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9-(2,6-Dihydroxyphenyl)-3,3,6,6-tetramethyl-*N*-(4-methylphenyl)-1,8-dioxo-1,2,3,4,5,6,7,8,9,10-decahydroacridine

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Key indicators

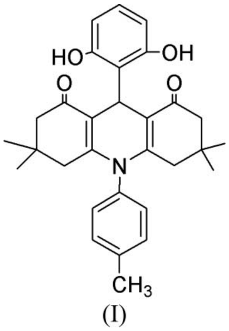
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.009 Å
R factor = 0.073
wR factor = 0.219
Data-to-parameter ratio = 14.0

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

The title compound, $C_{30}H_{33}NO_4$, was synthesized by the reaction of 2,6-dihydroxybenzaldehyde with *p*-toluidine and 5,5-dimethylcyclohexane-1,3-dione in glycol under microwave irradition. X-ray analysis reveals that the dihydropyridine ring is in a distorted boat conformation and that the cyclohexenone rings adopt enevelope conformations. In the crystal structure, molecules are linked by O—H···O hydrogen bonds around a centre of symmetry to form a dimer.

Comment

Acridine and its derivatives inhibit HIV-1 reverse transcriptase by intercalating the template-primer hybrid (Cellai *et al.*, 1994). Such compounds are well known as antimicrobials (Al-Ashmawi *et al.*, 1994) and antitumour agents (Wang *et al.*, 1993), and are used in the treatment of urinary incontinence (Ohnmacht, 1993). Recently, we reported the crystal structures of 10-cyclopropyl-9-(4-hydroxy-3-methoxyphenyl)-3,3,6,6-tetramethyl-1,2,3,4,5,6,7,8,9,10-decahydroacridine-1,8-dione (Tu *et al.*, 2004) and 9-(4-methoxyphenyl)-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione (Guo *et al.*, 2004). We now report the crystal structure of the title compound, (I).



(I)

The dihydropyridine ring in (I) adopts a distorted boat conformation. In this ring, atoms N1 and C7 deviate from the C1/C6/C8/C13 plane by 0.093 (8) and 0.200 (8) Å, respectively (Fig. 1). Both cyclohexenone rings adopt envelope conformations. The C18–C23 and C25–C30 benzene rings are nearly coplanar [dihedral angle = 8.4 (2)°] and form dihedral angles of 84.6 (2) and 87.5 (2)°, respectively, with the C1/C6/C8/C13 plane. An intramolecular O3—H3···N1 interaction is observed (Table 1). The crystal packing, shown in Fig. 2, reveals that the molecules exist as centrosymmetric O—H···O hydrogen-bonded dimers.

Experimental

Compound (I) was prepared by the reaction of 2,6-dihydroxybenzaldehyde (1 mmol) with *p*-toluidine (1 mmol) and 5,5-

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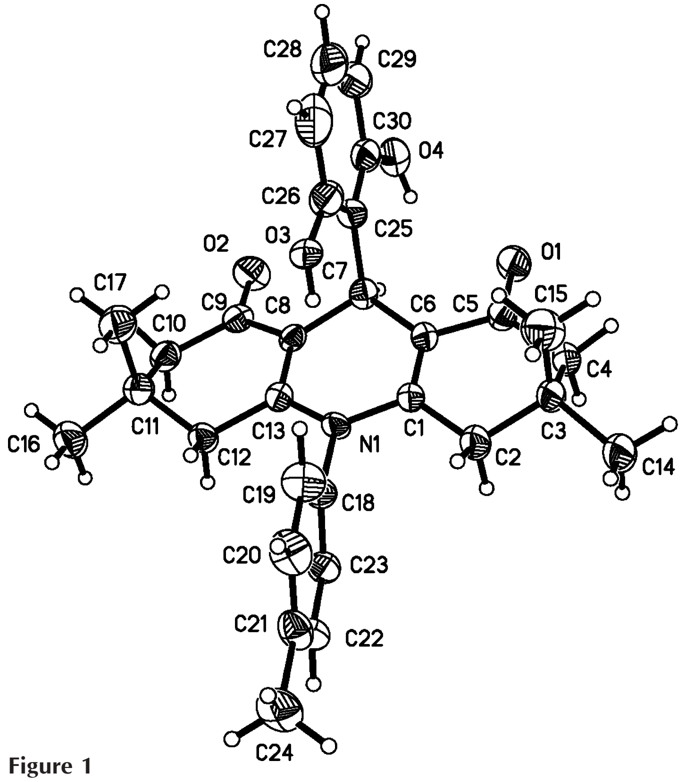

Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

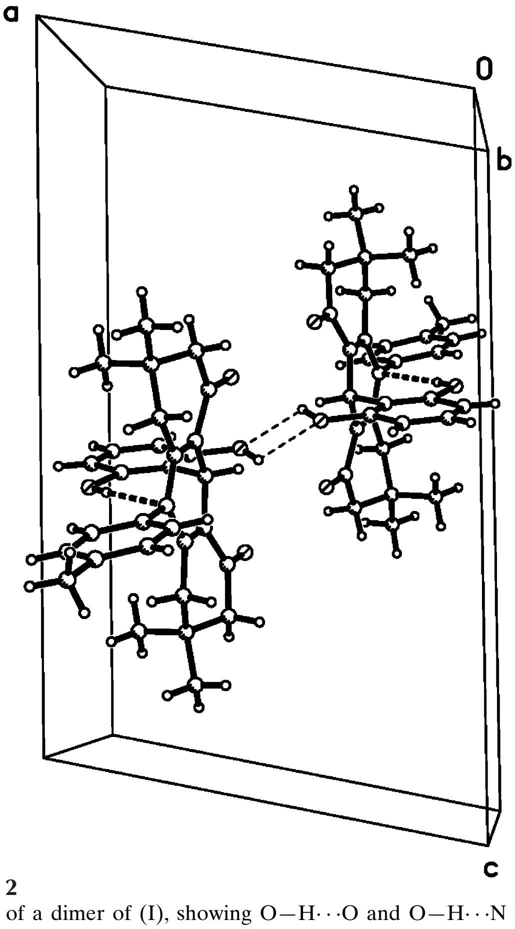

Figure 2
A view of a dimer of (I), showing O—H···O and O—H···N hydrogen bonds (dashed lines).

dimethylcyclohexane-1,3-dione (2 mmol) in glycol (1 ml) under microwave irradiation for 4 min (yield 91%; m.p. 531–532 K). Single crystals suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution.

Crystal data

$C_{30}H_{33}NO_4$
$M_r = 471.57$
Monoclinic, $P2_1/c$
$a = 11.995$ (4) Å
$b = 11.002$ (4) Å
$c = 19.965$ (7) Å
$\beta = 101.419$ (6)°
$V = 2582.5$ (15) Å^3
$Z = 4$

$D_x = 1.213$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1094 reflections
$\theta = 2.4$–19.3°
$\mu = 0.08$ mm^{-1}
$T = 298$ (2) K
Block, light yellow
0.38 × 0.17 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.970$, $T_{\max} = 0.988$
13000 measured reflections

4544 independent reflections
1460 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.118$
$\theta_{\max} = 25.0°$
$h = -14 \rightarrow 13$
$k = -13 \rightarrow 9$
$l = -23 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.073$
$wR(F^2) = 0.219$
$S = 1.01$
4544 reflections
324 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.072P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.31$ e Å^{-3}
$\Delta\rho_{\min} = -0.27$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0042 (10)

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···N1	0.82	2.44	3.233 (6)	164
O4—H4···O4$^{\rm i}$	0.82	2.30	2.587 (6)	101

Symmetry code: (i) $-x + 1, -y, -z$.

H atoms were placed in idealized positions and allowed to ride on their parent atoms, with O—H distances of 0.82 Å and C—H distances in the range 0.93–0.98 Å, and with $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(carrier) for methyl and hydroxy H atoms, and 1.2$U_{\rm eq}$(carrier) for other H atoms. Owing to the poor diffraction quality of the crystal, the higher angle reflections were very weak and only 32% of the reflections were found to be observed with $I > 2\sigma(I)$. This resulted in a high $R_{\rm int}$ value. The C6—O3 and C30—O4 distances were restrained to be 1.36 (1) Å. Owing to large displacement parameters, the U^{ij} components of atoms C27, C28 and C28 were restrained to isotropic behaviour.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; 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: *SHELXTL*.

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