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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

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Key indicators

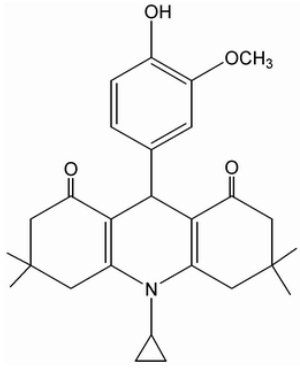
Single-crystal X-ray study
T = 193 K
Mean σ(C–C) = 0.003 Å
R factor = 0.061
wR factor = 0.139
Data-to-parameter ratio = 18.0

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

The title compound, $C_{27}H_{33}NO_4$, was synthesized by the reaction of dimedone with 3-methoxy-4-hydroxybenzaldehyde, cyclopropylamium chloride and NaOAc in glycol and water. X-ray analysis reveals that the dihydropyridine ring is in a distorted boat conformation. O—H···O hydrogen bonds involving the hydroxy and carbonyl O atoms link the screw-related molecules into zigzag chains along the b 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*-hydroxyacridine-1,8-dione derivatives (Tu, Miao *et al.*, 2004) and the crystal structure of 9-(4-hydroxy-3-methyoxyphenyl)-3,3,6,6,10-pentamethyl-1,2,3,4,5,6,7,8,9,10-decahydroacridine-1,8-dione (Tu, Zhang *et al.*, 2004). We now report the crystal structure of the title compound, (I).



(I)

The dihydropyridine ring in (I) is in a distorted boat conformation. In this ring, atoms N1 and C3 deviate from the C1/C2/C4/C5 plane by 0.211 (2) and 0.382 (3) Å, respectively (Fig. 1). Both cyclohexenone rings adopt envelope conformations. The dihedral angle between the C1/C2/C4/C5 plane and the benzene ring attached at atom C3 is 88.15 (5)°. The dihedral angle between the cyclopropyl and C1/C2/C4/C5 planes is 72.1 (1)°.

Screw-related molecules are linked *via* O—H···O hydrogen bonds (Table 2) between the hydroxy O4 and carbonyl O2 atoms, forming zigzag chains along the b axis (Fig. 2).

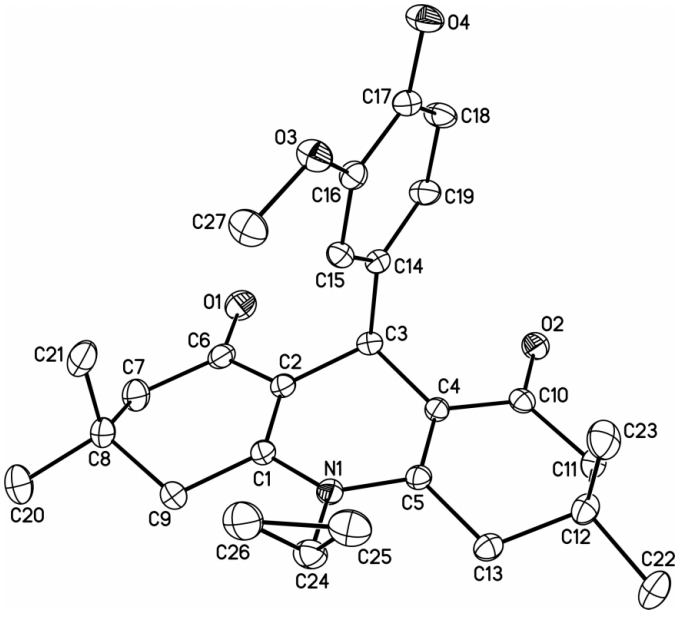


Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity.

Experimental

Compound (I) was prepared by the reaction of dimedone (4 mmol) with 4-hydroxy-3-methoxybenzaldehyde (2 mmol) and cyclopropylaminium chloride (3 mmol) and NaOAc (3 mmol) in a mixture of glycol (2 ml) and water (1 ml), under microwave irradiation (yield 85%, m.p. 547–548 K). Single crystals suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution.

