

Table 3. Asymmetry parameters (\AA) of the three molecules of (I)

Ring	Molecule	Asymmetry Parameter	Conformation
C7, C6, C12, C13, C14, N15	A	$\Delta_1(\text{C7})$ 0.010 (2)	Sofa
	B	$\Delta_1(\text{C7})$ 0.011 (1)	Sofa
	C	$\Delta_2(\text{C7-N15})$ 0.027 (2)	Half-chair
C7, C6, C12, C13', C14, N15	A	$\Delta_1(\text{C7})$ 0.012 (2)	Sofa
	B	$\Delta_1(\text{C7})$ 0.008 (1)	Sofa
	C	$\Delta_2(\text{C7-C6})$ 0.033 (2), $\Delta_1(\text{C7})$ 0.068 (2)	Half-chair/Sofa
C8, C7, N15, C16, C17, C18	A	$\Delta_2(\text{N15-C7})$ 0.028 (2), $\Delta_1(\text{C7})$ 0.041 (2)	Half-chair/Sofa
	B	$\Delta_1(\text{C7})$ 0.018 (2)	Sofa
	C	$\Delta_1(\text{C7})$ 0.043 (1), $\Delta_2(\text{C7-C8})$ 0.069 (1)	Sofa/Half-chair
C8, C7, N15, C16, C17', C18	A	$\Delta_1(\text{C7})$ 0.042 (2), $\Delta_2(\text{C7-C8})$ 0.070 (1)	Sofa/Half-chair
	B	$\Delta_1(\text{C7})$ 0.018 (2)	Sofa

The structure was solved by direct methods using the 'brute force' option (TREF 1000 with 500 reflections), since the structure solution was not possible with default options (SHELXS86; Sheldrick, 1990a). All the disordered atoms were located from the difference Fourier maps. The site occupancy factors of these atoms were refined with isotropic displacement parameters and values ranging from 0.4 to 0.6 were obtained in the early stages and later fixed at 0.5 during anisotropic refinements due to their large correlation with displacement parameters. The H atoms attached to all the normal atoms were located from difference Fourier maps and refined isotropically, while the H atoms attached to the disordered atoms were fixed geometrically and allowed to ride on their respective C atoms with an isotropic displacement parameter U of 0.08 \AA^2 .

Data collection: XSCANS (Siemens, 1994). Cell refinement: XSCANS. Data reduction: XSCANS. Program(s) used to solve structure: SHELXS86 (Sheldrick, 1990a). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: SHELXTL/PC (Sheldrick, 1990b). Software used to prepare material for publication: SHELXL93. Geometrical calculations: PARST (Nardelli, 1983b).

The authors thank the Malaysian Government and Universiti Sains Malaysia for research grant R & D No. 123-3417-2201. One of us (KS) thanks Universiti Sains Malaysia for a Visiting Post Doctoral Research Fellowship.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: KH1030). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Chinnakali, K., Selladurai, S., Sivakumar, K., Subramanian, K. & Natarajan, S. (1990). *Acta Cryst.* **C46**, 837–839.
 Chinnakali, K., Sivakumar, K. & Natarajan, S. (1990a). *Acta Cryst.* **C46**, 669–671.
 Chinnakali, K., Sivakumar, K. & Natarajan, S. (1990b). *Acta Cryst.* **C46**, 833–835.
 Chinnakali, K., Sivakumar, K. & Natarajan, S. (1992). *Acta Cryst.* **C48**, 386–387.
 Gridunova, G. V., Yufit, D. S., Struchkov, Yu. T., Khrolova, O. R., Reznichenko, A. Z. & Tavrizova, M. A. (1992). *Kristallografiya*, **37**, 366–372.

