Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

9-(4-Methoxyphenyl)-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 = 193 K
Mean σ(C–C) = 0.003 Å
R factor = 0.044
wR factor = 0.095
Data-to-parameter ratio = 8.8

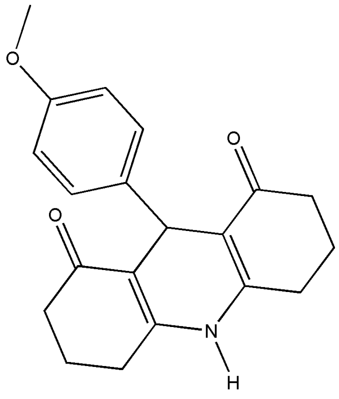
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 4 October 2004
Accepted 12 October 2004
Online 22 October 2004

The title compound, $C_{20}H_{21}NO_3$, was synthesized by the reaction of cyclohexane-1,3-dione with 4-methoxybenzaldehyde and ammonium acetate in glycol under microwave irradition. X-ray analysis reveals that the dihydropyridine ring is in a boat conformation and both cyclohexenone rings adopt envelope conformations. N—H···O hydrogen bonds link the glide-related molecules into zigzag chains along the *a* 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 because of their immense utility in the pharmaceutical and dye industries, and their use as 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 9-(substituted phenyl)-3,3,6,6-tetramethyl-1,2,3,4,5,6,7,8,9,10-decahydrocridine-1,8-dione (Tu *et al.*, 2002). We report here the crystal structure of the title compound, (I).



(I)

The dihydropyridine ring in (I) is in a boat conformation. In this ring, atoms N1 and C3 deviate from the C1/C2/C4/C5 plane by 0.171 (3) and 0.272 (4) Å, respectively (Fig. 1 and Table 1). Both cyclohexenone rings adopt envelope conformations. In the molecule, atoms C7and C8 are out of the C1/C2/C6/C9 plane by 0.0736 and -0.5918 Å, respectively, and atoms C11 and C12 are out of the C4/C5/C10/C13 plane by 0.1002 and -0.5522 Å, respectively. The dihedral angle between the C1/C2/C4/C5 plane and the benzene ring attached at atom C3 is 86.67 (7)°. The methoxy group is nearly coplanar with the attached benzene ring, with a C20—O3—C17—C18 torsion angle of 5.7 (3)°.

Glide-related molecules are linked by N—H···O hydrogen bonds (Table 2), forming zigzag chains along the *a* axis (Fig. 2).

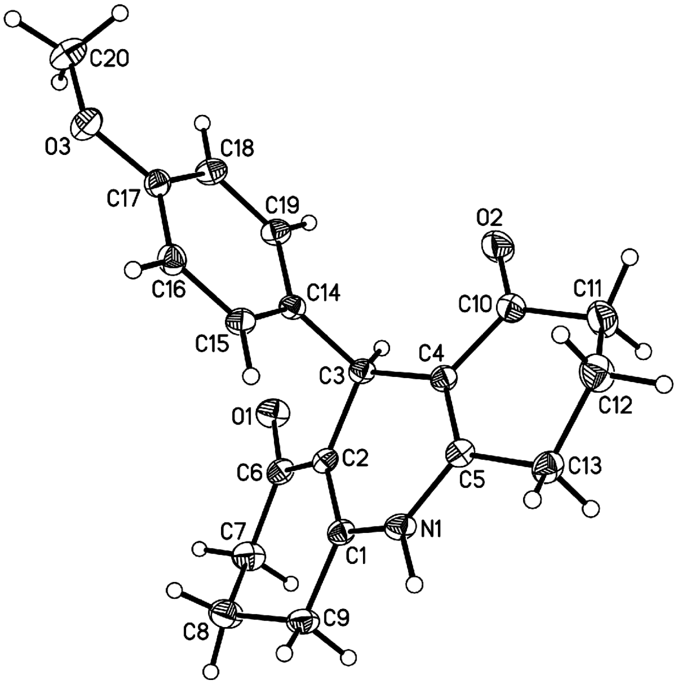


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

Experimental

Compound (I) was prepared by the reaction of cyclohexane-1,3-dione (1 mmol) with 3-methoxy-4-hydroxylbenzaldehyde (2 mmol) and ammonium acetate (2 mmol) in glycol (2 ml) under microwave irradiation (yield 95%, m.p. 573 K). Single crystals suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution.

