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Methyl 2-methyl-5-oxo-4-*p*-tolyl-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate

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Key indicators

Single-crystal X-ray study
T = 193 K
Mean σ(C–C) = 0.002 Å
R factor = 0.046
wR factor = 0.124
Data-to-parameter ratio = 16.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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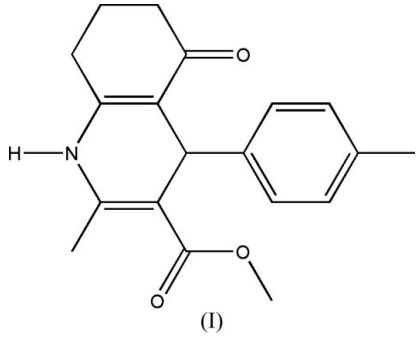
The title compound, $C_{19}H_{21}NO_3$, was synthesized by the reaction of cyclohexane-1,3-dione, 4-methylbenzaldehyde and methyl 3-aminobut-2-enoate in the presence of benzyltriethylammonium chloride in an aqueous medium. X-ray single-crystal analysis reveals that the heterocyclic ring and the fused six-membered ring adopt boat and envelope conformations, respectively. In the crystal structure, molecules are linked by intermolecular N—H···O hydrogen bonds.

Comment

Some derivatives of quinoline are naturally occurring alkaloids and are very attractive for their various bioactivities. For example, they have calcium modulatory properties (Rose & Draeger, 1992), antibacterial activity (Davies *et al.*, 2005), fungicidal activity (Warrior *et al.*, 2005), *etc*. As part of our programme aimed at developing new and environmentally friendly methodologies for the preparation of fine chemicals (Shi *et al.*, 2004), we have synthesized the title compound, (I), in an aqueous medium. We report here the synthesis and crystal structure of (I).



(I)

In the molecular structure of (I) (Fig. 1, Table 1), atoms C3 and N1 deviate from the mean plane of atoms C1/C2/C4/C5 in the same direction, by 0.368 (1) and 0.166 (1) Å, respectively, so the heterocyclic ring adopts a boat conformation. The fused cyclohexene ring can be regarded as having an envelope conformation, with atom C8 out of the plane of atoms C1/C2/C6/C7/C9 by 0.687 (1) Å. In addition, the benzene ring is almost perpendicular to the plane of atoms C1/C2/C4/C5, with a dihedral angle between them of 83.58 (5)°.

The crystal packing of (I) is stabilized by an intermolecular hydrogen bond (Table 2, Fig. 2).

Experimental

The title compound was prepared by the reaction of 1,3-cyclohexanedione (0.22 g, 2 mmol), 4-methylbenzaldehyde (0.24 g, 2 mmol) and methyl-3-aminobut-2-enoate (0.23 g, 2 mmol) in the

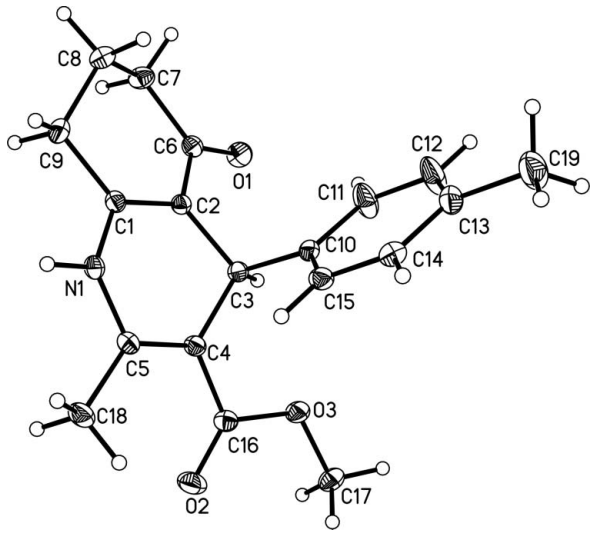


Figure 1
The structure of (I), showing 40% probability displacement ellipsoids and the atom-numbering scheme.

presence of triethylbenzylammonium chloride (0.2 g) in water (10 ml) at 298 K for 25 h (yield 83%). Single crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution.

Crystal data

$C_{19}H_{21}NO_3$
M_r = 311.37
Monoclinic, $P2_1/c$
a = 7.8338 (15) Å
b = 15.095 (3) Å
c = 13.962 (3) Å
β = 105.436 (5)°
V = 1591.5 (5) Å^3
Z = 4
D_x = 1.300 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7332 reflections
θ = 3.0–27.5°
μ = 0.09 mm^{-1}
T = 193 (2) K
Block, colourless
0.73 × 0.62 × 0.25 mm

Data collection

Rigaku Mercury diffractometer
ω scans
Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.939, T_{max} = 0.978
17357 measured reflections
3637 independent reflections
3378 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 27.5°
$h = -8 \rightarrow 10$
$k = -16 \rightarrow 19$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.124
S = 1.05
3637 reflections
216 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0636P)^2 + 0.5891P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C6	1.2354 (16)	O3—C17	1.4418 (15)
O2—C16	1.2108 (16)	N1—C1	1.3696 (16)
O3—C16	1.3495 (16)	N1—C5	1.3878 (16)
C16—O3—C17	115.62 (10)	C1—N1—C5	121.74 (10)

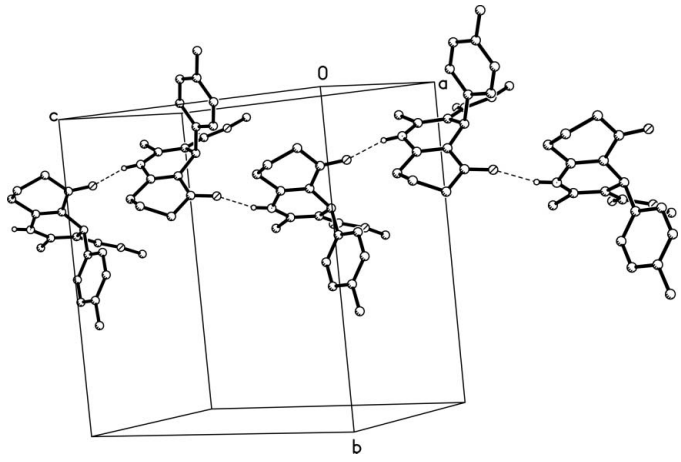


Figure 2
The molecular packing in the crystal structure of (I). Intermolecular hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonds have been omitted.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.85 (2)	2.02 (2)	2.8343 (15)	160 (2)

Symmetry code: (i) $x, -y+\frac{1}{2}, z+\frac{1}{2}$.

Atom H1, bonded to N1, was found in a Fourier map and refined with free coordinates and an isotropic displacement parameter. Other H atoms were positioned geometrically and treated as riding on their parent atoms with C—H = 0.95 Å and U_{iso}(H) = 1.2U_{eq}(C) for aromatic CH, C—H = 0.98 Å and U_{iso}(H) = 1.5U_{eq}(C) for methyl CH_3, C—H = 0.99 Å and U_{iso}(H) = 1.2U_{eq}(C) for methylene CH_2, and C—H = 1.00 Å and U_{iso}(H) = 1.2U_{eq}(C) for methine CH.

Data collection: *CrystalClear* (Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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