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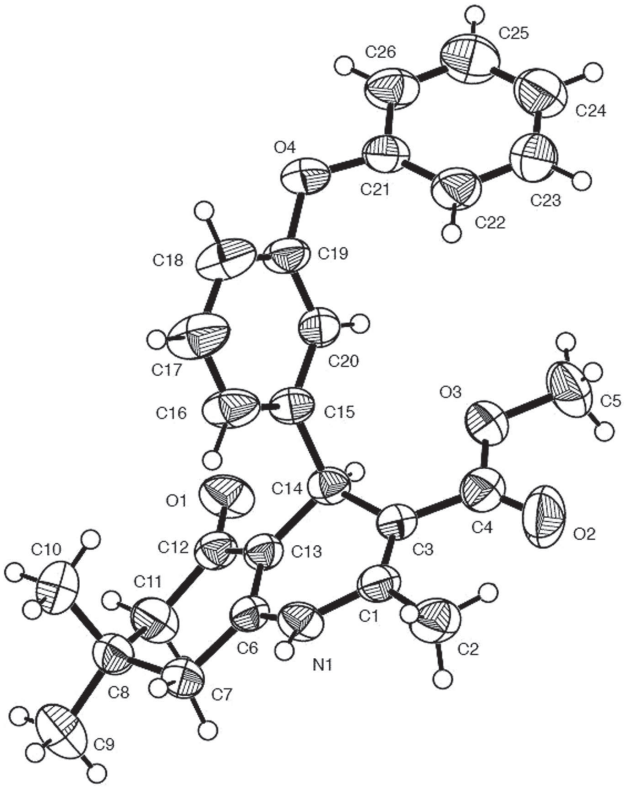
Crystal structure of methyl 4-(3-phenoxyphenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydro quinoline-3-carboxylate, $C_{26}H_{27}NO_4$



https://doi.org/10.1515/ncrs-2018-0135
Received July 12, 2018; accepted October 8, 2018; available online October 15, 2018

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Abstract

$C_{26}H_{27}NO_4$, monoclinic, $P2_1/c$ (no. 14), $a = 12.034(4)$ Å, $b = 15.247(5)$ Å, $c = 12.991(4)$ Å, $\beta = 115.379(6)°$, $V = 2153.6(12)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0531$, $wR_{ref}(F^2) = 0.1447$, $T = 296$ K.

CCDC no.: 1872063

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.

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.26 × 0.22 × 0.14 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.09 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω-scans
θ_{max}, completeness:	25°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	10723, 3776, 0.062
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1993
$N(param)_{refined}$:	281
Programs:	Bruker programs [1], SHELX [2], OLEX2 [3]