Crystal data

$C_{20}H_{21}NO_3$
$M_r = 323.38$
Orthorhombic, $Pna2_1$
$a = 14.1794$ (15) Å
$b = 8.6881$ (9) Å
$c = 13.2708$ (13) Å
$V = 1634.9$ (3) Å^3
$Z = 4$
$D_x = 1.314$ Mg m$^{-3$}
Mo $K\alpha$ radiation
Cell parameters from 6503 reflections
$\theta = 3.1$–27.5°
$\mu = 0.09$ mm^{-1}
$T = 193$ (2) K
Block, yellow
0.40 × 0.28 × 0.17 mm

Data collection

Rigaku Mercury diffractometer
ω scans
Absorption correction: multi-scan (Jacobson, 1998)
$T_{\min} = 0.966$, $T_{\max} = 0.985$
17 322 measured reflections
1956 independent reflections
1868 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.035$
$\theta_{\max} = 27.5°$
$h = -18 \rightarrow 18$
$k = -11 \rightarrow 11$
$l = -16 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.095$
$S = 1.10$
1956 reflections
222 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0403P)^2 + 0.3249P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.21$ e Å^{-3}
$\Delta\rho_{\min} = -0.15$ e Å^{-3}

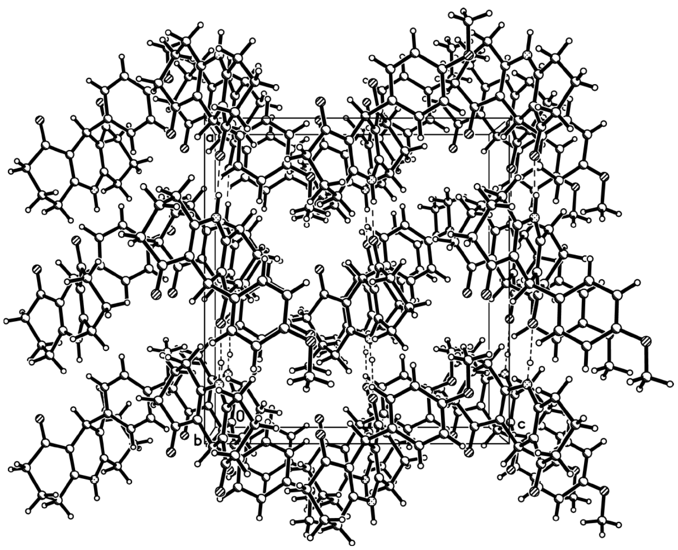


Figure 2
A molecular packing diagram for (I). Dashed lines indicate hydrogen bonds.

Table 1
Selected geometric parameters (Å, °).

O1—C6	1.242 (2)	N1—C1	1.373 (2)
O2—C10	1.226 (2)	N1—C5	1.384 (2)
O3—C17	1.374 (2)	C1—C2	1.361 (2)
O3—C20	1.425 (2)	C4—C5	1.349 (2)
C17—O3—C20	116.59 (15)	C4—C5—C13	123.72 (17)
C1—N1—C5	120.52 (15)	N1—C5—C13	115.59 (15)
C2—C1—C9	123.47 (16)	O2—C10—C11	121.26 (18)
N1—C1—C9	116.27 (15)	C4—C10—C11	117.27 (17)
C1—C2—C3	121.39 (15)	O3—C17—C18	124.12 (16)
C6—C2—C3	118.52 (14)	O3—C17—C16	116.23 (17)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1$^{\rm i}$	0.98 (3)	1.85 (3)	2.824 (2)	170 (2)

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

Atom H1 was located in a difference map and refined isotropically. All other H atoms were positioned geometrically and treated as riding, with C—H distances in the range 0.95–1.00 Å and $U_{\rm iso}$(H) = 1.2 or 1.5 times $U_{\rm eq}$(C). In the absence of significant anomalous dispersion effects, Friedel pairs were merged.

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*.

We thank the Natural Science Foundation of China (No. 20372057) and the Key Laboratory of Biotechnology for Medicinal Plants of Jiangsu Province (No. 01AXL 14) for finanical support.

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