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10-[2-(4-Hydroxyphenyl)ethyl]-3,3,6,6,9-pentamethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.059
wR factor = 0.180
Data-to-parameter ratio = 14.1

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

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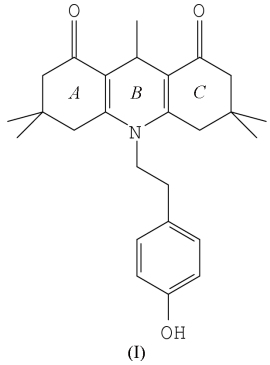
The central dihydropyridine ring in the title compound, $C_{26}H_{33}NO_3$, adopts a boat conformation, while the outer rings adopt sofa conformations. The packing is stabilized by O—H···O hydrogen bonds.

Comment

Acridine derivatives exhibit a wide spectrum of biological activities, such as antibacterial (Acheson, 1956), mutagenic, antitumour (Talacki *et al.*, 1974) and anti-amoebic (Prasad Krishna *et al.*, 1984). Acridines bind to DNA by intercalation (Lerman, 1961; Karle *et al.*, 1980; Nandi *et al.*, 1990; Reddy *et al.*, 1979; Sakore *et al.*, 1979). The use of decahydroacridine-1,8-diones as photo-sensitizers is also well known (Timpe *et al.*, 1993). Acridinediones act as laser dyes whose laser activity has been studied (Murugan *et al.*, 1998). In acridine-1,8-diones, the electron delocalization is along a stretch of nine non-H atoms, facilitating fluorescence and laser activity (Selladurai *et al.*, 1990). The derivatives of acridine are buckled and this buckling was a factor in considering their biological properties (Glusker *et al.*, 1972). The effectiveness of lasing can be controlled by the substituents at the 9- and 10-positions of the acridine chromophore. The present study of the title compound, (I), is a part of a series of investigations on the crystal structures of acridinedione derivatives (Jeyakanthan *et al.*, 2000).



(I)

The bond distances of C=O and C—OH are in good agreement with values observed in related structures (Allen *et al.*, 1987; Ganesh *et al.*, 1998; Jeyakanthan *et al.*, 2000). The bond lengths in the dihydropyridine ring range from 1.352 (3) to 1.501 (3) Å and show the alternating single- and double-bond character, as observed in related acridine structures (Selladurai *et al.*, 1990; Sivaraman *et al.*, 1994). The dihedral angle between the two outer rings, *A* and *C*, is 13.05 (8)°, demonstrating the buckling of the acridine nucleus. The acridine moiety is folded about the line passing through atoms C2 and N. The dihedral angle between the two halves of ring *B*, *i.e.* C2–C8/N and C2/C17–C22/N, is 15.21 (5)°.

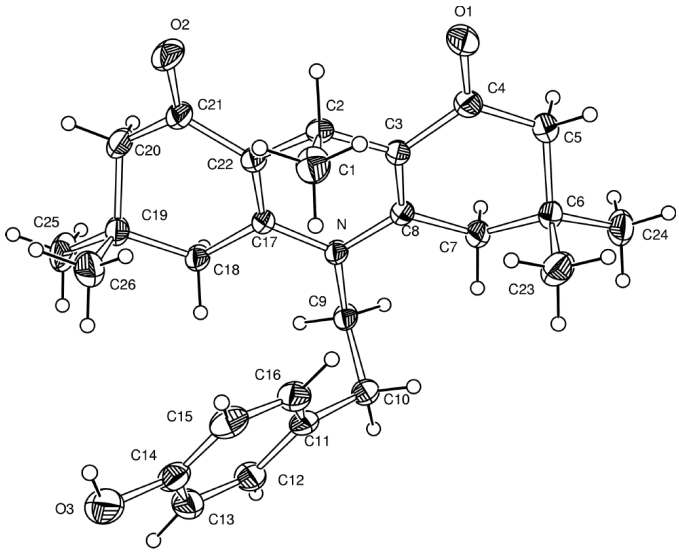


Figure 1
View of (I), shown with 30% probability displacement ellipsoids.

