Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1208). Services for accessing these data are described at the back of the journal.

References

B. A. Frenz & Associates Inc. (1983). *SDP-Plus Structure Determination Package*. College Station, Texas, USA, and Enraf–Nonius, Delft, The Netherlands.
Chasseau, D., Gaultier, J. & Hauw, C. (1971). *C. R. Acad. Sci Paris*, **274**, 1434–1437.
Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Fritchie, C. J. (1966). *Acta Cryst.* **20**, 892–898.
Goldberg, I. & Shmueli, U. (1973). *Acta Cryst.* B**29**, 421–431.
Hanson, A. W. (1965). *Acta Cryst.* **19**, 610–613.
Kistenmacher, T. J., Phillips T. E. & Cowan, D. O. (1974). *Acta Cryst.* B**30**, 763–768.
Sheldrick, G. M. (1990). *SHELXTL-Plus*. Version 4.0. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXL97. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

Acta Cryst. (1998). C**54**, 1534–1535

1,2,3,4,5,6,7,8,9,10-Decahydro-3,3,6,6-tetramethyl-1,8-dioxo-10-vinylacridin-9-ylmethyl Acetate

R. Sankaranarayanan,[a] D. Velmurugan,[a] P. Murugan[b] and N. Ramasubbu[c]

[a]*Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Madras 600 025, India,* [b]*Department of Organic Chemistry, University of Madras, Guindy Campus, Madras 600 025, India, and* [c]*Department of Oral Biology and Dental Research Institute, State University of New York at Buffalo, Buffalo, NY 14214, USA. E-mail: crystal@giasmd01.vsnl.net.in*

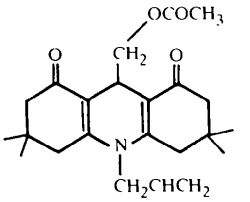
(*Received 12 August 1997; accepted 8 April 1998*)

Abstract

In the title compound, $C_{23}H_{31}NO_4$, the central piperidine ring adopts a sofa conformation, while that of the outer rings is half-chair. The molecule is folded about the line passing through the central C and N atoms. The puckering amplitude of the piperidine ring is small, due to π conjugation. The methyl acetate substituent occupies an axial position. The packing is stabilized by C—H···O hydrogen bonds.

Comment

The potency of acridines as antiviral and antibacterial agents is due to their ability to bind with DNA by intercalation (Neidle, 1979). Acridine diones are laser active and fluoresce well in alcohol solvents (Selladurai *et al.*, 1990). With a view to determining the conformation of this class of compounds, the title compound, (I), was considered for crystallographic study.



(I)

The bond distances of the keto groups of the acridine moiety [C6—O6 = 1.232 (5) and C4—O4 = 1.224 (5) Å] are longer than that of the carbonyl group of the acetoxy substituent [C17—O18 = 1.189 (7) Å], and this behaviour agrees with the literature (Gunasekaran *et al.*, 1996). The angles around N10 sum to 359.4 (5)°, which is indicative of sp^2 hybridization. The acridine moiety is folded about the line passing through C5 and N10, as seen from the dihedral angle of 3.10 (8)° between the two halves (C1–C5, C4a, C10, N10 and C5–C9, C6a, C9a, N10).

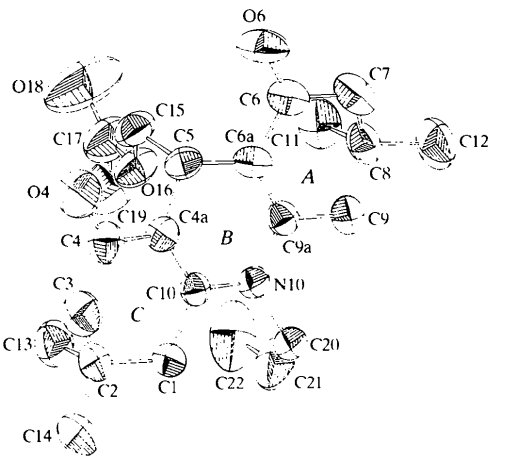


Fig. 1. *ORTEP*II (Johnson, 1976) plot, showing the molecular structure of the title compound and the atom-labelling scheme. Displacement ellipsoids are shown at the 50% probability level.

