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4-(4-Chlorophenyl)-1,7,7-trimethyl-1,2,3,4,5,6,7,8-octahydroquinoline-2,5-dione

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.008 Å
R factor = 0.059
wR factor = 0.178
Data-to-parameter ratio = 10.5

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

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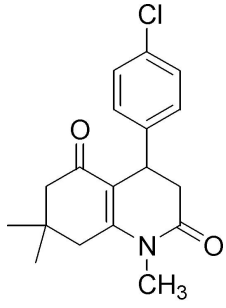
The title compound, $C_{18}H_{20}ClNO_2$, has been synthesized by the reaction of 4-chlorobenzaldehyde, Meldrum's acid, dimedone and CH_3NH_3Cl(NaOAc) in ethanol under microwave irradiation. The pyridone ring adopts a screw-boat conformation and the cyclohexenone ring an envelope conformation.

Comment

Since the discovery of the pharmocological 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-octahydroquinoline (Tu *et al.*, 2001). Recently, we achieved the introduction of a methyl group on the N atom. We report here the X-ray crystal structure of the title compound, 4-(4-chlorophenyl)-1,7,7-trimethyl-1,2,3,4,5,6,7,8-octahydroquinoline- 2,5-dione, (I).



(I)

In (I), the pyridone ring has a screw-boat conformation (Fig. 1), with atoms C1 and C2 deviating from the C3/C4/C9/N1 basal plane by 0.38 (3) and 0.81 (2) Å, respectively, and the cyclohexenone ring has an envelope conformation with atom C7 deviating from the C8/C9/C4/C5/C6 plane by 0.24 (5) Å. The dihedral angle between the benzene ring and the basal plane of the pyridone ring is 78.5 (6)°.

Experimental

The title compound, (I), was prepared by the reaction of 4-chlorobenzaldehyde (1 mmol), Meldrum's acid (1 mmol), dimedone (1 mmol) and CH_3NH_3Cl(NaOAc) (1.5 mmol) in ethanol under microwave irradiation (Tu *et al.*, 2001; yield 86%). Single crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol (95%) solution (m.p. 506–507 K).

Crystal data

$C_{18}H_{20}ClNO_2$
M_r = 317.80
Orthorhombic, $Pca2_1$
a = 17.492 (3) Å
b = 9.805 (2) Å
c = 9.634 (2) Å
V = 1652.3 (7) Å^3
Z = 4
D_x = 1.278 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1544 reflections
θ = 2.3–19.8°
μ = 0.24 mm^{-1}
T = 298 (2) K
Block, yellow
0.29 × 0.25 × 0.17 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.934, T_{max} = 0.961
8088 measured reflections
2127 independent reflections
1321 reflections with $I > 2\sigma(I)$
R_{int} = 0.050
θ_{max} = 25.0°
$h = -20 \rightarrow 18$
$k = -11 \rightarrow 11$
$l = -11 \rightarrow 7$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.179
S = 1.02
2127 reflections
202 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1083P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}
Absolute structure: Flack (1983), 568 Friedel pairs
Flack parameter = −0.14 (17)

Table 1
Selected geometric parameters (Å, °).

N1—C1	1.392 (8)	C2—C3	1.514 (8)
N1—C9	1.410 (6)	C3—C4	1.515 (7)
C1—C2	1.455 (8)	C4—C9	1.354 (7)
C1—N1—C9	119.8 (4)	C2—C3—C4	106.2 (4)
N1—C1—C2	118.0 (5)	C9—C4—C3	121.8 (4)
C1—C2—C3	112.9 (5)	C4—C9—N1	119.4 (4)

The H atoms were located geometrically and treated as riding, with C—H distances of 0.93–0.98 Å and with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C) for the others.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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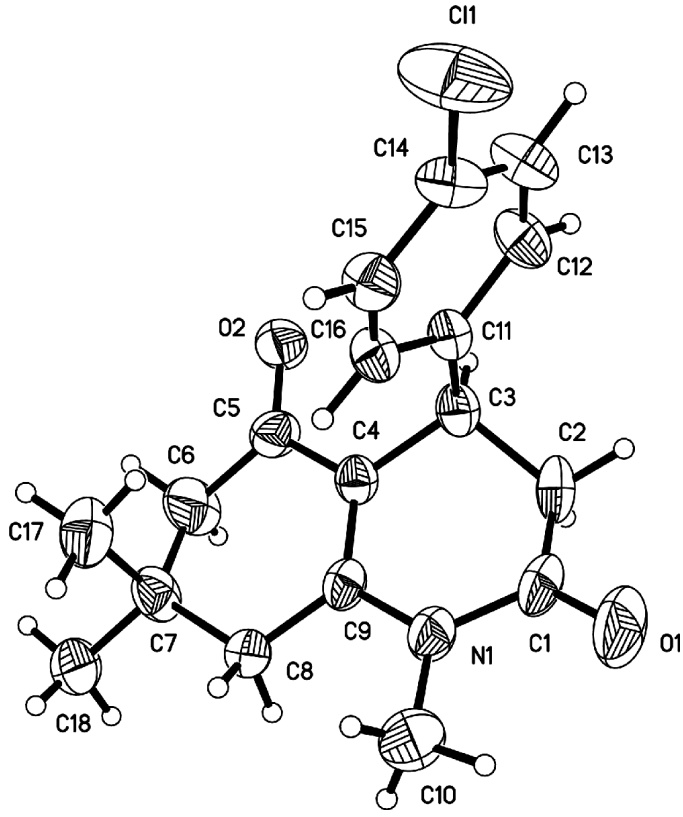

Figure 1
A view of the molecular structure of (I), showing 30% probability displacement ellipsoids.

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