- Jasinski, J. P. & Paight, E. S. (1994). *Acta Cryst.* **C50**, 1928–1930.
 Jasinski, J. P. & Woudenberg, R. C. (1995). *Acta Cryst.* **C51**, 107–109.
 Kunjappu, J. T. (1993). *J. Photochem. Photobiol. A*, **71**, 269–273.
 McCarthy, P. M. & Blanchard, G. J. (1993). *J. Phys. Chem.* **97**, 12205–12209, and references therein.
 Maeda, M. (1984). *Laser Dyes: Properties of Organic Compounds for Dye Lasers*, pp. 224–225, and references therein. New York: Academic Press.
 Nardelli, M. (1983a). *Acta Cryst.* **C39**, 1141–1142.
 Nardelli, M. (1983b). *Comput. Chem.* **7**, 95–98.
 Priyadarsini, K. I., Naik, D. B. & Moorthy, P. N. (1989). *J. Photochem. Photobiol. A*, **46**, 239–246.
 Sheldrick, G. M. (1990a). *Acta Cryst.* **A46**, 467–473.
 Sheldrick, G. M. (1990b). *SHELXTL/PC. Structure Determination Software Programs*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
 Siemens (1994). *XSCANS. X-ray Single Crystal Analysis System. Version 2.1*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Sivakumar, K. & Chinnakali, K. (1991). Unpublished results.
 Yip, B. C., Fun, H.-K., Sivakumar, K., Zhou, Z. Y., Shawkataly, B. O. & Teoh, S. G. (1995). *Acta Cryst.* **C51**, 956–958.

Acta Cryst. (1996). **C52**, 481–483

3,3,6,6-Tetramethyl-10-(4-methylphenyl)-3,4,6,7,9,10-hexahydro-1,8(2H,5H)-acridinedione

J. SIVARAMAN,^a K. SUBRAMANIAN,^a D. VELMURUGAN,^b
 E. SUBRAMANIAN^b AND P. S. SHANMUGASUNDRAM^c

^aDepartment of Physics, Anna University, Madras 25, India,
^bDepartment of Crystallography and Biophysics, University of Madras, Madras 25, India, and ^cDepartment of Organic Chemistry, University of Madras, Madras 25, India

(Received 6 July 1994; accepted 24 July 1995)

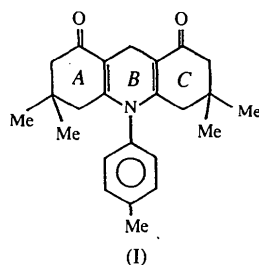
Abstract

The title molecule, $\text{C}_{24}\text{H}_{29}\text{NO}_2$, consists of a partially hydrogenated acridine ring system with one phenyl substituent on the dihydropyridine ring. The compound crystallizes with a half molecule per asymmetric unit, which has twofold symmetry. The dihydropyridine ring is almost planar, whereas both the outer rings adopt half-chair conformations.

Comment

Compounds of the acridine type have long been known to be potent frameshift mutagens in viruses and bacteria (Acheson, 1956). Acridines bind to DNA by intercalation (Lerman, 1961; Karle, Cysyk & Karle, 1980;

Nandi, Debnath & Maiti, 1990; Reddy, Seshadri, Sakore & Sobell, 1979; Sakore, Reddy & Sobell, 1979; Peacocke & Skerrett, 1956). In addition to this, these compounds are said to possess anti-amoebic (Prasad Krishna, Banasal, Das & Srivastava, 1984), hypertensive, anti-inflammatory and anti-implantation (Asthana, Rastogi, Ghose & Das, 1991) activities. The DNA-intercalation property of acridine is generally acknowledged to be partly responsible for its biological activity. The molecular geometry and atomic numbering scheme of 3,3,6,6-tetramethyl-10-(4-methylphenyl)-3,4,6,7,9,10-hexahydro-1,8(2*H*,5*H*)-acridinedione, (I), are depicted in Fig. 1. A twofold axis bisecting the molecule passes through atoms C(5), N(6), C(10), C(13) and C(13'). This twofold axis is parallel to the *b* axis passing through $(\frac{1}{2}, y, \frac{1}{4})$.



