Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

10-Cyclopropyl-9-(4-methoxyphenyl)-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) = 0.002 Å
R factor = 0.056
wR factor = 0.141
Data-to-parameter ratio = 17.2

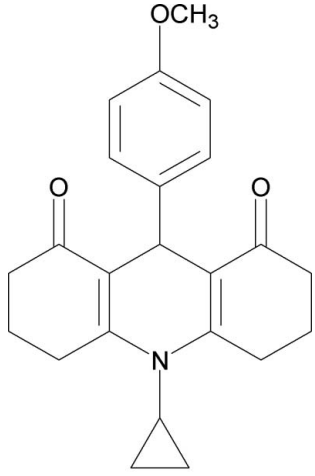
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 13 April 2005
Accepted 20 May 2005
Online 28 May 2005

The title compound, $C_{23}H_{25}NO_3$, was synthesized by the reaction of 1,3-cyclohexanedione with 4-methoxybenzaldehyde, cyclopropylaminium chloride and NaOAc in glycol and water. X-ray analysis reveals that the dihydropyridine ring is in a distorted boat conformation.

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 as a result of their immense utility in the pharmaceutical and dye industries, and 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). We have recently reported the synthesis of *N*-hydroxylacridine-1,8-dione derivatives (Tu, Miao *et al.*, 2004), and the structures of 9-(4-hydroxy-3-methoxyphenyl)-3,3,6,6,10-pentamethyl-1,2,3,4,5,6,7,8,9,10-decahydroacridine-1,8-dione (Tu, Zhang & Zhu, 2004) and 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, Zhang & Xu, 2004). We now report the structure of the title compound, (I) (Fig. 1).



(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 0.282 (2) and 0.441 (3) Å, respectively. Both the cyclohexenone rings adopt sofa conformations. The dihedral angle between the C1/C2/C4/C5 plane and the benzene ring attached at atom C3 is 94.19 (5)°. The methoxy group is almost in the plane of the benzene ring, with a C23—O3—C17—C18 torsion angle of 176.90 (15)°.

Experimental

Compound (I), was prepared by the reaction of 1,3-cyclohexanedione (4 mmol) with 4-methoxybenzaldehyde (2 mmol), cyclopropylamium chloride (3 mmol) and NaOAc (3 mmol) in a mixture of glycol (2 ml) and water (1 ml) under microwave irradiation (yield 94%, m.p. 506–507 K). Single crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution.

Crystal data

$C_{23}H_{25}NO_3$
M_r = 363.44
Monoclinic, $P2_1/c$
a = 11.2668 (10) Å
b = 16.4542 (12) Å
c = 10.8049 (9) Å
β = 111.798 (2)°
V = 1859.9 (3) Å^3
Z = 4
D_x = 1.298 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6571 reflections
θ = 3.1–27.5°
μ = 0.09 mm^{-1}
T = 193 (2) K
Block, colorless
0.50 × 0.30 × 0.20 mm

Data collection

Rigaku Mercury CCD diffractometer
ω scans
Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.959, T_{max} = 0.983
20572 measured reflections
4232 independent reflections
3626 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 27.5°
$h = -14 \rightarrow 14$
$k = -21 \rightarrow 18$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.142
S = 1.11
4232 reflections
246 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.062P)^2 + 0.8612P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.79 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C6	1.229 (2)	C4—C5	1.356 (2)
O2—C10	1.227 (2)	C4—C10	1.453 (2)
N1—C5	1.395 (2)	C5—C13	1.511 (2)
N1—C1	1.403 (2)	C6—C7	1.505 (2)
C1—C2	1.358 (2)	C7—C8	1.519 (2)
C1—C9	1.502 (2)	C8—C9	1.520 (2)
C2—C6	1.464 (2)	C10—C11	1.514 (3)
C2—C3	1.505 (2)	C11—C12	1.497 (3)
C3—C4	1.519 (2)	C12—C13	1.514 (3)
C5—N1—C1	117.76 (13)	N1—C5—C13	118.01 (15)
C2—C1—N1	119.34 (14)	O1—C6—C2	121.36 (15)
C2—C1—C9	123.01 (14)	O1—C6—C7	121.01 (15)
N1—C1—C9	117.40 (14)	C2—C6—C7	117.59 (14)
C1—C2—C6	121.09 (14)	C6—C7—C8	111.78 (14)
C1—C2—C3	120.04 (14)	C7—C8—C9	111.31 (14)
C6—C2—C3	118.70 (13)	C1—C9—C8	111.73 (14)
C2—C3—C4	107.55 (12)	O2—C10—C4	121.93 (16)
C5—C4—C10	121.42 (15)	O2—C10—C11	120.65 (17)
C5—C4—C3	119.96 (14)	C4—C10—C11	117.41 (17)
C10—C4—C3	118.61 (14)	C12—C11—C10	113.43 (17)
C4—C5—N1	119.24 (14)	C11—C12—C13	112.09 (18)
C4—C5—C13	122.69 (15)	C5—C13—C12	110.51 (16)
C1—C2—C3—C14	−89.65 (17)	C23—O3—C17—C18	176.90 (15)

