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5-(2-Chlorophenyl)-7,7-dimethyl-10-(4-methylphenyl)-7,8-dihydro-5*H*-indeno[1,2-*b*]quinoline-9,11(6*H*,10*H*)-dione dimethylformamide solvate

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Key indicators

Single-crystal X-ray study
T = 153 K
Mean σ(C–C) = 0.004 Å
R factor = 0.062
wR factor = 0.158
Data-to-parameter ratio = 13.8

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

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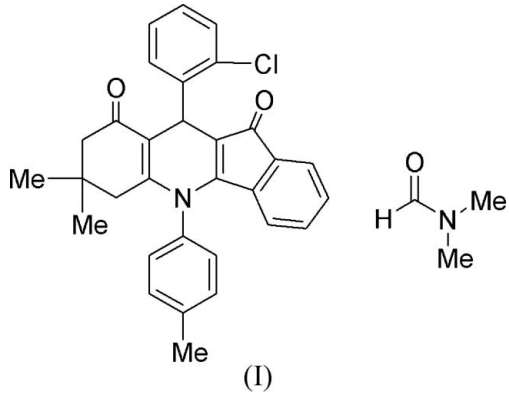
The title compound, $C_{31}H_{26}ClNO_2\cdot C_3H_7NO$, was synthesized by the reaction of 2-chlorobenzaldehyde, 5,5-dimethyl-3-(4-methylanilino)cyclohex-2-enone and 1,3-indenedione in an ionic liquid medium. The 1,4-dihydropyridine ring adopts a boat conformation, while the cyclohexenone ring adopts an envelope conformation. In the crystal structure, C—H···O hydrogen bonds link the indeno[1,2-*b*]quinoline molecules and the dimethylformamide solvent molecules to form a three-dimensional network.

Comment

It is known that many quinoline-containing compounds exhibit a wide spectrum of pharmacological activities, such as antiplasmodial (Beagley *et al.*, 2003), antibacterial (Fokialakis *et al.*, 2002), antiproliferative (Fossa *et al.*, 2002), antimalarial (Ryckebusch *et al.*, 2003) and anticancer activities (Morgan *et al.*, 2002). In order to establish the conformational aspects of these potentially biologically active compounds, we report here the structure of the title quinoline derivative, (I).



(I)

The 1,4-dihydropyridine ring of (I) adopts a boat conformation (Fig. 1). Atoms C3 and N1 deviate from the basal plane defined by the atoms C1/C2/C4/C5 by 0.242 (4) and 0.090 (1) Å, respectively. Similar distortions were observed in 2-amino-4-(2-chlorophenyl)-7,7-dimethyl-1-(4-methylphenyl)-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carbonitrile (Jiang *et al.*, 2006), 7,7-dimethyl-2-(4-bromophenyl)-4-phenyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline (Shi *et al.*, 2002) and 3,3,6,6-tetramethyl-9-(4-chlorophenyl)-10-(4-methylphenyl)-1,2,3,4,5,6,7,8,9,10-decahydroacridine-1,8-dione (Wang *et al.*, 2003). The cyclohexenone ring, C1/C2/C6–C9, adopts an envelope conformation; atom C8 deviates from the mean plane of the remaining atoms by 0.623 (4) Å. A similar conformation has been found in the structure of 7,7-dimethyl-

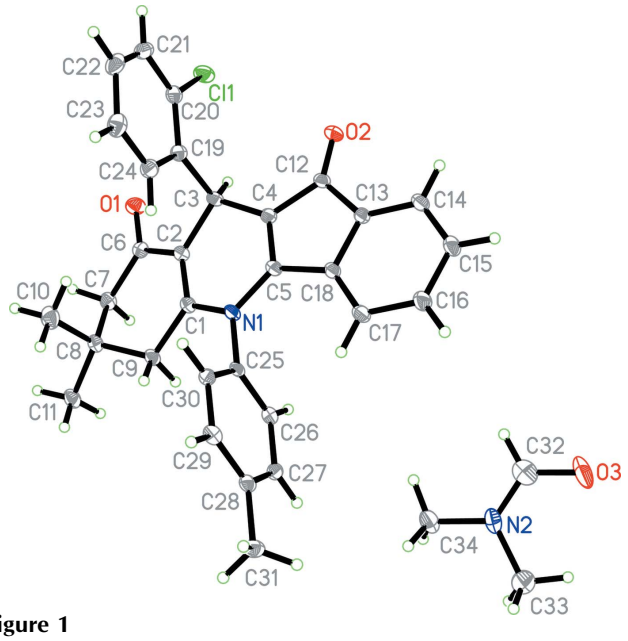


Figure 1
The asymmetric unit of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