The average $C_{sp^2}-C_{sp^2}$ bond length within the planar phenyl ring [$(\Delta/\sigma)^2 = 19.5$] is 1.389(1) Å and this agrees well with the expected value (1.39 Å). The mean-plane calculations indicate that ring A [C(1)-C(4a), C(7)] is in a half-chair conformation [$\Delta C_2 = 16.26^\circ$] and that the dihydropyridine ring is nearly planar [$(\Delta/\sigma)^2 = 4.8$]. The dihedral angle between rings

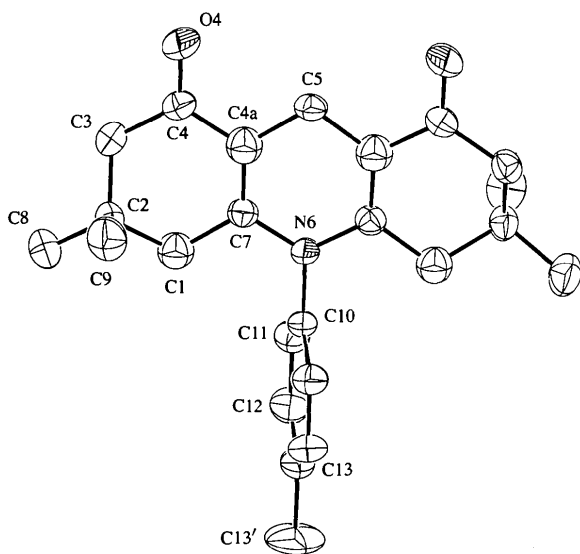


Fig. 1. A perspective view showing the atomic numbering scheme of the title molecule. Displacement ellipsoids are plotted at the 50% probability level.

A and C is $10.6(1)^\circ$ and shows considerable buckling of the acridine nucleus. The N(6) atom is almost in the plane of the dihydropyridine ring and the sum of the valency angles around N(6) is 360° , which is the theoretical value for sp^2 hybridization. The methyl C(9) and C(8) atoms in the acridine ring system adopt axial and equatorial positions, respectively [deviations of $-1.804(6)$ and $0.302(6)$ Å for C(9) and C(8), respectively], whereas the ketone O(4) atom and the methyl C(13') atom [deviations of $-0.125(5)$ and $0.000(0)$ Å for O(4) and C(13'), respectively] have inclined and coplanar orientations, respectively. The plane of the phenyl ring is orthogonal with respect to the acridine moiety, the angle between them being $101.8(2)^\circ$. Finally, the molecular packing diagram (Fig. 2) displays a noteworthy feature of this structure. Alternate symmetry-related acridine molecules are stacked in an almost parallel manner. This type of stacking is also found in 9-aminoacridine (Talacki, Carrell & Glusker, 1974) and in charge-transfer compounds (Karle, Bultman & Jard, 1976), and is also observed in 9-methyl-10-(4-methylphenyl)-3,4,6,7,9,10-hexahydro-1,8-(2*H*,5*H*)-acridinedione (Sivaraman, Subramanian, Velmurugan, Subramanian & Ramakrishnan, 1994). It is also similar to the stacking of bases in DNA which are separated by $3.4(2)$ Å (Lehninger, 1970). In addition to these stacking interactions, the packing of the molecules is purely the result of van der Waals interactions.

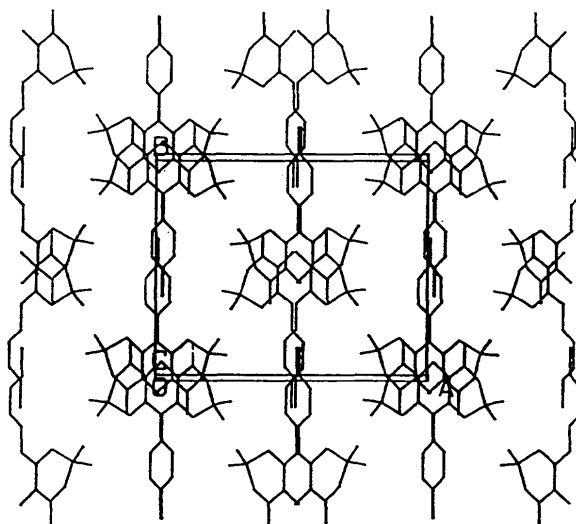


Fig. 2. Packing of the title molecules in the unit cell.

Experimental

The title compound was synthesized by condensation of cyclohexane-1,3-dione with aldehyde, furnishing the tetraketone which on reaction with aromatic amine afforded the acridinedione. The crystal density D_m was measured by flotation in water.

