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The crystal structure of ethyl 2-methyl-5-oxo-4-(2-methoxyphenyl)-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate, $C_{20}H_{23}N_2O_4$

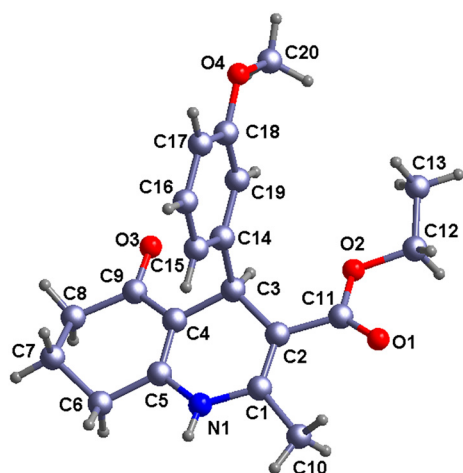


Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.29 × 0.24 × 0.21 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.09 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω scans
$\theta_{\max}$, completeness:	25.2°, 99 %
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	4,498, 3,113, 0.014
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2,534
$N(\text{param})_{\text{refined}}$:	229
Programs:	Bruker, ¹ Olex2, ² SHELX ^{3,4}

1 Source of materials

All reagents were purchased from commercial reagent companies and used directly without further purification. The title compound was prepared by a modified literature method with minor optimizations.⁵ A solution containing 2-methoxybenzaldehyde (0.272 g, 2 mmol), cyclohexane-1,3-dione (0.224 g, 2 mmol), ammonium acetate (0.154 g, 20 mmol), and ethyl acetoacetate (0.260 g, 2 mmol) in ethanol (50 mL) was refluxed for 5 h followed by ambient temperature cooling. Slow solvent evaporation at room temperature afforded colorless crystalline product, achieving a 65.3 % isolated yield based on 2-methoxybenzaldehyde.

2 Experimental details

The structure was solved by Direct Methods with the SHELXS-2018 program. All H-atoms from C and N atoms were positioned with idealized geometry and refined isotropically ($U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ or $U_{\text{eq}}(\text{N})$) using a riding model with C–H = 0.930, 0.960, 0.970 and 0.980 Å, and C–H = 0.860 Å, respectively.

3 Comment

To date, some ethyl 2-methyl-5-oxo-4-(aryl)-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate based 4-arylpolyhydroquinoline derivatives have been reported for their biological activity,

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Abstract

$C_{20}H_{23}N_2O_4$, triclinic, $P\bar{1}$ (no. 2), $a = 7.519(5)$ Å, $b = 9.445(7)$ Å, $c = 13.385(10)$ Å, $\alpha = 80.837(11)^\circ$, $\beta = 82.430(12)^\circ$, $\gamma = 69.438(11)^\circ$, $V = 875.8(11)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0445$, $wR_{\text{ref}}(F^2) = 0.1382$, $T = 296(1)$ K.

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The molecular structure is shown in the figure. Table 1 contains the crystallographic data and the list of the atoms including atomic coordinates and displacement parameters can be found in the cif-file attached to this article.

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including ethyl 2-methyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate,⁵ ethyl 2-methyl-4-(1-naphthyl)-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate,⁶ ethyl 2-methyl-5-oxo-4-phenyl-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate,⁷ ethyl 2-methyl-5-oxo-4-(2,3,4-trimethoxyphenyl)-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate,⁸ ethyl 2-methyl-4-(2-methylphenyl)-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate,⁹ and ethyl 2-methyl-5-oxo-4-(3,4,5-trimethoxyphenyl)-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate.¹⁰ Herein we reported the crystal structure of the title compound, ethyl 2-methyl-5-oxo-4-(2-methoxyphenyl)-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate, C₂₀H₂₃N₂O₄.

The asymmetric unit contains only one molecule of the title compound. The adjacent ketone ring adopts a flattened chair conformation with the aryl group and the six-membered nitrogen heterocyclic ring are nearly planar, respectively. A one-dimensional chain structure is formed by connecting a classical hydrogen bond N1–H1...O3 and a non-classical hydrogen bond C10–H10A...O3. All bond lengths are normal and consistent with similar structures reported in the literature.^{5–11}

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