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9-[4-(1,2,3,4,5,6,7,8,9,10-Decahydro-3,3,6,6-tetramethyl-1,8-dioxoacridin-9-yl)phenyl]-1,2,3,4,5,6,7,8,9,10-decahydro-3,3,6,6-tetramethylacridine-1,8-dione dihydrate

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.059
wR factor = 0.160
Data-to-parameter ratio = 14.9

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

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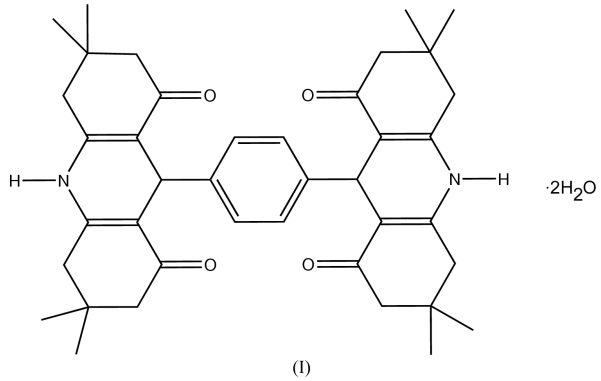
The title compound, $C_{40}H_{48}N_2O_4\cdot 2H_2O$, was synthesized by the reaction of dimedone with terephthalaldehyde and NH_4OAc in glycol. X-ray analysis reveals that the dihydropyridine and cyclohexenone rings adopt half-boat conformations. The screw-related molecules form N—H···N and O—H···O hydrogen-bonded chains along the c axis.

Comment

Acridine derivatives containing the 1,4-dihydropyridine unit belong to a special class of compounds, not only because of their interesting chemical and physical properties but also owing to their immense utility in the pharmaceutical and dye industries; they are also well-known therapeutic agents (Wysocka-Skrzela & Ledochowski, 1976; Nasim & Brychey, 1979; Thull & Testa, 1994; Reil *et al.*, 1994; Mandi *et al.*, 1994). Recently, we have reported the synthesis of *N*-hydroxylacridine derivatives (Tu *et al.*, 2004), but so far, there are few reports on the syntheses, structures and properties of compounds that contain acridine ring systems. We report here the crystal structure of the title compound, (I).



(I)

The molecular structure of (I) is shown in Fig. 1. The molecule is plane-related symmetrical, with a plane perpendicular to the benzene ring, containing the central benzene ring, which divides the molecule equally. The dihydropyridine and cyclohexenone rings adopt half-boat conformations, with atoms C10, C6 and C14 deviating from the mean planes through the remaining atoms in the corresponding rings by 0.300 (4), 0.620 (6) and 0.644 (5) Å, respectively. The dihedral angle between the N1/C4/C9/C11/C16 plane and the benzene ring attached at atom C10 is 89.87 (9)°. The water molecules link screw-related molecules through N—H···N and O—H···O hydrogen bonding to form chains along the c axis (Table 2 and Fig. 2).

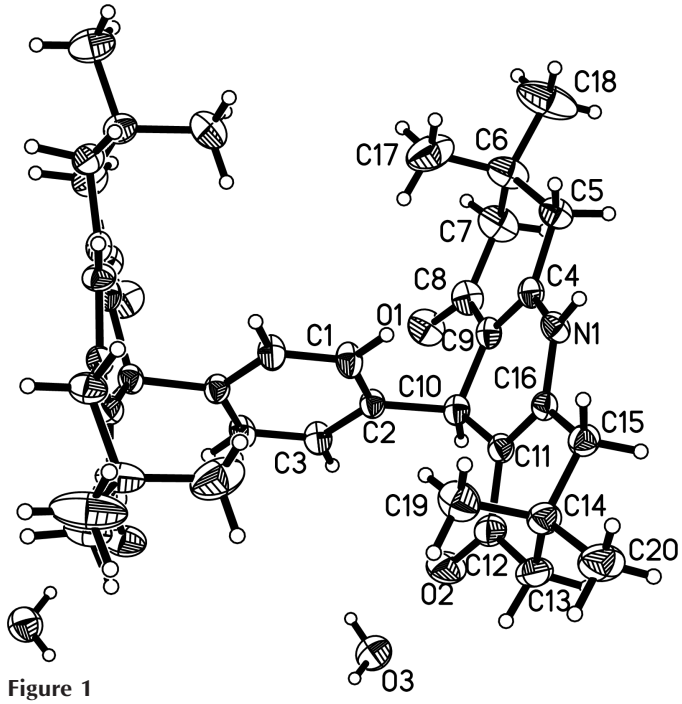


Figure 1
The structure of (I), showing 40% probability displacement ellipsoids and the atom-numbering scheme for contents of the asymmetric unit. Unlabelled atoms are related to labelled atoms by $3/2 - x$, $1/2 - y$, z

Experimental

The title compound was prepared by the reaction of dimedone (1.09 g, 7.78 mmol) with terephthalaldehyde (0.26 g, 1.94 mmol) and NH_4OAc (0.3 g, 3.90 mmol) in glycol (2 ml) under microwave irradiation. Single crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of a mixed ethanol/water (95:5 *v*/*v*) solution.

