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9-(4-Chlorophenyl)-3,3,6,6-tetramethyl-1,2,3,4,-5,6,7,8,9,10-decahydroacridine-1,8-dione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.010 Å
R factor = 0.051
wR factor = 0.208
Data-to-parameter ratio = 8.0

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

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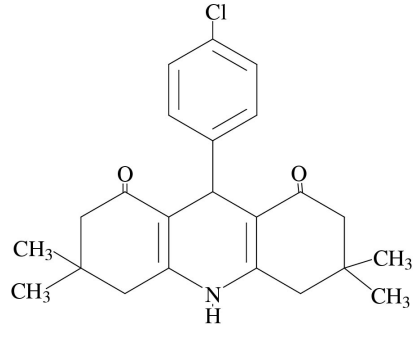
In the molecule of the title compound, $C_{23}H_{26}ClNO_2$, the dihydropyridine plane is approximately bisected by the plane of the orthogonal phenyl ring and the two fused rings are in the same boat main plane. A striking feature of the title compound is seen in the formation of a linear structure through N—H···O hydrogen bonds.

Comment

A great deal of work has been directed toward the synthesis of novel derivatives of 1,4-dihydropyridines (1,4-DHP) because they can act as calcium channel antagonists or agonists (Goldmann & Stoltefuss, 1991). Of particular interest is knowing which conformation in 1,4-DHPs gives optimum results and, consequently, the relationship between the conformation and the pharmacological effect. It has been proved that cyclohexanone rings in the 1,4-DHP system lead to compounds with a positive inotropic effect, that is, they promote instead of blocking the entry of calcium to the intracellular space due to conformational changes (Martin *et al.*, 1995). Furthermore, although the crystal structures of many aryl-ring substituent derivatives of 1,4-DHPs having the antagonist activity have been determined by X-ray studies (Fossheim, 1985, 1986; Fossheim *et al.*, 1988), that of the cyclohexanone-ring substituted 1,4-DHP is still unknown. Taking into account the above-mentioned aspects, we report herein the synthesis and crystal structure of a new 1,4-DHP compound with cyclohexanone rings, namely 9-(4-chlorophenyl)-3,3,6,6-tetramethyl-1,2,3,4,5,6,7,8,9,10-decahydroacridine-1,8-dione, (I).



(I)

As in the previously determined structures of 4-aryl-substituted 1,4-DHPs, there exists a flattened-boat conformation in (I) in which the aryl substituent is in a pseudo-axial

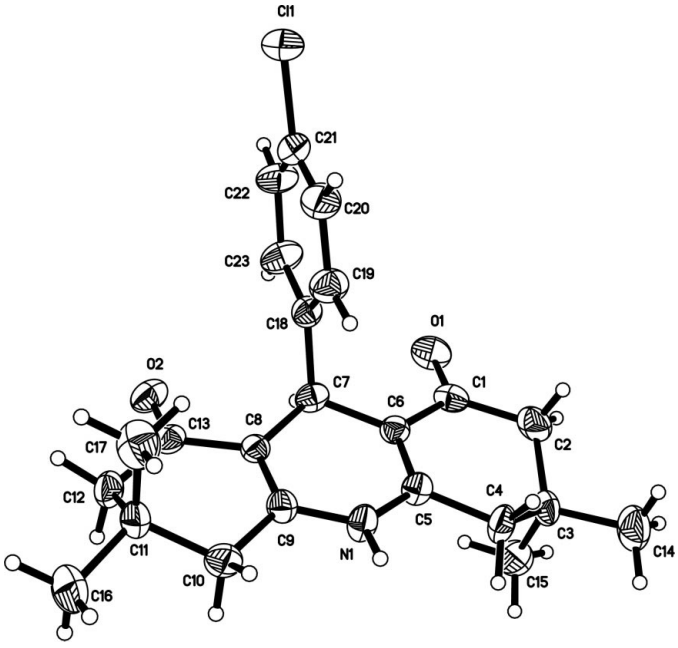


Figure 1
View of the title compound shown with 30% probability ellipsoids.

