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

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Key indicators

Single-crystal X-ray study
T = 193 K
Mean σ(C–C) = 0.005 Å
R factor = 0.077
wR factor = 0.175
Data-to-parameter ratio = 14.7

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

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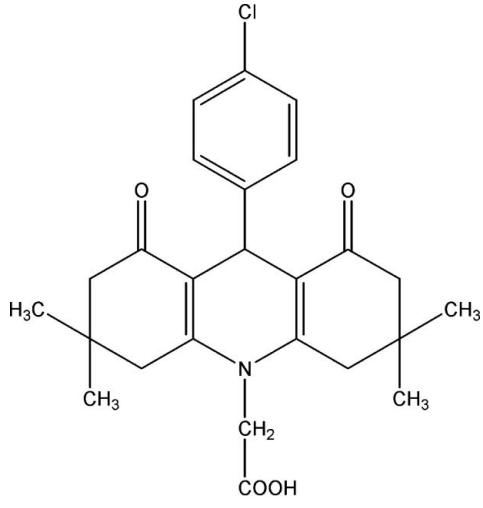
The title compound, $C_{25}H_{28}ClNO_4$, was synthesized by the reaction of dimedone with 4-chlorobenzaldehyde and glycine in glycol under microwave irradition. X-ray analysis reveals that the dihydropyridine ring is in a boat conformation and both cyclohexenone rings adopt envelope conformations.

Comment

Many natural and synthetic compounds containing the acridine skeleton display interesting biological and physical activities (Wysocka-Skrzela & Ledochowski, 1976). Acridinedione, for example, has been identified as an antimalarial and antitumour agent (Khurana *et al.*, 1990). Decahydroacridine-1,8-dione derivatives have been reported to have high fluorescence efficiency and can be used as fluorescent molecular probes for monitoring polymerization processes (Popielarz *et al.*, 1997). Furthermore, acridinedione dyes have already been reported as a class of laser dyes operating in the blue–green region (Prabahar *et al.*, 1991). By virtue of their bichromophoric structure, incorporating both a heterocyclic N atom and carbonyl groups, these dyes have been revealed to act not only as electron donors but also as electron acceptors (Timpe *et al.*, 1991). Previously, we have reported the crystal structures of acridindione deratives (Tu *et al.*, 2004). In this paper, we report the crystal structure of the title compound, (I).



(I)

The dihydropyridine ring in (I) has a boat conformation, with atoms C3 and N1 deviating from the C1/C2/C4/C5 mean plane by 0.396 (5) and 0.187 (5) Å, respectively (Fig. 1). Both cyclohexenone rings adopt envelope conformations. The

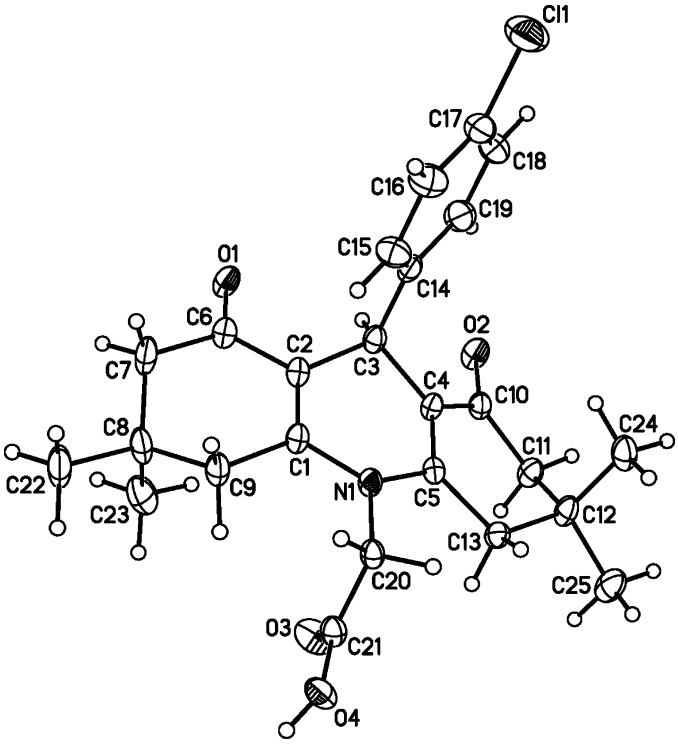


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

dihedral angle between the C14–C19 benzene ring and the C1/C2/C4/C5 plane is 80.8 (1)°. The carboxymethyl substituent at atom N1 is planar, and it is oriented orthogonal to the C1/C2/C4/C5 plane [dihedral angle 90.0 (1)°].

