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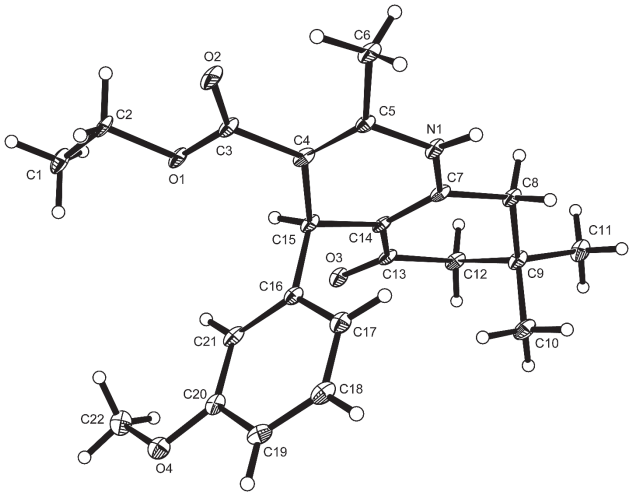
Crystal structure of 4-(3-Methoxy-phenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydro-quinoline-3-carboxylic acid ethyl ester, $C_{22}H_{27}NO_4$



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Abstract

$C_{22}H_{27}NO_4$, triclinic, $P\bar{1}$ (no. 2), $a = 7.468(3)$ Å, $b = 9.442(3)$ Å, $c = 14.134(5)$ Å, $\alpha = 82.725(5)°$, $\beta = 84.077(5)°$, $\gamma = 72.585(5)°$, $V = 941.0(5)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0727$, $wR_{ref}(F^2) = 0.1796$, $T = 296(2)$ K.

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The asymmetric unit of the title 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

The title compound was synthesized according to a reported procedure [5]. A mixture of 5,5-dimethyl-cyclohexane-1,3-dione (10 mmol), 3-methoxy-benzaldehyde (10 mmol),

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.26 × 0.21 × 0.17 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.9 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$2\theta_{max}$, completeness:	50°, >98%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	4743, 3261, 0.041
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2704
$N(param)_{refined}$:	245
Programs:	Bruker programs [6], SHELX [7]

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

Atom	x	y	z	U_{iso}*/U_{eq}
N1	0.6026(3)	0.1298(3)	0.11843(16)	0.0180(6)
H1D	0.4921	0.1524	0.0978	0.022*
O1	1.0086(3)	−0.0713(2)	0.36393(14)	0.0204(5)
O2	0.7263(3)	−0.1111(2)	0.38943(15)	0.0260(5)
O3	1.2161(3)	0.1701(2)	0.06689(14)	0.0212(5)
O4	1.1329(3)	0.4378(2)	0.40568(15)	0.0248(5)
C1	1.2622(4)	−0.1894(4)	0.4635(2)	0.0281(8)
H1A	1.2994	−0.2560	0.5199	0.042*
H1B	1.2763	−0.0937	0.4708	0.042*
H1C	1.3401	−0.2292	0.4091	0.042*
C2	1.0621(4)	−0.1730(3)	0.4492(2)	0.0221(7)
H2A	1.0466	−0.2693	0.4419	0.027*
H2B	0.9826	−0.1339	0.5043	0.027*
C3	0.8341(4)	−0.0535(3)	0.3397(2)	0.0181(6)
C4	0.7908(4)	0.0411(3)	0.2491(2)	0.0176(6)
C5	0.6352(4)	0.0496(3)	0.20711(19)	0.0168(6)
C6	0.4880(4)	−0.0269(3)	0.2434(2)	0.0224(7)
H6A	0.5188	−0.0811	0.3047	0.034*
H6B	0.4835	−0.0948	0.1992	0.034*
H6C	0.3675	0.0464	0.2497	0.034*
C7	0.7415(4)	0.1741(3)	0.06243(19)	0.0165(6)
C8	0.7034(4)	0.2221(3)	−0.0400(2)	0.0178(6)
H8A	0.5722	0.2790	−0.0440	0.021*
H8B	0.7252	0.1344	−0.0735	0.021*
C9	0.8268(4)	0.3174(3)	−0.0897(2)	0.0196(7)
C10	0.7643(4)	0.4727(3)	−0.0553(2)	0.0258(7)

Table 2 (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
H10A	0.7754	0.4643	0.0124	0.039*
H10B	0.6357	0.5208	−0.0691	0.039*
H10C	0.8424	0.5307	−0.0876	0.039*
C11	0.8093(4)	0.3323(4)	−0.1969(2)	0.0258(7)
H11A	0.8861	0.3918	−0.2286	0.039*
H11B	0.6805	0.3794	−0.2105	0.039*
H11C	0.8504	0.2351	−0.2193	0.039*
C12	1.0303(4)	0.2388(3)	−0.0669(2)	0.0199(7)
H12A	1.0760	0.1513	−0.1016	0.024*
H12B	1.1058	0.3052	−0.0903	0.024*
C13	1.0608(4)	0.1911(3)	0.0374(2)	0.0169(6)
C14	0.9033(4)	0.1659(3)	0.09989(19)	0.0159(6)
C15	0.9264(4)	0.1288(3)	0.20561(19)	0.0168(6)
H15A	1.0552	0.0654	0.2145	0.020*
C16	0.8953(4)	0.2689(3)	0.25579(19)	0.0172(6)
C17	0.7256(4)	0.3810(3)	0.2511(2)	0.0215(7)
H17A	0.6322	0.3730	0.2151	0.026*
C18	0.6963(4)	0.5044(4)	0.3003(2)	0.0240(7)
H18A	0.5813	0.5782	0.2981	0.029*
C19	0.8320(4)	0.5206(3)	0.3521(2)	0.0223(7)
H19A	0.8098	0.6045	0.3847	0.027*
C20	1.0042(4)	0.4105(3)	0.35557(19)	0.0194(7)
C21	1.0338(4)	0.2855(3)	0.3071(2)	0.0193(7)
H21A	1.1488	0.2118	0.3094	0.023*
C22	1.3154(4)	0.3342(4)	0.4058(2)	0.0281(8)
H22A	1.3924	0.3659	0.4440	0.042*
H22B	1.3709	0.3291	0.3414	0.042*
H22C	1.3062	0.2374	0.4318	0.042*

ammonium acetate (10 mmol) and ethyl acetoacetate (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 placed in calculated positions and included in the refinement in the riding model approximation, with $U_{iso}(H) = 1.2U_{eq}(C)$. H atoms of the methyl group were allowed to rotate with a fixed angle around the C—C bond to best fit the experimental electron density, with $U_{iso}(H) = 1.5U_{eq}(C)$. The quality of the crystals were not 100% satisfactory, which is expressed by large R factors and the refined U_{anis} ellipsoids.

Discussion

4-Arylpolyhydroquinoline is one of the most commonly encountered heterocyclic moiety, which forms the important component of pharmacophores for a number of compounds having medicinal significance including anticancer, antiviral, antibacterial, antioxidant, anti-HIV, antihyperglycemic and anti-dyslipidemic activities [1, 2]. Recognizing the considerable importance of the compounds, research focused on the synthesis of new 4-arylpolyhydroquinoline [3, 4].

In the crystal structure of the title compound, the six-membered ring containing a nitrogen atom is obviously folded. The same is true for the annulated six-membered ring (*cf*. the figure). The bond lengths and angles are in the expected ranges.

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