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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

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Key indicators

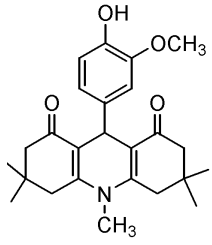
Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.003 Å
R factor = 0.042
wR factor = 0.108
Data-to-parameter ratio = 14.0

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

The title compound, $C_{25}H_{31}NO_4$, was synthesized by the reaction of dimedone with 4-hydroxy-3-methoxybenzaldehyde and CH_3NH_2HCl(NaOAc) in glycol. 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 glide-related molecules into zigzag 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 due to their immense utility in the pharmaceutical and dye industries, and they are 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 derivatives (Tu *et al.*, 2004). Now, we report here 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 C7 deviate from the C1/C6/C8/C13 plane by 0.196 (2) and 0.429 (3) Å, respectively (Fig. 1 and Table 1). Both cyclohexenone rings adopt half-chair conformations. The dihedral angle between the C1/C6/C8/C13 plane and the benzene ring attached at atom C7 is 69.1 (1)°. The methoxy group is slightly twisted out of the benzene ring plane, with a C20—O4—C18—C19 torsion angle of −12.0 (3)°.

Glide-related molecules are linked *via* an O—H···O hydrogen bond (Table 2) between the hydroxyl O3 and carbonyl O2 atoms, forming zigzag chains along the *c* axis (Fig. 2).

Experimental

The title compound was prepared by the reaction of dimedone with 4-hydroxy-3-methoxybenzaldehyde and CH_3NH_2HCl(NaOAc) in glycol under microwave irradiation (yield 71%; m.p. 522–523 K).

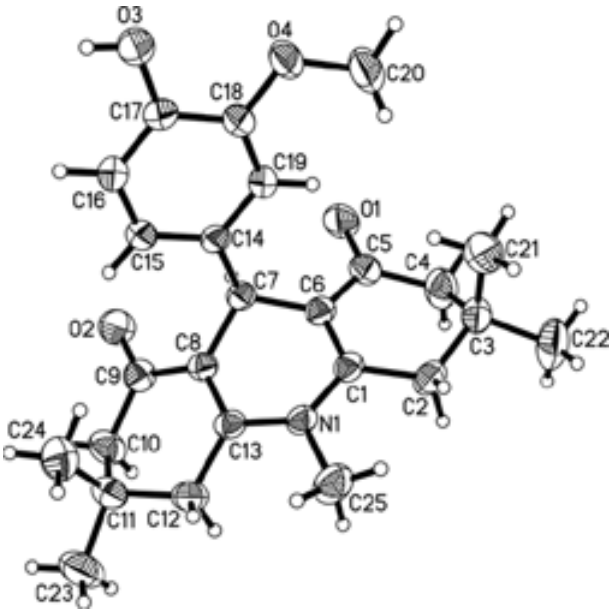


Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

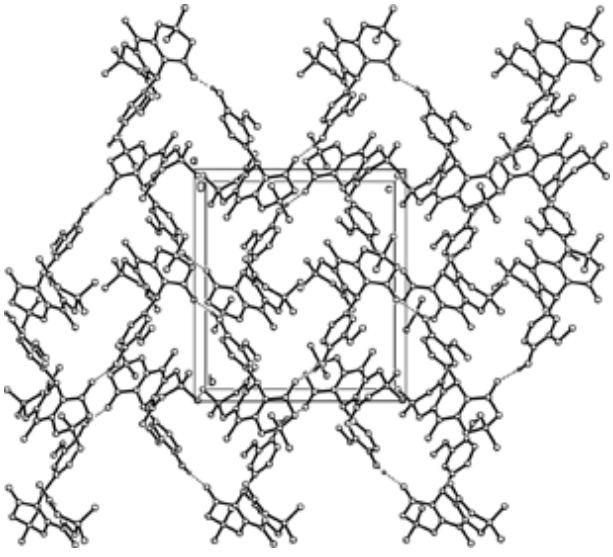


Figure 2
The molecular packing of (I), viewed along the *a* axis. Dashed lines indicate hydrogen bonds.

Single crystals of (I) suitable for X-ray diffraction were obtained from an ethanol solution by slow evaporation.

Crystal data

$C_{25}H_{31}NO_4$
M_r = 409.51
Monoclinic, $P2_1/c$
a = 10.640 (2) Å
b = 14.884 (2) Å
c = 13.604 (2) Å
β = 91.15 (1)°
V = 2154.0 (6) Å^3
Z = 4
D_x = 1.263 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 41 reflections
θ = 3.7–15.2°
μ = 0.09 mm^{-1}
T = 295 (2) K
Block, yellow
0.58 × 0.30 × 0.22 mm

Data collection

Siemens $P4$ diffractometer
ω scans
Absorption correction: none
4445 measured reflections
3899 independent reflections
2409 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
θ_{max} = 25.3°
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 17$
$l = -16 \rightarrow 16$
3 standard reflections
every 97 reflections
intensity decay: 3.4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.108
S = 0.91
3899 reflections
279 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0566P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: *SHELXTL* (Sheldrick, 1997)
Extinction coefficient: 0.0075 (9)

Table 1
Selected geometric parameters (Å, °).

O1—C5	1.242 (2)	N1—C1	1.386 (2)
O2—C9	1.232 (2)	N1—C13	1.400 (2)
O3—C17	1.360 (2)	N1—C25	1.479 (2)
O4—C18	1.376 (2)	C1—C6	1.360 (2)
O4—C20	1.415 (2)	C8—C13	1.363 (2)
C18—O4—C20	117.25 (16)	C1—C2—C3	115.55 (16)
C1—N1—C13	119.07 (14)	C5—C4—C3	113.21 (17)
C1—N1—C25	119.77 (16)	O3—C17—C16	123.97 (18)
C13—N1—C25	119.55 (16)	O3—C17—C18	117.74 (17)
C6—C1—C2	122.50 (17)	O4—C18—C19	124.00 (17)
N1—C1—C2	117.68 (15)	O4—C18—C17	115.69 (17)

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3O···O1^{i}	0.82	1.88	2.698 (2)	175

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

H atoms were placed in geometrically idealized positions and allowed to ride on their parent atoms, with an O—H distance of 0.82 Å and C—H distances in the range 0.93–0.98 Å, and with U_{iso}(H) = 1.2 or 1.5 (for methyl H) times U_{eq}(C,O).

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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