}) = 1.2$ times $U_{\text{eq}}(\text{C})$.

Discussion

4-Arylpolyhydroquinoline derivatives form an important class of compounds. These compounds may possess several types of pharmacological properties such as anticancer, anti-HIV, anticoagulant, spasmolytic, and antibacterial activity [4]. A large number of 4-arylpolyhydroquinoline derivatives have been reported to show substantial cytotoxic activity *in vitro* and *in vivo* [5, 6]. Recognizing the considerable importance of the compounds, the research focused on the synthesis of the 4-arylpolyhydroquinoline derivatives.

The structure of the compound was measured by X-ray diffraction analysis. The molecular structural unit of the title compound is given in Figure. Within the structure of the title compound, the dihedral angle of six-membered ring containing one N atom [C(4), C(7), N(1), C(12), C(13) and C(5)] and the adjacent five-membered ring [C(4)–C(17) and S(1)] is 89.76°. The bond lengths of C–C, C–N, C–O and C–S are in the range of 1.348–1.527 Å, 1.368–1.389 Å, 1.214 Å–1.459 Å and 1.726 Å–1.734 Å, respectively. These bond distances are all in their normal ranges [7, 8]. The title compound is similar

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Abstract

$C_{18}H_{21}NO_3S$, triclinic, $P\bar{1}$ (no. 2), $a = 7.2345(18)$ Å, $b = 10.842(3)$ Å, $c = 11.573(3)$ Å, $\alpha = 69.174(4)^\circ$, $\beta = 77.335(4)^\circ$, $\gamma = 79.555(4)^\circ$. $V = 822.5(4)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0471$, $wR_{\text{ref}}(F^2) = 0.1419$, $T = 296(2)$ K.

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The crystal structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

A mixture of 1,3-cyclohexanedione (7 mmol), ammonium acetate (10 mmol), ethyl acetoacetate (5 mmol), and 5-methyl-2-thiophene (4 mmol) in ethanol (100 mL) was refluxed for 4 h and then cooled to room temperature. After

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
N1	−0.0921(2)	0.27240(17)	0.02578(15)	0.0190(4)
H1	−0.193961	0.253779	0.011751	0.023 [*]
S1	0.07521(7)	0.02829(5)	0.28637(5)	0.0211(2)
O1	0.2045(2)	0.43030(15)	0.25907(14)	0.0255(4)
O2	−0.1100(2)	0.49484(15)	0.26980(14)	0.0258(4)
O3	0.57341(19)	0.23021(15)	−0.03886(14)	0.0237(4)
C1	0.3335(4)	0.4176(3)	0.4375(3)	0.0411(7)
H1A	0.340028	0.463922	0.493154	0.062 [*]
H1B	0.278649	0.336355	0.484693	0.062 [*]
H1C	0.459498	0.397958	0.395516	0.062 [*]
C2	0.2124(3)	0.5027(2)	0.3426(2)	0.0320(6)
H2A	0.084913	0.523494	0.384258	0.038 [*]
H2B	0.266392	0.585311	0.294770	0.038 [*]
C3	0.0326(3)	0.43363(19)	0.23015(18)	0.0195(5)
C4	0.0435(3)	0.35199(19)	0.15056(18)	0.0170(4)
C5	−0.1092(3)	0.34746(19)	0.10382(18)	0.0184(4)
C6	−0.3069(3)	0.4173(2)	0.1250(2)	0.0221(5)
H6A	−0.371304	0.426861	0.057519	0.033 [*]
H6B	−0.298854	0.503505	0.127990	0.033 [*]
H6C	−0.376663	0.366158	0.202863	0.033 [*]
C7	0.0812(3)	0.22713(19)	−0.02948(18)	0.0176(4)
C8	0.0785(3)	0.1806(2)	−0.13623(19)	0.0201(5)
H8A	0.059769	0.256734	−0.210212	0.024 [*]
H8B	−0.027066	0.128356	−0.115312	0.024 [*]
C9	0.2650(3)	0.0967(2)	−0.1642(2)	0.0232(5)
H9A	0.269859	0.079576	−0.241670	0.028 [*]
H9B	0.272066	0.012108	−0.097161	0.028 [*]
C10	0.4332(3)	0.1702(2)	−0.1763(2)	0.0249(5)
H10A	0.550866	0.113332	−0.187878	0.030 [*]
H10B	0.434066	0.248825	−0.250282	0.030 [*]
C11	0.4264(3)	0.21076(19)	−0.06364(18)	0.0179(4)
C12	0.2412(3)	0.23164(19)	0.01011(18)	0.0160(4)
C13	0.2288(3)	0.26383(19)	0.12868(18)	0.0168(4)
H13	0.335685	0.313348	0.117563	0.020 [*]
C14	0.2448(3)	0.1385(2)	0.23996(18)	0.0180(4)
C15	0.3791(3)	0.0925(2)	0.31522(19)	0.0215(5)
H15	0.482205	0.137531	0.305798	0.026 [*]
C16	0.3462(3)	−0.0317(2)	0.41012(19)	0.0246(5)
H16	0.426200	−0.075785	0.468711	0.029 [*]
C17	0.1879(3)	−0.0795(2)	0.40724(19)	0.0228(5)
C18	0.1092(4)	−0.2060(2)	0.4894(2)	0.0307(6)
H18A	−0.015(4)	−0.194(3)	0.537(3)	0.046 [*]
H18B	0.190(4)	−0.252(3)	0.551(3)	0.046 [*]
H18C	0.104(4)	−0.267(3)	0.441(3)	0.046 [*]

with the known compound 4-(thien-2-yl)-2-methyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylic acid ethoxycarbonylphenylmethylester [9]. The $-\text{C}_{10}\text{H}_{11}\text{O}_3$ group in the 4-(thien-2-yl)-2-methyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylic acid ethoxycarbonylphenylmethylester [9] is replaced by $-\text{CH}_2\text{CH}_3$ group in the title compound. In addition, the title compound exhibits a layered supra-molecular structure connected via $\text{N}-\text{H}\cdots\text{N}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds. By means of these interactions the two-dimensional layered supra-molecular structure was generated.

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