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9-(3,4-Dichlorophenyl)-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 = 298 K
Mean σ(C–C) = 0.009 Å
R factor = 0.069
wR factor = 0.186
Data-to-parameter ratio = 14.8

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

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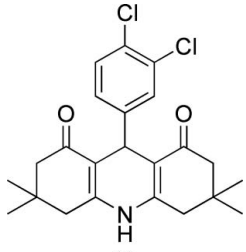
The title compound, $C_{23}H_{25}Cl_2NO_2$, was synthesized by the reaction of 5,5-dimethylcyclohexane-1,3-dione with 3,4-dichlorobenzaldehyde and ammonium acetate under solvent-free conditions at 353 K. X-ray analysis reveals that the dihydropyridine and cyclohexene rings adopt envelope conformations.

Comment

The solvent-free reaction has attracted great attention in recent years (Tanaka & Toda, 2000) and has proved to have many advantages: reduced pollution, low costs, and simplicity in process and handling. 1,4-Dihydropyridines are well known compounds, as a consequence of their pharmacological profile as calcium channel modulators (Janis *et al.*, 1987), which have become almost indispensible for the treatment of cardiovascular diseases such as hypertension, cardiac arrhythmia and angina. The discovery of acridines as antimalarial and antitumor agents has attracted the attention of organic chemists and thus led to intensive interest in the synthesis of several drugs based on acridine (Khurana *et al.*, 1990; Matsumoto *et al.*, 1983). We report here the crystal structure of the title compound, (I), which was synthesized by the solvent-free reaction of 5,5-dimethylcyclohexane-1,3-dione and 3,4-dichlorobenzaldehyde and ammonium acetate at 353 K.



(I)

In (I) (Fig. 1), the dihydropyridine ring is in an envelope conformation, with atom C7 deviating from the C1/C6/C8/C13/N1 plane by 0.189 (8) Å. Both cyclohexene rings adopt envelope conformations: atom C3 deviates from the C1/C2/C4–C6 plane by 0.659 (9) Å and atom C11 deviates from the C8–C10/C12/C13 plane by 0.602 (9) Å. The dihedral angle between the C1/C6/C8/C13/N1 and C1/C2/C4–C6 planes is 6.8 (4)°, and that between the C1/C6/C8/C13/N1 and C8–C10/C12/C13 planes is 3.5 (4)°. The dichlorophenyl group is twisted away from the C1/C6/C8/C13/N1 plane by 83.2 (1)°.

The crystal packing shows that intermolecular N—H···O hydrogen bonds (Table 1) link the molecules into a chain along the *a* axis (Fig. 2).

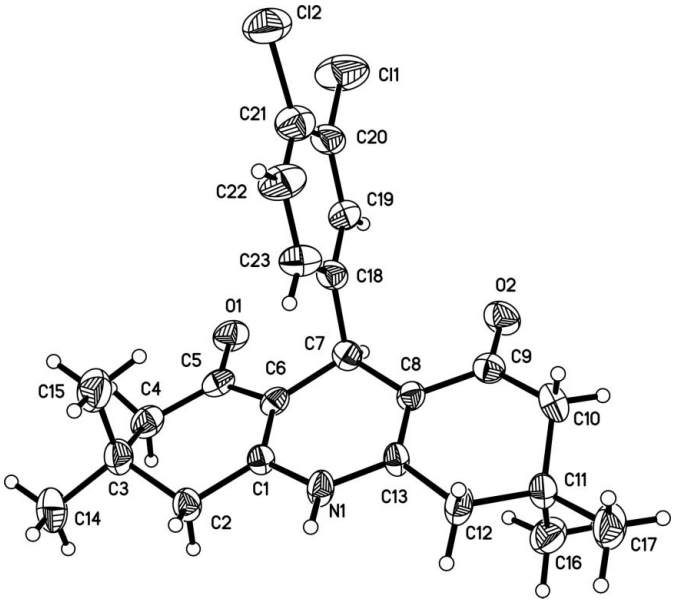


Figure 1
The structure of (I), showing 40% probability displacement ellipsoids and the atom-numbering scheme.

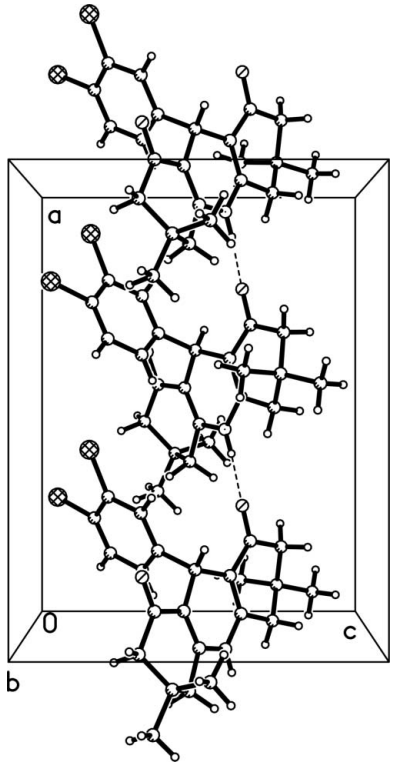


Figure 2
A view of the N—H···O hydrogen-bonded (dashed lines) chain in (I).

Experimental

Compound (I) was prepared by the reaction of 5,5-dimethylcyclohexane-1,3-dione (4 mmol, 0.560 g) with 3,4-dichlorobenzaldehyde (2 mmol, 0.350 g) and NH_4OAc (3 mmol, 0.231 g) under solvent-free conditions. Single crystals suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution.

Crystal data

$C_{23}H_{25}Cl_2NO_2$
M_r = 418.34
Orthorhombic, $Pna2_1$
a = 14.155 (3) Å
b = 14.352 (3) Å
c = 10.704 (3) Å
V = 2174.6 (9) Å^3
Z = 4
D_x = 1.278 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1556 reflections
θ = 2.4–18.8°
μ = 0.32 mm^{-1}
T = 298 (2) K
Block, colourless
0.43 × 0.40 × 0.38 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.876, T_{max} = 0.889
10745 measured reflections
3745 independent reflections
1776 reflections with $I > 2\sigma(I)$
R_{int} = 0.081
θ_{max} = 25.0°
$h = -12 \rightarrow 16$
$k = -17 \rightarrow 14$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.069
$wR(F^2)$ = 0.186
S = 1.01
3745 reflections
253 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0795P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.74 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}
Absolute structure: Flack (1983), 1745 Friedel pairs
Flack parameter: 0.09 (14)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.86	1.85	2.708 (6)	174

Symmetry code: (i) $x+\frac{1}{2}, -y+\frac{3}{2}, z$.

H atoms were placed in geometrically idealized positions (N—H = 0.86 Å and C—H = 0.93–0.98 Å) and allowed to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C,*N*) or 1.5U_{eq}(methyl C).

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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