The sum of the angles around N is 359.6 (2)°, a clear indication of sp^2 hybridization. The bond lengths involving atom N conform to standard Csp^2—Nsp^2 bonds. The puckering amplitude of the central pyridine ring is small, owing to the π-conjugation along C3—C8—N—C17—C22, as indicated by the distances C3—C8, C8—N, N—C17 and C17—C22 (Table 1). The planar phenyl ring is orthogonal to the dihydropyridine ring, the dihedral angle between them being 39.85 (8)°.

The deviations of atoms O1 and O2 from the mean planes through rings *C* and *A* are 0.0585 (2) and −0.1894 (2) Å, respectively. The C4—C5—C6—C23 and C4—C5—C6—C24 torsion angles show that methyl atoms C23 and C24 are axial and equatorial, respectively, to ring C. The C21—C20—C19—C26 and C21—C20—C19—C25 torsion angles show that methyl atoms C26 and C25 are axial and equatorial, respectively, to ring *A*.

The total puckering amplitudes (Cremer & Pople, 1975) of rings *A*, *B* and *C* give a quantitative evaluation of puckering and asymmetry parameters. The asymmetry parameters (Nardelli, 1995) are Q_T = 0.5066 (5) Å and ΔC_2(C17–C22) = 0.0365 (1)°, *i.e.* a sofa conformation for ring *A*; Q_T = 0.2746 (3) Å, ΔC_2(C2–C22) = 0.0590 (4)° and ΔC_S(C2) = 0.0216 (2)° for ring *B*, *i.e.* a boat conformation; Q_T = 0.4707 (9) Å and ΔC_S(C3) = 0.0233 (4)° for ring *C*, *i.e.* a sofa conformation. In addition to van der Waals interactions, the packing in the crystal is stabilized by O—H···O hydrogen bonds (Table 2).

Experimental

A solution of 2,2′-ethylenebis(dimedone) (1.5 g, 0.0049 mol) in acetic acid was refluxed for 13 h. The reaction mixture was poured on to ice, and the resulting brown solid was filtered off, dried and recrystallized from a mixture of chloroform and methanol (1:1) to afford the title compound (1.2 g, 60.3% yield)

Crystal data

$C_{26}H_{33}NO_3$	D_x = 1.214 Mg m^{-3}
M_r = 407.53	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 25 reflections
a = 11.350 (2) Å	θ = 2.1–25.0°
b = 15.5152 (15) Å	μ = 0.08 mm^{-1}
c = 13.084 (2) Å	T = 293 (2) K
β = 104.677 (14)°	Block, orange
V = 2228.9 (6) Å^3	0.30 × 0.30 × 0.25 mm
Z = 4	

Data collection

Enraf–Nonius CAD-4 diffractometer	θ_{max} = 25.0°
ω scans	$h = -13 \rightarrow 13$
Absorption correction: none	$k = 0 \rightarrow 18$
4093 measured reflections	$l = 0 \rightarrow 15$
3914 independent reflections	3 standard reflections
2417 reflections with $I > 2\sigma(I)$	frequency: 120 min
R_{int} = 0.043	intensity decay: <2%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1125P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.059	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.180	$\Delta\rho_{max}$ = 0.24 e Å^{-3}
S = 1.01	$\Delta\rho_{min}$ = −0.30 e Å^{-3}
3914 reflections	Extinction correction: *SHELXL*97
278 parameters	Extinction coefficient: 0.011 (2)
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

N—C8	1.393 (3)	O2—C21	1.225 (3)
N—C17	1.399 (3)	O3—C14	1.372 (3)
N—C9	1.478 (3)	C3—C8	1.352 (3)
O1—C4	1.237 (3)	C17—C22	1.362 (3)
C8—N—C17	119.3 (2)	O1—C4—C5	120.6 (2)
C8—N—C9	119.2 (2)	C3—C8—N	120.8 (2)
C17—N—C9	121.1 (2)	N—C8—C7	118.5 (2)
O1—C4—C3	119.7 (2)	N—C9—C10	113.2 (2)
C4—C5—C6—C23	−70.7 (3)	C26—C19—C20—C21	63.9 (3)
C4—C5—C6—C24	168.4 (2)	C25—C19—C20—C21	−175.6 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O1^{i}	0.82	1.89	2.681 (3)	161

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

All H atoms were fixed geometrically and allowed to ride on their attached non-H atoms, with C—H = 0.96 Å. The torsion angles about C—CH_3 and C—OH bonds were refined with a rotating-group model.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 for Windows (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1995).

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