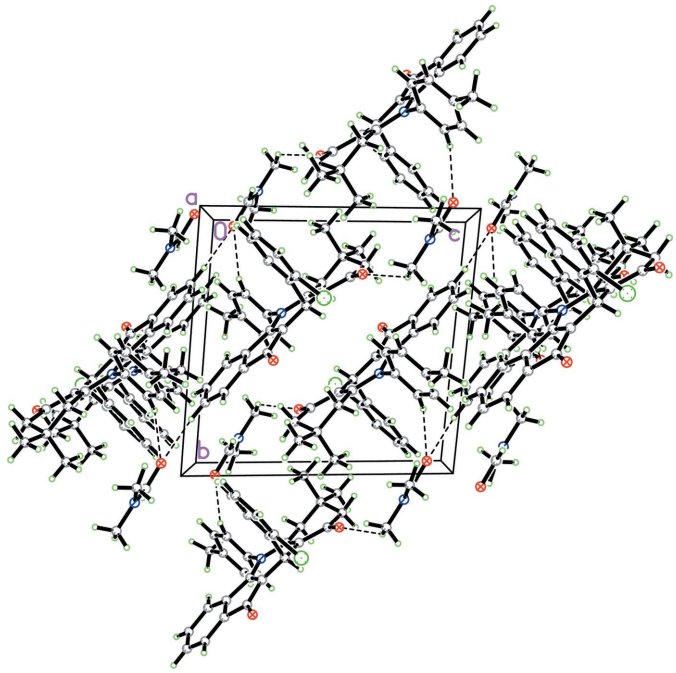


Figure 2
The packing of (I), with hydrogen bonds drawn as dashed lines.

2-amino-3-cyano-4-(3,4-methylenedioxyphenyl)-5-oxo-5,-6,7,8-tetrahydro-4*H*-benzo[*b*]pyran (Wang *et al.*, 2002). The benzene rings subtend a dihedral angle of 10.7 (1)° with one another.

Molecules of (I) are linked together and to molecules of the solvent by C—H···O hydrogen bonds, forming a three-dimensional network (Table 2, Fig. 2).

Experimental

The title compound, (I), was prepared by the reaction of 2-chlorobenzaldehyde (1 mmol, 0.14 g), 5,5-dimethyl-3-(4-methylanilino)cyclohex-2-enone (1 mmol, 0.23 g) and 1,3-indenedione (1 mmol, 0.15 g) in an ionic liquid, [Bmim$^+$][BF$_4^-$] (Bmin$^+$ is the 1-butyl-3-methyl imidazolium cation) (5.0 ml) at 363 K for 6 h (yield 98%; m.p. 536–538 K). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of a dimethylformamide solution. Elemental analysis, calculated: C 73.83, H 6.01, N 5.06%; found: C 73.89, H 5.90, N 5.01%.

Crystal data

$C_{31}H_{26}ClNO_2\cdot C_3H_7NO$
M_r = 553.07
Triclinic, $P\overline{1}$
a = 10.872 (2) Å
b = 11.655 (2) Å
c = 11.689 (2) Å
α = 92.100 (4)°
β = 99.730 (4)°
γ = 106.147 (4)°
V = 1396.8 (4) Å^3
Z = 2
D_x = 1.315 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.18 mm^{-1}
T = 153 (2) K
Block, red
0.68 × 0.23 × 0.15 mm

Data collection

Rigaku Mercury diffractometer
ω scans
Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.688, T_{max} = 0.974
13755 measured reflections
5071 independent reflections
4222 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 25.4°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.158
S = 1.08
5071 reflections
367 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0722P)^2 + 1.2467P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.91 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C5	1.371 (3)	C2—C3	1.517 (3)
N1—C1	1.414 (3)	C3—C4	1.505 (4)
C1—C2	1.354 (4)	C4—C5	1.358 (4)
C5—N1—C1	118.0 (2)	C4—C3—C2	107.5 (2)
C2—C1—N1	121.1 (2)	C5—C4—C3	123.4 (2)
C1—C2—C3	123.8 (2)	C4—C5—N1	122.5 (2)
C5—N1—C1—C2	−9.5 (4)	C2—C3—C4—C5	−20.0 (3)
N1—C1—C2—C3	−4.6 (4)	C3—C4—C5—N1	8.8 (4)
C1—C2—C3—C4	17.9 (3)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C34—H34A···O1^{i}	0.98	2.51	3.259 (4)	133
C30—H30···O3^{ii}	0.95	2.55	3.444 (4)	157
C15—H15···O3^{iii}	0.95	2.44	3.371 (4)	167

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $x, y-1, z$; (iii) $-x+1, -y+2, -z$.

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.95 Å and U_{iso}(H) = 1.2U_{eq}(C) for aromatic H, C—H = 1.00 Å and U_{iso}(H) = 1.2U_{eq}(C) for CH, C—H = 0.99 Å, U_{iso}(H) = 1.2U_{eq}(C) for CH_2, and C—H = 0.98 Å and U_{iso}(H) = 1.5U_{eq}(C) for CH_3 atoms.

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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