Crystal data

$C_{27}H_{33}NO_4$
M_r = 435.54
Monoclinic, $P2_1/n$
a = 9.6559 (11) Å
b = 14.9830 (16) Å
c = 16.5393 (19) Å
β = 102.754 (2)°
V = 2333.8 (5) Å^3
Z = 4
D_x = 1.240 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7990 reflections
θ = 3.1–27.5°
μ = 0.08 mm^{-1}
T = 193 (2) K
Block, colourless
0.30 × 0.22 × 0.19 mm

Data collection

Rigaku Mercury diffractometer
ω scans
Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.976, T_{max} = 0.985
25 647 measured reflections
5340 independent reflections
4297 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 27.5°
$h = -12 \rightarrow 12$
$k = -19 \rightarrow 19$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.139
S = 1.13
5340 reflections
296 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0508P)^2 + 0.8174P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0030 (9)

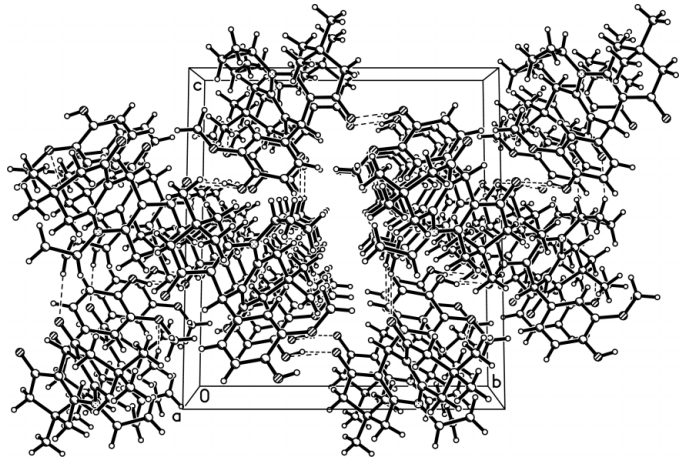


Figure 2
The molecular packing of (I), viewed along the *a* axis. Dashed lines indicate hydrogen bonds.

Table 1
Selected geometric parameters (Å, °).

O1—C6	1.228 (2)	N1—C5	1.396 (2)
O2—C10	1.240 (2)	N1—C1	1.401 (2)
O3—C16	1.374 (2)	N1—C24	1.451 (2)
O3—C27	1.421 (2)	C1—C2	1.349 (2)
O4—C17	1.366 (2)	C4—C5	1.357 (2)
C16—O3—C27	118.10 (14)	C21—C8—C20	108.79 (18)
C5—N1—C24	118.64 (14)	C1—C9—C8	112.71 (15)
C1—N1—C24	122.59 (15)	C11—C12—C22	110.10 (16)
C2—C1—C9	122.44 (16)	C11—C12—C23	110.54 (16)
N1—C1—C9	117.42 (15)	C22—C12—C23	109.61 (16)
C2—C3—C4	108.13 (13)	C11—C12—C13	108.22 (14)
C2—C3—C14	114.34 (14)	C22—C12—C13	108.27 (16)
C4—C3—C14	109.99 (13)	C23—C12—C13	110.06 (16)
C4—C5—C13	122.64 (15)	C5—C13—C12	112.58 (14)
N1—C5—C13	117.14 (14)	O3—C16—C15	125.72 (15)
C6—C7—C8	114.11 (16)	O3—C16—C17	114.12 (15)
C7—C8—C9	107.50 (16)	O4—C17—C18	118.82 (16)
C7—C8—C21	110.59 (17)	O4—C17—C16	122.12 (16)
C9—C8—C21	111.21 (17)	N1—C24—C26	118.32 (17)
C7—C8—C20	109.92 (18)	N1—C24—C25	117.21 (16)
C9—C8—C20	108.80 (17)	C26—C24—C25	59.28 (15)
C2—C3—C14—C15	41.7 (2)	C27—O3—C16—C15	−3.3 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4···O2^{i}	0.84	1.95	2.713 (2)	151
O4—H4···O3	0.84	2.24	2.687 (2)	114

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

H atoms were treated as riding, with an O—H distance of 0.84 Å and C—H distances of 0.95–1.00 Å, and with U_{iso}(H) = 1.2U_{eq}(C) and 1.5U(C_{methyl},O).

Data collection: *CrystalClear* (Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2000–2003); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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