Crystal data

$C_{40}H_{48}N_2O_4{\cdot}2H_2O$
$M_r = 656.84$
Tetragonal, $P4_2/n$
$a = 16.334$ (2) Å
$c = 13.975$ (2) Å
$V = 3728.5$ (8) Å^3
$Z = 4$
$D_x = 1.170$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 33 reflections
$\theta = 3.2$–$11.0°$
$\mu = 0.08$ mm^{-1}
$T = 294$ (2) K
Prism, colourless
0.40 × 0.40 × 0.36 mm

Data collection

Siemens *P*4 diffractometer
ω scans
4104 measured reflections
3296 independent reflections
1412 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 25.0°$
$h = 0 \rightarrow 19$
$k = 0 \rightarrow 19$
$l = -1 \rightarrow 16$
3 standard reflections
every 97 reflections
intensity decay: 2.2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.160$
$S = 0.81$
3296 reflections
221 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0816P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.41$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}

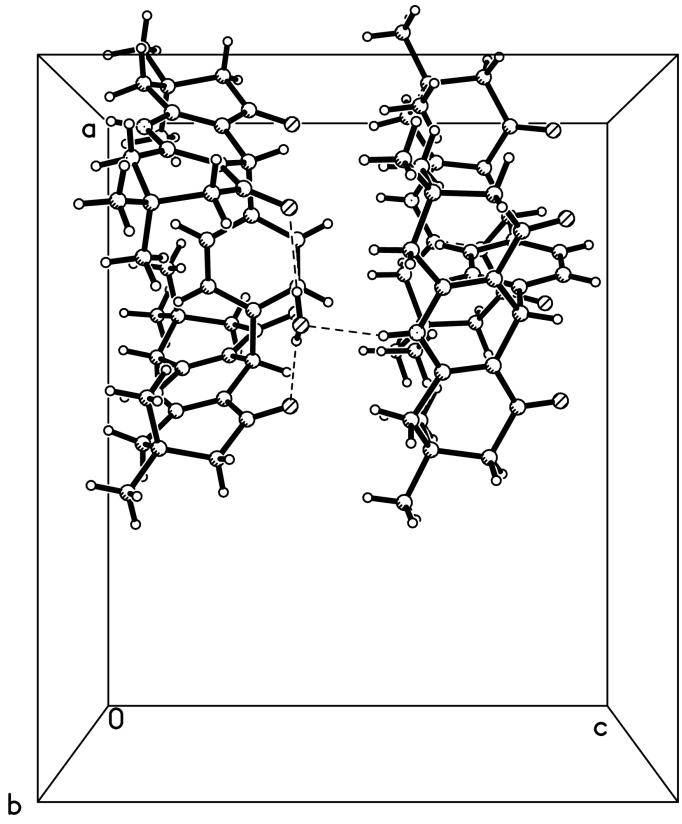


Figure 2
The packing of the molecules of (I), viewed down the *b* axis. Intermolecular hydrogen bonds are shown as dashed lines.

Table 1
Selected geometric parameters (Å, °).

O1—C8	1.235 (4)	C1—C1^{i}	1.366 (6)
O2—C12	1.237 (4)	C3—C3^{i}	1.378 (6)
N1—C16	1.370 (4)	C4—C9	1.356 (4)
N1—C4	1.382 (4)	C11—C16	1.361 (4)
C9—C4—C5	124.8 (3)	C11—C16—C15	124.0 (3)
N1—C4—C5	115.9 (3)	N1—C16—C15	116.0 (3)

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

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O3^{ii}	0.86	2.16	2.959 (3)	155
O3—H3A···O1^{iii}	0.85	2.19	2.991 (4)	157
O3—H3B···O2^{iv}	0.85	2.20	3.008 (4)	159

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

After location, H atoms of the water molecule were allowed to ride on the O atom, with $U_{iso}(H) = 1.5U_{eq}(O)$. The remaining H atoms were placed in calculated positions (C—H = 0.93–0.98 Å and N—H = 0.86 Å) and included in the final cycles of refinement using a riding model, with $U_{iso}(H) = 1.2U_{eq}$(parent atom). The U_{ij} components of atom C17 were approximated to isotropic behaviour. The highest peak in the final difference map is located 1.76 Å from atom H17*B*.

Owing to the poor diffraction quality of the crystal, the ratio of observed to unique reflections is low (43%).

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Sheldrick, 1997); program(s) used to solve structure: *SHELXTL*; program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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