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

Atom	x	y	z	U_{iso}*/U_{eq}
N1	0.16092(19)	0.31601(12)	0.22203(17)	0.0465(6)
H1	0.130592	0.340954	0.263662	0.056*
O1	0.46967(16)	0.30769(12)	0.09418(17)	0.0645(6)
O2	0.53423(19)	0.33662(15)	0.2782(2)	0.0861(7)
O3	0.08226(17)	0.14870(13)	−0.10303(15)	0.0628(6)
O4	0.24832(17)	0.45409(13)	−0.27330(15)	0.0670(6)
C1	0.5963(3)	0.3089(2)	0.1114(3)	0.0875(11)
H1A	0.600229	0.298227	0.040199	0.131*
H1B	0.631700	0.365129	0.140462	0.131*
H1C	0.641300	0.264073	0.165015	0.131*
C2	0.4505(3)	0.32186(17)	0.1879(3)	0.0565(8)
C3	0.3201(2)	0.31462(15)	0.1622(2)	0.0427(6)
C4	0.2815(2)	0.33390(16)	0.2417(2)	0.0461(7)
C5	0.3543(3)	0.37321(19)	0.3564(2)	0.0666(9)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H5A	0.302798	0.379746	0.395419	0.100*
H5B	0.422315	0.335525	0.399715	0.100*
H5C	0.384716	0.429630	0.347731	0.100*
C6	0.0899(2)	0.26007(15)	0.1386(2)	0.0402(6)
C7	0.1224(2)	0.23886(16)	0.0542(2)	0.0403(6)
C8	0.2288(2)	0.28412(16)	0.0439(2)	0.0422(6)
H8	0.270546	0.240712	0.017138	0.051*
C9	0.0557(2)	0.17138(16)	−0.0259(2)	0.0439(7)
C10	−0.0451(2)	0.12551(18)	−0.0083(2)	0.0575(8)
H10A	−0.009948	0.075937	0.042039	0.069*
H10B	−0.103847	0.102612	−0.081000	0.069*
C11	−0.1138(2)	0.18249(18)	0.0415(2)	0.0509(7)
C12	−0.1848(2)	0.2544(2)	−0.0424(3)	0.0780(10)
H12A	−0.128674	0.289913	−0.059001	0.117*
H12B	−0.243694	0.228190	−0.111539	0.117*
H12C	−0.226903	0.290223	−0.009666	0.117*
C13	−0.2024(3)	0.1260(2)	0.0679(3)	0.0762(10)
H13A	−0.245698	0.162045	0.099348	0.114*
H13B	−0.260296	0.098877	−0.000877	0.114*
H13C	−0.157281	0.081529	0.121965	0.114*
C14	−0.0188(2)	0.22358(17)	0.1504(2)	0.0480(7)
H14A	−0.057712	0.270212	0.173852	0.058*
H14B	0.009086	0.179562	0.210023	0.058*
C15	0.1875(2)	0.35910(16)	−0.0415(2)	0.0431(6)
C16	0.0969(3)	0.41621(19)	−0.0468(2)	0.0644(8)
H16	0.059445	0.408564	0.002091	0.077*
C17	0.0606(3)	0.4846(2)	−0.1234(3)	0.0789(10)
H17	−0.001536	0.522138	−0.126305	0.095*
C18	0.1152(3)	0.49782(19)	−0.1953(2)	0.0681(9)
H18	0.090722	0.544152	−0.246798	0.082*
C19	0.2057(2)	0.44237(18)	−0.1904(2)	0.0510(7)
C20	0.2418(2)	0.37279(17)	−0.1155(2)	0.0459(7)
H20	0.302703	0.334790	−0.114434	0.055*
C21	0.3700(3)	0.44142(16)	−0.2508(2)	0.0508(7)
C22	0.3921(3)	0.42298(18)	−0.3434(2)	0.0629(8)
H22	0.326952	0.415379	−0.414799	0.075*
C23	0.5111(3)	0.4158(2)	−0.3300(3)	0.0742(9)
H23	0.526774	0.402879	−0.392486	0.089*
C24	0.6070(3)	0.4276(2)	−0.2251(3)	0.0756(9)
H24	0.687565	0.423065	−0.216469	0.091*
C25	0.5842(3)	0.4461(2)	−0.1330(3)	0.0706(9)
H25	0.649444	0.453956	−0.061768	0.085*
C26	0.4654(3)	0.45300(18)	−0.1453(2)	0.0614(8)
H26	0.449853	0.465448	−0.082617	0.074*

Source of material

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

Experimental details

H atoms were positioned geometrically and refined using a riding model, with C—H = 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

The progress achieved in the synthesis of heterocyclic compounds with biological potential is due to improvement of the methodological study of tested substances too. It is known that many quinoline derivatives have biological activity [4].

In the crystal structure of the title compound (*cf*. the figure), the six-membered ring containing nitrogen atom is nearly planar. The structural features are similar as those in compounds sharing the same structural core like the similar 3-methoxy [4] or the 3-cyanophenyl [5] derivative. Compounds sharing the quinoline core of this compound are so numerous that the aforementioned ones are typical examples. The bond lengths of these structures are in agreement with those in previously reported ones.

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