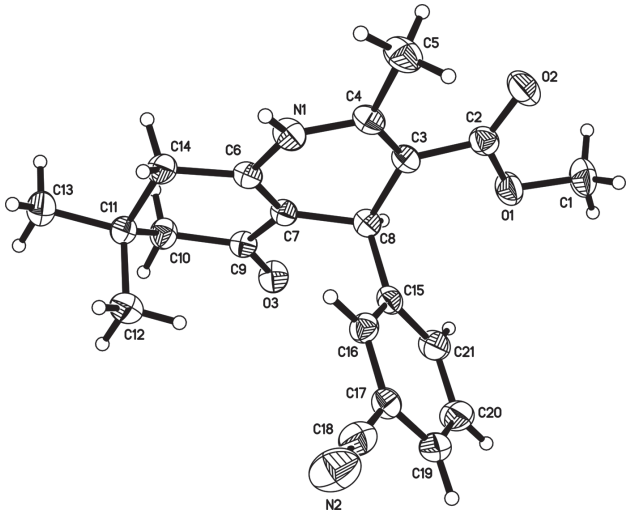
Qing-Hua Meng*, Xu Long, Jing-Li Liu, Shuan Zhang and Guang-Hui Zhang

Crystal structure of methyl 4-(3-cyanophenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate, $C_{21}H_{22}N_2O_3$



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Abstract

$C_{21}H_{22}N_2O_3$, orthorhombic, *Pbcn* (no. 60), $a = 15.093(2)$ Å, $b = 13.896(2)$ Å, $c = 17.501(2)$ Å, $V = 3670.5(8)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0446$, $wR_{ref}(F^2) = 0.1208$, $T = 293(2)$ 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 methyl 3-aminocrotonate (10 mmol), aromatic aldehyde (10 mmol) and 1,1-dimethyl-3,5-cyclohexanedione (10 mmol) in ethanol (100 mL) was refluxed for 2−3 h and

***Corresponding author: Qing-Hua Meng,** School of Pharmacy, Shaanxi University of Chinese Medicine, Xi'An, 712046, China, e-mail: mengqinghua66@126.com
Xu Long, Jing-Li Liu, Shuan Zhang and Guang-Hui Zhang: School of Pharmacy, Shaanxi University of Chinese Medicine, Xi'An, 712046, China

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.36 × 0.32 × 0.27 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.09 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
θ_{max}, completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	17564, 3233, 0.060
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2199
$N(param)_{refined}$:	236
Programs:	Olex2 [5], SHELX [6], Bruker [7]

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

Atom	x	y	z	U_{iso}*/U_{eq}
O1	0.51281(9)	0.13879(10)	0.17343(9)	0.0584(4)
O2	0.58481(9)	0.26593(12)	0.12781(9)	0.0709(5)
O3	0.19325(9)	0.10459(9)	0.13142(8)	0.0541(4)
N1	0.33369(10)	0.40305(11)	0.11403(10)	0.0492(4)
H1	0.327435	0.464506	0.111885	0.059*
N2	0.25381(17)	0.43032(17)	0.44731(12)	0.0878(7)
C1	0.59677(14)	0.09112(17)	0.18260(16)	0.0717(7)
H1A	0.587326	0.027537	0.202447	0.108*
H1B	0.625982	0.086956	0.133989	0.108*
H1C	0.633016	0.127062	0.217494	0.108*
C2	0.51565(13)	0.22887(15)	0.14597(12)	0.0469(5)
C3	0.42776(12)	0.27212(13)	0.14175(11)	0.0412(5)
C4	0.41775(13)	0.36524(13)	0.12290(11)	0.0451(5)
C5	0.48936(14)	0.43779(15)	0.10879(15)	0.0649(7)
H5A	0.463115	0.498732	0.096318	0.097*
H5B	0.524995	0.444447	0.153921	0.097*
H5C	0.525839	0.416683	0.067103	0.097*
C6	0.26024(12)	0.34614(12)	0.10856(11)	0.0394(5)
C7	0.26559(12)	0.25222(12)	0.12626(10)	0.0382(4)
C8	0.34742(12)	0.21186(12)	0.16387(11)	0.0405(5)
H8	0.356576	0.146392	0.144607	0.049*
C9	0.19078(12)	0.19009(12)	0.11329(11)	0.0405(5)
C10	0.11040(13)	0.23249(14)	0.07638(13)	0.0510(5)
H10A	0.058953	0.195208	0.091460	0.061*
H10B	0.116438	0.226984	0.021373	0.061*
C11	0.09429(12)	0.33774(13)	0.09664(11)	0.0445(5)
C12	0.07020(15)	0.34682(17)	0.18076(13)	0.0651(6)
H12A	0.016957	0.311168	0.190748	0.098*
H12B	0.117547	0.321655	0.211461	0.098*
H12C	0.060886	0.413351	0.193154	0.098*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C13	0.01877(14)	0.37804(17)	0.04839(14)	0.0645(6)
H13A	−0.034654	0.343082	0.059141	0.097*
H13B	0.010244	0.444800	0.060444	0.097*
H13C	0.033181	0.371619	−0.004797	0.097*
C14	0.17852(12)	0.39360(13)	0.07916(12)	0.0478(5)
H14A	0.183742	0.401359	0.024258	0.057*
H14B	0.173880	0.457261	0.101540	0.057*
C15	0.33687(11)	0.20645(12)	0.25016(11)	0.0418(5)
C16	0.31046(12)	0.28630(13)	0.29134(11)	0.0443(5)
H16	0.298384	0.343567	0.265880	0.053*
C17	0.30178(12)	0.28213(14)	0.36984(12)	0.0476(5)
C18	0.27420(15)	0.36518(17)	0.41251(13)	0.0598(6)
C19	0.32006(14)	0.19792(16)	0.40863(13)	0.0600(6)
H19	0.315435	0.195367	0.461576	0.072*
C20	0.34502(16)	0.11848(17)	0.36812(14)	0.0683(7)
H20	0.356706	0.061141	0.393604	0.082*
C21	0.35302(14)	0.12261(14)	0.28983(13)	0.0571(6)
H21	0.369679	0.067639	0.263138	0.069*

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.

Experimental details

H atoms bonded to C and N atoms were positioned geometrically and refined using a riding model, with C—H = 0.96 Å and N—H = 0.86 Å with U_{iso}(H) = 1.2 times U_{eq}(C) and 1.2 times U_{eq}(N).

Comment

4-Arylpolyhydroquinolines belonging to the heterocyclic family, are natural and synthetic products that have been reviewed for their wide range of biological activities as antibacterial, anti-tumor, anti-inflammatory and antioxidant agents, etc. [1–4].

In the title crystal structure (Figure), the dihedral angle of best planes of the six-membered ring containing one nitrogen atom and the adjacent six-membered carbon ring is 86.9°. The dihedral angle of phenyl ring and the six-membered ring containing one nitrogen atom is about 8°. The bond lengths of C—C and C—O are in the range of 1.350(3)−1.527(3) Å and 1.197(3)−1.430(3) Å, respectively. The bond lengths of C—N1 are 1.382(2) and 1.366(2) Å. All of above bond distances are in their normal ranges and they are comparable with previously reported compounds [8].

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