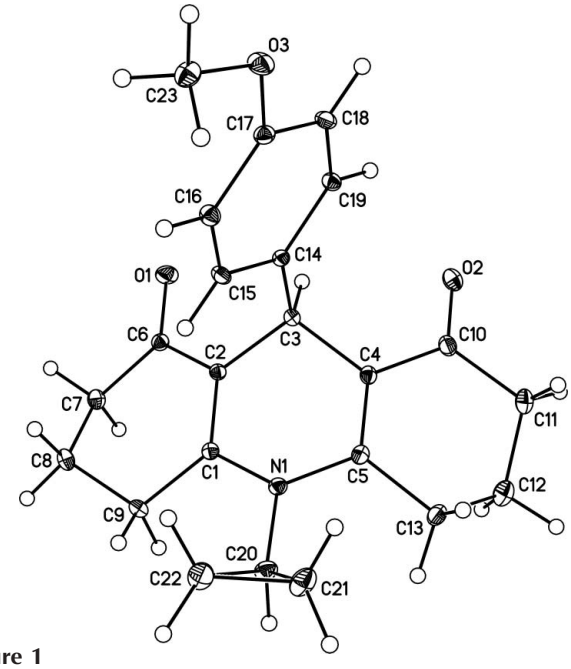


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

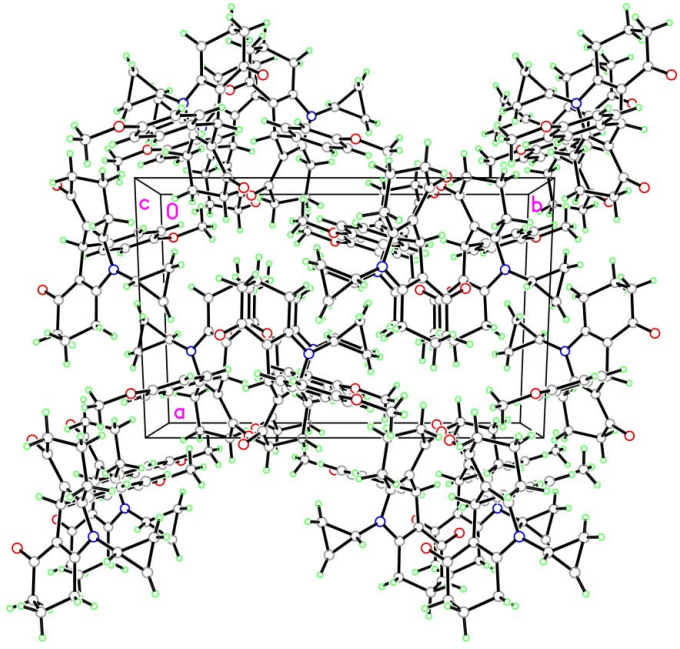


Figure 2
The molecular packing of (I), viewed along the *c* axis.

H atoms were treated using a riding-model approximation, with C—H distances of 0.95–1.00 Å, and with U_{iso}(H) = 1.2U_{eq}(C) and 1.5U_{eq}(C_{methyl}).

Data collection: *CrystalClear* (Rigaku Corporation, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2003); 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*.

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 financial support.

References

Jacobson, R. (1998). Private communication to the Rigaku Corporation, Tokyo, Japan.
Mandi, Y., Regely, K., Ocsovszky, I., Barbe, J., Galy, J. P. & Molnar, J. (1994). *Anticancer Res.* **14**, 2633–2636.
Nasim, A. & Brychey, T. (1979). *Mutat. Res.* **65**, 261–288.
Reil, E., Scoll, M., Masson, K. & Oettmeier, W. (1994). *Biochem. Soc. Trans.* **22**, 62s.
Rigaku Corporation (1999). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2003). *CrystalStructure*. Rigaku/MSC, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.
Sheldrick, G. M. (1997*a*). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Thull, U. & Testa, B. (1994). *Biochem. Pharmacol.* **47**, 2307–2310.
Tu, S. J., Miao, C. B., Gao, Y., Fang, F., Zhuang, Q. Y., Feng,Y. J. & Shi, D. Q. (2004). *Synlett*, **2**, 255–258.
Tu, S. J., Zhang, X. J. & Xu, J. N. (2004). *Acta Cryst.* E**60**, o2328–o2330.
Tu, S. J., Zhang, X. J. & Zhu, S. L. (2004). *Acta Cryst.* E**60**, o1870–o1872.
Wysocka-Skrzela, B. & Ledochowski, A. (1976). *Rocz. Chem.* **50**, 127–131.