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7,8-Dihydro-7,7-dimethyl-5-*p*-tolyl-10-(3,4,5-trimethoxyphenyl)-5*H*-indeno[1,2-*b*]quinoline-9,11(6*H*,10*H*)-dione hemihydrate

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The title compound, $C_{34}H_{33}NO_5\cdot 0.5H_2O$, has been synthesized by reacting 3,4,5-trimethoxybenzaldehyde, 5,5-dimethyl-3-(*p*-tolylamino)cyclohex-2-enone and indan-1,3-dione, under microwave irradiation, in a mixture of glycol and acetic acid as solvent. The central N-substituted ring adopts a boat conformation. The uncoordinated water molecule is disordered over two positions, both lying on a twofold symmetry axis.

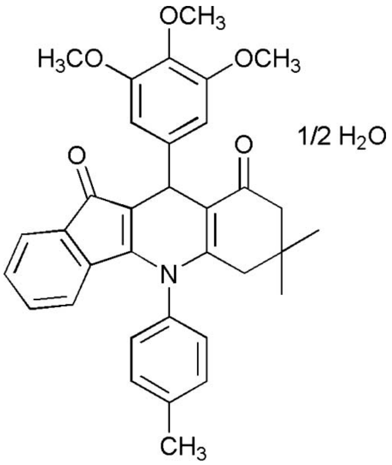
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.006 Å
Disorder in main residue
R factor = 0.073
wR factor = 0.239
Data-to-parameter ratio = 14.2

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

Comment

1,4-Dihydropyridines (1,4-DHPs) are well known because of their pharmacological profiles as calcium channel modulators (Stout & Meyers, 1982). With a 1,4-DHP parent nucleus, indenoquinoline belongs to a class of compounds which are special not only because of their interesting chemical and physical properties, but also due to their immense utility in the pharmaceutical industry. The discovery of indenoquinolines as new potent cytotoxic and antitumor agents has attracted the attention of organic chemists (Yamato *et al.*, 1989; Deady *et al.*, 1999, 2000; Chen *et al.*, 2002). It is well established that chemical modifications of the indenoquinoline skeleton may bring remarkable changes in biological activity (Deady *et al.*, 1999). In this paper, we report the crystal structure of such a compound, (I).



(I)

In (I), the central dihydropyridine ring adopts a boat conformation, with atoms N1 and C10 deviating from the C1/C9/C11/C16 mean plane by 0.0974 (3) and 0.2571 (3) Å, respectively (Fig. 1). The cyclohexene ring adopts an envelope conformation, with atom C14 deviating from the C11/C12/C13/C15/C16 mean plane by 0.668 (3) Å. The dihedral angle

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between the C1/C9/C11/C16 plane and the C24–C29 benzene ring is 83.13 (13)°.

Experimental

Compound (I) was prepared by reacting 3,4,5-trimethoxybenzaldehyde (1 mmol), 5,5-dimethyl-3-(*p*-tolylamino)cyclohex-2-enone (1 mmol) and indan-1,3-dione (1 mmol) in a mixture of glycol (0.5 ml) and acetic acid (1.0 ml), under microwave irradiation for 7 min at 200 W power and 393 K (microwave oven EmrysTM Creator from Personal Chemistry, Uppsala, Sweden). Upon completion, monitored by thin-layer chromatography, the reaction mixture was cooled to 298 K and then poured into cold water. The solid product was filtered off, washed with water and EtOH (95%), and subsequently dried and recrystallized from EtOH (95%), to give pure (I). Single crystals suitable for X-ray diffraction were obtained by slow evaporation of a 95% aqueous ethanol solution (yield 91%; m.p. 471–473 K). IR (ν, cm^{-1}): 1687 and 1644 (C=O).

Crystal data

$C_{34}H_{33}NO_5\cdot0.5H_2O$
$M_r = 544.62$
Monoclinic, $C2/c$
$a = 25.094\ (6)$ Å
$b = 12.755\ (3)$ Å
$c = 19.891\ (5)$ Å
$\beta = 103.926\ (4)°$
$V = 6179\ (3)$ Å^3
$Z = 8$
$D_x = 1.171$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.08$ mm^{-1}
$T = 298\ (2)$ K
Block, red
$0.50 \times 0.46 \times 0.26$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.962$, $T_{\max} = 0.980$
15711 measured reflections
5381 independent reflections
2112 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.097$
$\theta_{\max} = 25.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.073$
$wR(F^2) = 0.239$
$S = 1.00$
5381 reflections
378 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1027P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.68$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0011 (3)

The single crystal used was a poorly diffracting sample [$I/\sigma(I)$ = 4.59 for 5381 independent reflections], giving a poor $R_{\rm int}$ index and a structure of limited precision. The water molecule is located on a twofold axis and disordered over two sites, O6 and O6′, for which occupation factors were refined and converged to 0.725 (10) and 0.275 (10), respectively. Atoms O6 and O6′ were constrained to have identical U^{ij} parameters. A methyl group belonging to a methoxy group is also disordered over two positions, C31 and C31′, with occupation factors 0.56 (6) and 0.44 (6), respectively. For *p*-tolyl and methoxy groups, atoms closer than 1.7 Å were restrained within an s.u. value of 0.04 Å^2 to have the same U^{ij} components. For the main residue, all H atoms were positioned geometrically and treated as riding, with C—H distances constrained to 0.93 (aromatic CH), 0.96 (methyl CH_3), 0.97 (methylene CH_2) or 0.98 Å (methine CH), and $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C) for methyl H atoms and 1.2$U_{\rm eq}$(C) for other H atoms. Atoms H1 and H2, bonded to O6 and O6′, were also placed in sensible calculated positions and refined with O—H bond lengths constrained to 0.85 Å and with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(O).

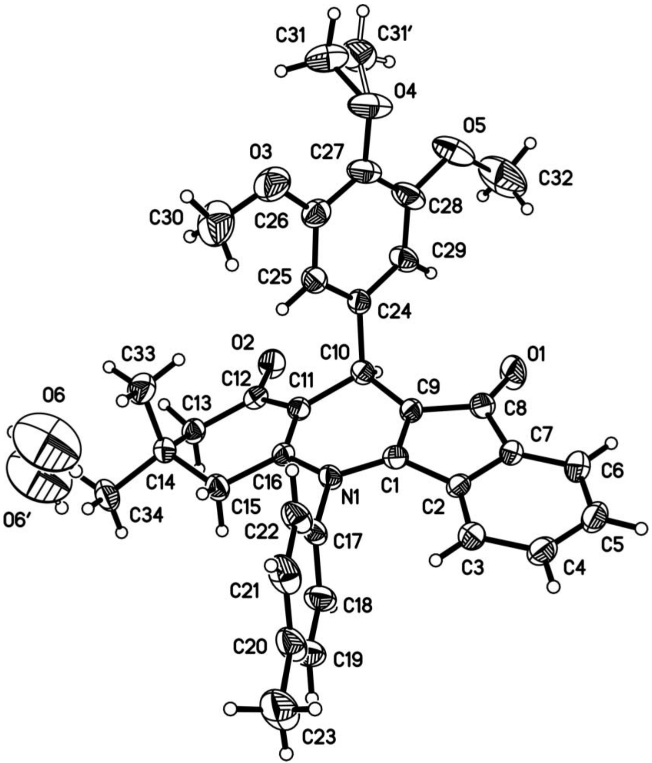

Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids for non-H atoms. Atoms C31 and C31′ correspond to a single methyl group disordered over two positions. O6 and O6′ are disordered alternative positions for the water molecule.

Data collection: *SMART* (Bruker, 1999); 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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