Crystal dataC₂₄H₂₉NO₂*M_r* = 363

Orthorhombic

*Pbcn**a* = 15.899 (1) Å*b* = 12.272 (2) Å*c* = 10.618 (1) Å*V* = 2071.71 (1) Å³*Z* = 4*D_x* = 1.169 Mg m⁻³*D_m* = 1.182 Mg m⁻³Cu *K*α radiation

λ = 1.54184 Å

Cell parameters from 20

reflections

θ = 15–25°

μ = 0.54 mm⁻¹*T* = 293 K

Needle shaped

0.50 × 0.20 × 0.15 mm

Brown

C(1)—C(2)—C(3)	108.9 (4)	C(9)—C(2)—C(3)	109.9 (4)
C(2)—C(3)—C(4)	114.4 (4)	C(3)—C(4)—O(4)	121.2 (4)
C(3)—C(4)—C(4a)	118.9 (4)	O(4)—C(4)—C(4a)	119.9 (4)
C(4)—C(4a)—C(7)	120.4 (3)	C(7)—N(6)—C(10)	119.8 (2)
C(4a)—C(7)—C(1)	122.6 (6)	N(6)—C(10)—C(11)	119.6 (2)
C(4a)—C(7)—N(6)	120.8 (3)	C(10)—C(11)—C(12)	119.1 (4)
C(7)—C(1)—C(2)	114.6 (4)	C(11)—C(12)—C(13)	121.8 (4)
C(1)—C(2)—C(8)	108.2 (4)	C(12)—C(13)—C(13')	121.2 (2)
C(8)—C(2)—C(9)	109.9 (4)		

The structure was solved by direct methods. The H atoms located from a difference Fourier map were refined for one cycle, whereas the geometrically fixed atoms [H(5*a*), H(5*b*), H(13*a*), H(13*b*) and H(13*c*)] were not refined. Full-matrix least-squares refinement of anisotropic displacement parameters for all non-H and of isotropic displacement parameters for H atoms was carried out.

Cell refinement and data reduction: *SDP* (Frenz, 1978). Program used to solve structure: *SHELXS86* (Sheldrick, 1985). Program used to refine structure: *SHELXL76* (Sheldrick, 1976). Software used to prepare material for publication: *PARST* (Nardelli, 1983) and *PLATON93* (Spek, 1993).

Data collection

Enraf–Nonius CAD-4

diffractometer

ω/2θ scans

Absorption correction:

ψ scan (Frenz, 1978)

T_{min} = 0.38, *T_{max}* = 0.51

1812 measured reflections

1788 independent reflections

1355 observed reflections

[*I* > 3σ(*I*)]*R_{int}* = 0.051θ_{max} = 65°*h* = 0 → 19*k* = 0 → 14*l* = 0 → 12

2 standard reflections

monitored every 100

reflections

intensity decay: <2%

RefinementRefinement on *F**R* = 0.056*wR* = 0.062*S* = 1.38

1355 reflections

168 parameters

w = 1/[σ²(*F*) + 0.013077*F*²](Δ/σ)_{max} = 0.098Δρ_{max} = 0.474 e Å⁻³Δρ_{min} = -0.399 e Å⁻³

Atomic scattering factors

from *International Tables*for *X-ray Crystallography*

(1974, Vol. IV)