The crystal packing, shown in Fig. 2, reveals that intermolecular O—H···O and C—H···O hydrogen bonds (Table 1) form a three-dimensional network.

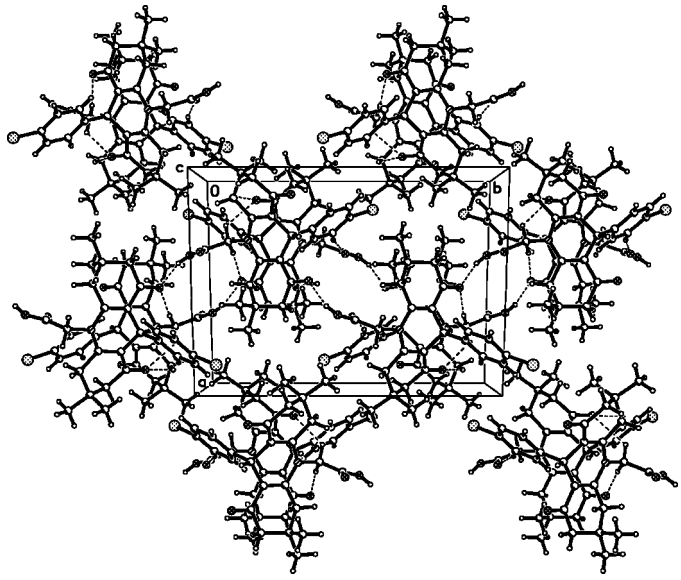


Figure 2
A molecular packing diagram for (I), projected along the *c* axis. Dashed lines indicate hydrogen bonds.

Experimental

Compound (I) was prepared by the reaction of 4-chlorobenzaldehyde (1 mmol) with glycine (1.5 mmol) and dimedone (2 mmol) in glycol (1 ml) under microwave irradiation (yield 86%; m.p. 563–564 K). Single crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution. Spectroscopic analysis: IR (KBr, ν, cm^{-1}): 3421 (OH), 1738 (CO), 1643 (CO); ^{1}H NMR (DMSO-d_6, δ, p.p.m.): 0.89 (6H, *s*, 2CH$_3$), 1.00 (6H, *s*, 2CH$_3$), 2.05–2.80 (8H, *m*, 4CH$_2$), 4.62 (2H, *s*, CH$_2$), 4.98 (H, *s*, CH), 7.09–7.38 (4H, *m*, ArH), 13.36 (1H, *s*, COOH).

Crystal data

$C_{25}H_{28}ClNO_4$	D_x = 1.277 Mg m^{-3}
M_r = 441.93	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 5995 reflections
a = 12.655 (3) Å	θ = 3.1–25.3°
b = 16.582 (3) Å	μ = 0.20 mm^{-1}
c = 11.500 (3) Å	T = 193 (2) K
β = 107.708 (6)°	Block, light yellow
V = 2298.9 (9) Å^3	0.25 × 0.18 × 0.15 mm
Z = 4	

Data collection

Rigaku MERCURY CCD area-detector diffractometer	4190 independent reflections
ω scans	2642 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (Jacobson, 1998)	R_{int} = 0.060
T_{min} = 0.952, T_{max} = 0.971	θ_{max} = 25.4°
11302 measured reflections	$h = -14 \rightarrow 15$
	$k = -19 \rightarrow 19$
	$l = -12 \rightarrow 13$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0583P)^2 + 0.99P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.077	
$wR(F^2)$ = 0.175	
S = 1.12	$(\Delta/\sigma)_{max}$ = 0.001
4190 reflections	$\Delta\rho_{max}$ = 0.20 e Å^{-3}
286 parameters	$\Delta\rho_{min}$ = −0.21 e Å^{-3}
H-atom parameters constrained	

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4···O1^{i}	0.84	1.75	2.565 (4)	163
C13—H13B···O2^{ii}	0.99	2.57	3.339 (4)	134
C16—H16···O3^{ii}	0.95	2.41	3.339 (5)	166
C20—H20A···O1^{ii}	0.99	2.41	3.397 (4)	175
C20—H20B···O2^{ii}	0.99	2.52	3.229 (4)	128

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

H atoms were placed in idealized positions, with O—H = 0.84 Å and C—H = 0.95–1.00 Å, and allowed to ride on their parent atoms, with U_{iso}(H) = 1.5U_{eq}(carrier) for methyl and carboxyl H atoms and 1.2 U_{eq}(C) for others.

Data collection: *CrystalClear* (Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2003); 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: *SHELXL97*.

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