The atoms of the acetoxy substituent, which occupies an axial position, lie in a plane making a dihedral angle of 80.0 (2)° with the plane through the central ring. The total puckering amplitudes (Cremer & Pople, 1975) of the rings give a quantitative evaluation of puckering [Q_T = 0.492 (5), 0.168 (4), 0.486 (5) for rings *A*, *B* and

C, respectively], and the asymmetry parameters (Duax *et al.*, 1976) reveal sofa conformations for the three rings. The puckering of the central *B* ring is quite small, owing to the π conjugation along the C4a—C10—N10—C9a—C6a system, as indicated by the values of the distances: C4a—C10 = 1.354 (5), C10—N10 = 1.387 (5), C9a—N10 = 1.396 (5) and C6a—C9a = 1.351 (5) Å.

In addition to the van der Waals interactions, two intermolecular C—H···O hydrogen bonds stabilize the molecular packing: C20···O6^{i} = 3.355 (6), H20*B*···O6^{i} = 2.62 Å and C20—H20*B*···O6^{i} = 133°, and C9···O6^{i} = 3.334 (6), H9*B*···O6^{i} = 2.48 Å and C9—H9*B*···O6^{i} = 147° [symmetry code: (i) $x + \frac{1}{2}, -y - \frac{1}{2}, -z$].

Experimental

The title compound was synthesized by the procedure of Murugan & Ramakrishnan (1997), in which a mixture of the tetraketone (5 mmol) and allyl amine (5 mmol) was refluxed in acetic acid for 6–7 h. The reaction mixture was cooled and poured into crushed ice. The separated solid was filtered and recrystallized from methanol–chloroform (1:2).

Crystal data

$C_{23}H_{31}NO_4$
M_r = 385.49
Orthorhombic
$P2_12_12_1$
a = 9.747 (5) Å
b = 14.882 (4) Å
c = 15.480 (5) Å
V = 2245.4 (15) Å^3
Z = 4
D_x = 1.140 Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 25 reflections
θ = 5–20°
μ = 0.619 mm^{-1}
T = 293 (2) K
Rectangular
0.40 × 0.25 × 0.20 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2395 measured reflections
2395 independent reflections
1496 reflections with $I > 2\sigma(I)$

θ_{max} = 69.83°
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 18$
$l = 0 \rightarrow 18$
3 standard reflections every 200 reflections
intensity decay: <2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.158
S = 1.089
2395 reflections
259 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0691P)^2 + 0.1522P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.153 e Å^{-3}
$\Delta\rho_{min}$ = −0.167 e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.0029 (4)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

C1—C10	1.508 (6)	C6—C6a	1.455 (6)
C4—C4a	1.447 (6)	C9—C9a	1.510 (5)
C4a—C5	1.500 (6)	C21—C22	1.269 (9)
C5—C6a	1.503 (6)		
C10—N10—C9a	120.0 (3)	C9a—N10—C20	119.4 (3)
C10—N10—C20	120.0 (3)	C22—C21—C20	127.0 (6)
C10—C4a—C5—C15	107.3 (4)	C15—C5—C6a—C9a	−105.6 (5)

It was not possible to define the absolute structure, given it involves such weak anomalous scatterers as O, N and C.

Data collection: *SDP* (Frenz, 1978). Cell refinement: *SDP*. Data reduction: *SDP*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93 and *PARST* (Nardelli, 1983, 1995).

RS thanks DST for a fellowship and DV thanks UGC (India) for providing funding under the UGC Career Award scheme. We thank Dr M. J. Levine for use of the CAD-4 facility at the Research Center in Oral Biology, New York, USA, funded by USPHS grant DE08240.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1326). Services for accessing these data are described at the back of the journal.

References

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Duax, W. L., Weeks, C. M. & Rohrer, D. C. (1976). *Topics in Stereochemistry*, Vol. 9, edited by E. L. Eliel & N. Allinger, pp. 271–383. New York: John Wiley.
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.
Gunasekaran, K., Velmurugan, D., Murugan, P., Ramakrishnan, V. T., Panneerselvam, K. & Soriano-García, M. (1996). *Acta Cryst.* C**52**, 2609–2612.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Murugan, P. & Ramakrishnan, V. T. (1997). *Indian J. Heterocycl. Chem.* **7**, 31–34.
Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Neidle, S. (1979). *Prog. Med. Chem.* **16**, 151–221.
Selladurai, S., Subramanian, K. & Ramakrishnan, V. T. (1990). *J. Crystallogr. Spectrosc. Res.* **20**, 227–232.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.