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, least-squares-planes data and torsion angles have been deposited with the IUCr (Reference: SZ1019). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Acheson, R. M. (1956). In *Acridines*, 1st ed. London: Arnold Press.
- Asthana, P., Rastogi, S., Ghose, S. & Das, S. R. (1991). *Indian J. Chem.* **30B**, 893–900.
- Frenz, B. A. (1978). *The Enraf–Nonius CAD-4 SDP – a Real-Time System for Concurrent X-ray Data Collection and Crystal Structure Solution. Computing in Crystallography*, edited by H. Schenk, R. Olthof-Hazekamp, H. van Koningsveld & G. C. Bassi, pp. 64–71. Delft University Press.
- Karle, I. L., Bultman, J. D. & Jard, L. (1976). *Acta Cryst.* **B32**, 1963–1968.
- Karle, M. J., Cysyk, L. R. & Karle, L. (1980). *Acta Cryst.* **B36**, 3012–3016.
- Lehninger, A. L. (1970). *Biochemistry*, p. 98. New York: Worth Publishing Inc.
- Lerman, L. S. (1961). *J. Mol. Biol.* **3**, 18–30.
- Nandi, R., Debnath, D. & Maiti, M. (1990). *Biochem. Biophys. Acta*, **104**, 339–342.
- Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.
- Peacocke, A. R. & Skerrett, J. N. H. (1956). *Trans. Faraday Soc.* **52**, 261–279.
- Prasad Krishna, B. N., Banasal, I., Das, P. & Srivastava, R. (1984). *Curr. Sci.* **53**, 778–780.
- Reddy, B. C., Seshadri, T. P., Sakore, T. P. & Sobell, H. M. (1979). *J. Mol. Biol.* **135**, 787–812.
- Sakore, T. D., Reddy, S. B. & Sobell, M. H. (1979). *J. Mol. Biol.* **135**, 763–785.
- Sheldrick, G. M. (1976). *SHELXL76. Program for Crystal Structure Determination*. University of Cambridge, England.
- Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures*. University of Göttingen, Germany.
- Sivaraman, J., Subramanian, K., Velmurugan, D., Subramanian, E. & Ramakrishnan, V. T. (1994). *Acta Cryst.* **C50**, 2011–2013.
- Spek, A. L. (1993). *PLATON93. Program for the Automated Analysis of Molecular Geometry*. University of Utrecht, The Netherlands.
- Talacki, R., Carrell, H. L. & Glusker, J. P. (1974). *Acta Cryst.* **B30**, 1044–1047.

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$B_{eq} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B_{eq}</i>
C(1)	0.3897 (3)	0.3318 (4)	0.0871 (5)	3.4 (1)
C(2)	0.3116 (3)	0.3922 (4)	0.0365 (4)	3.3 (1)
C(3)	0.3382 (3)	0.5038 (4)	-0.0108 (5)	3.3 (1)
C(4)	0.3905 (3)	0.5679 (3)	0.0805 (4)	3.0 (1)
C(4a)	0.4452 (2)	0.5096 (3)	0.1673 (4)	2.6 (1)
O(4)	0.3900 (3)	0.6681 (3)	0.0801 (4)	4.7 (1)
C(5)	1/2	0.5783 (4)	1/4	2.8 (2)
N(6)	1/2	0.3427 (3)	1/4	2.7 (1)
C(7)	0.4457 (2)	0.3994 (3)	0.1696 (4)	2.7 (1)
C(8)	0.2748 (4)	0.3253 (5)	-0.0721 (6)	4.6 (2)
C(9)	0.2458 (4)	0.4047 (6)	0.1415 (6)	5.0 (2)
C(10)	1/2	0.2243 (4)	1/4	2.7 (2)
C(11)	0.5414 (3)	0.1685 (4)	0.1551 (5)	3.5 (1)
C(12)	0.5409 (4)	0.0552 (4)	0.1561 (6)	4.3 (1)
C(13)	1/2	-0.0036 (5)	1/4	4.5 (2)
C(13')	1/2	-0.1273 (12)	1/4	8.2 (9)

Table 2. Selected geometric parameters (Å, °)

C(1)—C(2)	1.545 (7)	C(4)—O(4)	1.230 (5)
C(2)—C(3)	1.519 (7)	C(4a)—C(5)	1.497 (5)
C(3)—C(4)	1.500 (7)	C(7)—N(6)	1.399 (4)
C(4)—C(4a)	1.455 (6)	N(6)—C(10)	1.453 (6)
C(4a)—C(7)	1.355 (5)	C(10)—C(11)	1.385 (6)
C(7)—C(1)	1.499 (6)	C(11)—C(12)	1.391 (7)
C(9)—C(2)	1.537 (8)	C(12)—C(13)	1.392 (7)
C(2)—C(8)	1.532 (8)	C(13)—C(13')	1.518 (16)