Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

Liang-Ce Rong,* Xiao-Yue Li, Chang-Sheng Yao, Hai-Ying Wang and Da-Qing Shi

Department of Chemistry, Xuzhou Normal University, Xuzhou 221116, People's Republic of China

Correspondence e-mail: lcrong2005@yahoo.com

Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.010 Å
R factor = 0.078
wR factor = 0.269
Data-to-parameter ratio = 14.1

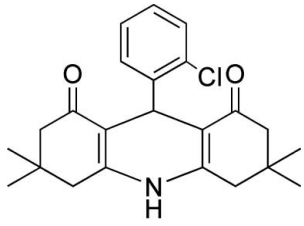
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 25 April 2006
Accepted 12 May 2006

The title compound, $C_{23}H_{26}ClNO_2$, was synthesized by the reaction of 5,5-dimethylcyclohexane-1,3-dione with 4-chloro-*N*-(2-chlorobenzylidene)benzenamine under solvent-free conditions using triethylbenzylammonium chloride as catalyst at 353 K. The dihydropyridine ring of the molecule adopts a half-chair conformation.

Comment

1,4-Dihydropyridines (1,4-DHPS) are well known compounds for their pharmacological profile in calcium channel modulations (Bossert *et al.*, 1981). In particular, dihydropyridine drugs such as nifedipine, nicardipine, amlodipine and others are effective cardiovascular agents for the treatment of hypertension. They have many significant biological activities: anti-atherosclerotic, antitumor, hepatoprotective, antidiabetic and anti-ischemic agents (Mauzeral & Westheimer, 1955; Di Stilo *et al.*, 1998; Shan *et al.*, 2004; Suarez *et al.*, 2003).



(I)

The solvent-free reaction has attracted great attention in recent years (Tanaka & Toda, 2000) and has been proved to have many advantages: high efficiency and selectivity, easy separation and purification, mild reaction conditions, reduced pollution, and low cost. Solvent-free reactions have performed well recently (Kaupp *et al.*, 2003; Goud *et al.*, 1995; Annunziata *et al.*, 1997).

We report here the crystal structure of the title compound, (I), which has been synthesized by the solvent-free reaction of 5,5-dimethylcyclohexane-1,3-dione and 4-chloro-*N*-(2-chlorobenzylidene)benzenamine at 353 K.

In (I), the dihydropyridine ring is in a half-chair conformation, with atom C7 deviating from the C1/C6/C8/C13/N1 plane by 0.121 (8) Å (Fig. 1). Both cyclohexene rings adopt half-chair conformations: atom C3 deviates from the C1/C2/C4–C6 plane by 0.639 (9) Å and atom C11 deviates from the C8–C10/C12/C13 plane by 0.644 (9) Å.

The dihedral angle between the C1/C6/C8/C13/N1 and C1/C2/C4–C6 planes is 3.3 (4)°. The dihedral angle between the C1/C6/C8/C13/N1 and C8–C10/C12/C13 planes is 2.4 (4)°.

Experimental

Compound (I) was prepared by the reaction of 5,5-dimethylcyclohexane-1,3-dione (4 mmol) with 4-chloro-*N*-(2-chlorobenzylidene)benzenamine (2 mmol) under solvent-free condition using TEBA (triethylbenzylammonium chloride) as catalyst at 353 K.

Crystal data

$C_{23}H_{26}ClNO_2$
M_r = 383.90
Monoclinic, $C2/c$
a = 17.004 (6) Å
b = 11.429 (4) Å
c = 21.741 (10) Å
β = 111.858 (15)°
V = 3921 (3) Å^3
Z = 8
D_x = 1.301 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 298 (2) K
Block, colourless
0.24 × 0.15 × 0.13 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.951, T_{max} = 0.973
9874 measured reflections
3463 independent reflections
1617 reflections with $I > 2\sigma(I)$
R_{int} = 0.123
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.078
$wR(F^2)$ = 0.269
S = 1.07
3463 reflections
245 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0343P)^2 + 19.16P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.64 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}

All H atoms were positioned geometrically and treated as riding, with C—H distances of 0.93–0.98 Å and N—H = 0.86 Å, and with U_{iso}(H) set at 1.2U_{eq}(C,N). or 1.5U_{eq}(methyl C).

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

The authors thank the Natural Science Foundation of the Education Committee of Jiangsu Province and the Natural Science Foundation (No. 04XLB14) of Xuzhou Normal University for financial support.

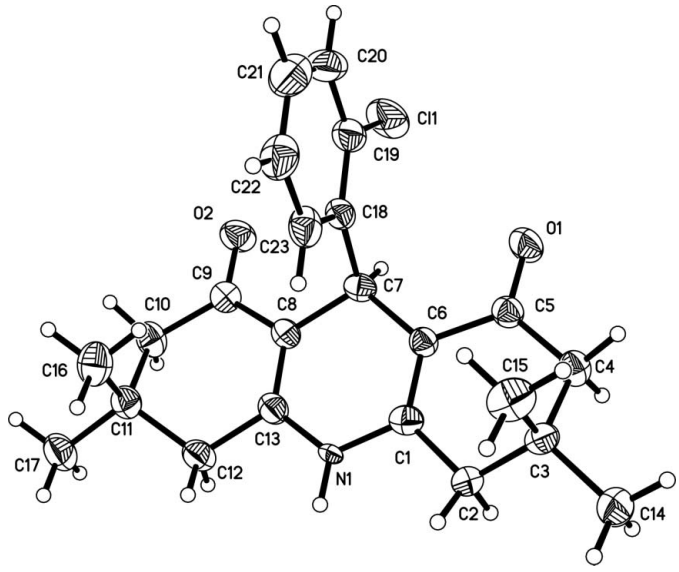


Figure 1
The molecular structure of (I), showing 40% probability displacement ellipsoids and the atom-numbering scheme.

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