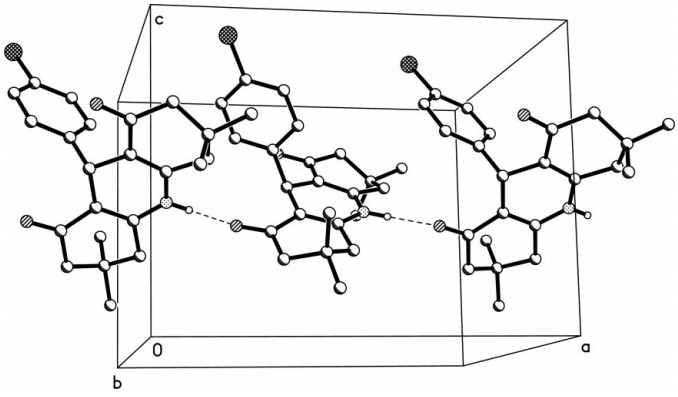


Figure 2
Packing diagram of the title compound.

position, orthogonal to the dihydropyridine plane, as shown in Fig. 1. The dihydropyridine plane is approximately bisected by the plane of the phenyl ring, indicated by the magnitude of the dihedral angle between the two planes, which is 87.2°. The two fused rings are in the same plane, with atoms C3 and C11 displaced from this plane.

The sum of the bond angles around the amino N atom (359.9°) shows that it is essentially sp^2 hybridized, which is similar to previous results (Fossheim, 1987). The H atom thus deviates only slightly from the plane containing C5, C9 and N1. Fossheim (1987) predicted that, for the above reason, the requirement for a strong linear hydrogen bond is best fulfilled when the acceptor atom of the receptor lies approximately in the DHP ring. In this compound, the formation of N—H···O hydrogen bonds links the molecules to form linear chains, as shown in Fig. 2.

Experimental

Compound (I) was prepared by the reaction of 4-chlorobenzaldehyde (2 mmol), dimedone (4 mmol) and ammonium bicarbonate (3 mmol) under microwave irradiation for 4 min. The reaction mixture was cooled and washed with ethanol. The yellow solid obtained was purified by recrystallization from 95% ethanol producing single crystals suitable for X-ray diffraction. Yield: 92%; m.p.: 569–571 K. Analysis calculated for the title compound: C 71.95, H 6.83, N 3.65%; found: C 71.66, H 6.99, N 3.42%. FT–IR data (KBr pellet, cm^{-1}): 3383 (NH), 1623 (C═O), 1603 (N—C═O). ^{1}H NMR ($CDCl_3$, δ, p.p.m.): 0.93 (*s*, 6H, 2-CH_3), 1.05 (*s*, 6H, 2 CH_3), 2.21–2.25 (*m*, 8H, 4-CH_2), 5.06 (*s*, 1H, CH), 7.12–7.32 (*m*, 4H, ArH), 7.72 (*s*, 1H, NH).

Crystal data

$C_{23}H_{26}ClNO_2$
M_r = 383.90
Orthorhombic, $Pna2_1$
a = 14.125 (3) Å
b = 14.118 (3) Å
c = 10.719 (2) Å
V = 2137.5 (7) Å^3
Z = 4
D_x = 1.193 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1922 reflections
θ = 2.0–25.0°
μ = 0.20 mm^{-1}
T = 293 (2) K
Prism, yellow
0.35 × 0.30 × 0.15 mm

Data collection

Bruker CCD diffractometer
ω scans
Absorption correction: multi-scan [*SAINT* (Bruker, 1998) and *SADABS* (Sheldrick, 1997)]
T_{min} = 0.935, T_{max} = 0.971
1983 measured reflections
1983 independent reflections
1361 reflections with $I > 2\sigma(I)$
θ_{max} = 25.0°
$h = 0 \rightarrow 16$
$k = -16 \rightarrow 0$
$l = 0 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.208
S = 1.03
1983 reflections
248 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1373P)^2 + 0.7655P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: none
Absolute structure: Flack (1983)
Flack parameter = 0.2 (2)

Table 1
Selected geometric parameters (Å, °).

Cl1—C21	1.729 (8)	N1—C9	1.331 (8)
O1—C1	1.242 (8)	N1—C5	1.359 (8)
O2—C13	1.220 (7)		
C9—N1—C5	123.1 (5)	O2—C13—C8	119.3 (6)
O1—C1—C6	120.5 (6)	O2—C13—C12	121.1 (6)
O1—C1—C2	121.7 (6)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O2^{i}	0.86	1.88	2.735 (6)	178

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

The methyl groups were allowed to rotate about their local threefold axis.

Data collection and cell refinement: *SMART* (Bruker, 1998); data reduction: *SAINT* (Bruker, 1998); structure solution: *SHELXS*97 (Sheldrick, 1997); structure refinement: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998).

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