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1-Cyclopentyl-4-(4-fluorophenyl)-7,7-dimethyl-4,6,7,8-tetrahydroquinoline-2,5-dione

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Key indicators

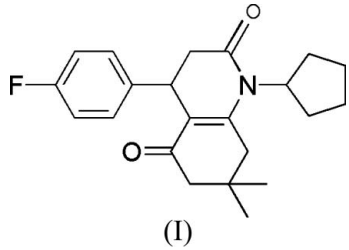
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.045
wR factor = 0.122
Data-to-parameter ratio = 14.9

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

The title compound, $C_{22}H_{26}FNO_2$, was synthesized by the reaction of 4-fluorobenzaldehyde, Meldrum's acid, dimedone and cyclopentylamine hydrochloride, together with sodium acetate, in ethanol under microwave irradiation. The pyridone ring adopts a screw-boat conformation and the cyclohexenone ring an envelope conformation.

Comment

Since the discovery of the pharmacological effects of 1,4-dihydropyridines (1,4-DHPs) as calcium channel blockers (Janis *et al.*, 1987), a great deal of work has been directed towards the synthesis of novel 1,4-DHPs as possible calcium antagonists (Bossert & Vater, 1989). In fact, it is well established that slightly modified structures of DHP exhibit a calcium agent effect (Schramm *et al.*, 1983). We have reported the synthesis of substituted 2,5-dioxo-1,2,3,4,5,6,7,8-octahydroquinolines (Tu *et al.*, 2001), and recently we achieved the introduction of a cyclopentyl group on the N atom. We report here the X-ray crystal structure of the title compound, 1-cyclopentyl-7,7-dimethyl-4-(4-fluorophenyl)-4,6,7,8-tetrahydroquinoline-2,5-dione, (I).



(I)

In (I), the pyridone ring has a screw-boat conformation (Fig.1), with atoms C16 and C17 deviating from the C7/C8/C9/N1 basal plane by 0.87 (3) and 0.47 (3) Å, respectively. The cyclohexenone ring has an envelope conformation, with atom C3 deviating from the C1/C2/C6/C7/C8 plane by 0.65 (3) Å. The dihedral angle between the benzene ring and the basal plane of the pyridone ring is 113.29 (7)°.

Experimental

The title compound, (I), was prepared by the reaction of 4-fluorobenzaldehyde (1 mmol), Meldrum's acid (1 mmol), dimedone (1 mmol) and cyclopentylamine hydrochloride (1.5 mmol), in the presence of sodium acetate (1.5 mmol), in ethanol under microwave irradiation (yield 92%). Single crystals suitable for X-ray diffraction were obtained by slow evaporation of an ethanol (95%) solution (m.p. 405–406 K)

Crystal data

$C_{22}H_{26}FNO_2$	D_x = 1.260 Mg m^{-3}
M_r = 355.44	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 2854 reflections
a = 10.3031 (16) Å	θ = 2.2–25.5°
b = 10.7991 (17) Å	μ = 0.09 mm^{-1}
c = 17.207 (3) Å	T = 294 (2) K
β = 101.942 (3)°	Prism, colorless
V = 1873.1 (5) Å^3	0.28 × 0.26 × 0.24 mm
Z = 4	

Data collection

Bruker SMART CCD-1000 area-detector diffractometer	3825 independent reflections
	2433 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.030
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	θ_{max} = 26.4°
	$h = -12 \rightarrow 8$
T_{min} = 0.972, T_{max} = 0.979	$k = -13 \rightarrow 13$
10342 measured reflections	$l = -18 \rightarrow 21$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0531P)^2 + 0.3765P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.045	
$wR(F^2)$ = 0.122	
S = 1.01	$(\Delta/\sigma)_{max} < 0.001$
3825 reflections	$\Delta\rho_{max}$ = 0.14 e Å^{-3}
256 parameters	$\Delta\rho_{min}$ = −0.23 e Å^{-3}
H-atom parameters constrained	

H atoms were positioned geometrically and refined as riding, with C—H = 0.93–0.98 Å and with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C) for other H atoms. In the cyclopentane ring the two central methylene groups, C20, C21 and their attached H atoms, are disordered over two positions, with refined site occupancy factors of 0.590 (13) and 0.410 (13). Likewise, the H atoms attached to C19 and C22 are disordered, with the same occupancy factors. C—C bond lengths in the cyclopentane ring have been restrained to 1.52 (1) Å; 1–3 distances have been restrained to 2.5 (1) Å. Atom C21′ was restrained to approximate isotropic behaviour.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; 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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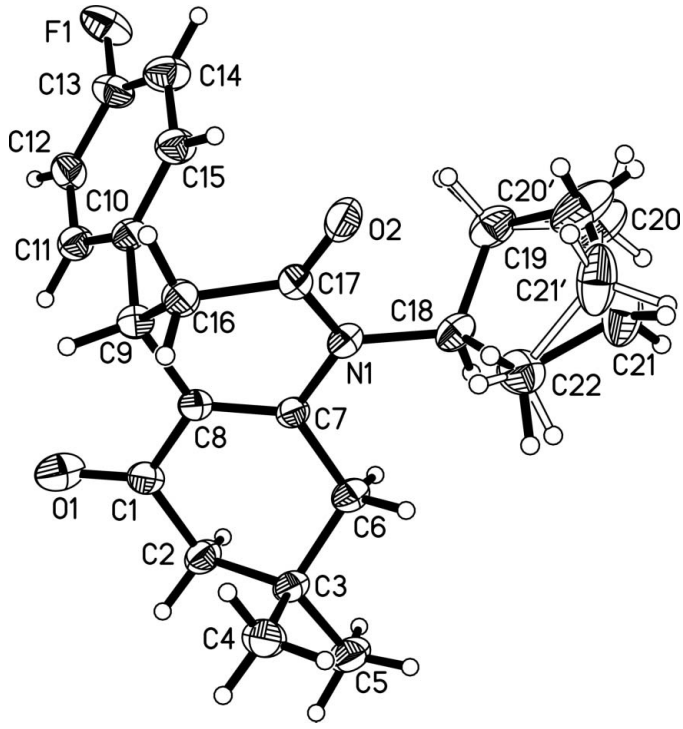

Figure 1
A view of the molecular structure of (I), showing 30% probability displacement ellipsoids. Both components are shown for the disordered methylene groups C20 and C21.

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