1,2,3,4,5,6,7,8-Octahydro-3,3,6,6-tetramethylacridine-1,8-dione

R. Sankaranarayanan,[a] S. Shanmuga Sundara Raj,[b] D. Velmurugan[a] and Hoong-Kun Fun[b]

[a]Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, and [b]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia. E-mail: crystal@giasmd01.vsnl.net.in

Abstract

In the title compound, $C_{17}H_{21}NO_2$, the central pyridine ring is almost planar and the outer rings are half-chairs. The O atoms of the keto groups deviate significantly from the mean plane passing through their corresponding rings. The molecule contains a pseudo-mirror plane which passes through the central N and C atoms and the molecule is slightly folded along an axis passing through the same atoms. The dihedral angle between the outer rings is 6.5 (2)°. The packing is stabilized by an intermolecular C—H···O hydrogen bond.

Comment

Acridines are found to have a wide range of biological activities, such as mutagenic, antitumour (Talacki et al., 1974), antibacterial (Achenson, 1956), antiamoebic (Prasad Krishna et al., 1984), hypertensive, antiinflammatory and antiimplantation (Asthana et al., 1991) activities. A drug containing the acridine moiety has been found to possess antiprotozoal activity (Karolak-Wojciechowska et al., 1996). The ability of acridine to intercalate between the base-pairs of DNA is also well known (Neidle, 1979; Fan et al., 1997). Acridine compounds are considered to be efficient drugs for the treatment of Alzheimer's disease (Bandoli et al., 1994). The use of decahydroacridine-1,8-diones as photo-sensitizers is also well known (Timpe et al., 1993). In addition, acridine diones act as laser dyes whose laser activity has been studied extensively (Murugan et al., 1998). Because of these wide-ranging biological and photochemical properties, an X-ray diffraction study has been undertaken for the title compound, (I), so that its structure–activity correlations could be studied.
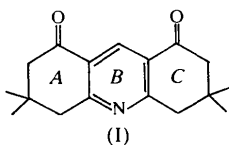
Interestingly, the molecule of (I) contains a pseudo-mirror plane which passes through N10 and C5. A comparison of equivalent bond lengths and torsion angles across this pseudo-mirror plane indicates the presence of this non-crystallographic symmetry element. The keto bond distances C4=O4 [1.218 (3) Å] and C6=O6 [1.225 (4) Å] are comparable with those in similar structures (Sankaranarayanan et al., 1998; Ganesh et al., 1998). The deviations of atoms O4 and O6 from the mean planes passing through rings A and C are −0.287 (3) and −0.299 (3) Å, respectively. The π conjugation along C5—C6a—C9a—N10—C10—C4a [C5—C6a = 1.383 (4), C6a—C9a = 1.400 (4), C9a—N10 = 1.347 (3), N10—C10 = 1.350 (3), C10—C4a = 1.400 (4) and C5—C4a = 1.396 (4) Å] indicates the strong aromaticity in the central ring B, which makes all the atoms of the ring lie almost in a plane, with the maximum deviation being 0.006 (3) Å for C6a and C9a. This planarity of the central ring B is further supported by the low value of the puckering amplitude (Cremer & Pople, 1975), $Q_T = 0.009$ (3) Å, when compared with the values for the other rings, A and C, for which $Q_T = 0.470$ (3) and 0.477 (3) Å, respectively. The asymmetry parameter (Nardelli, 1983a) for ring C is $\Delta C_2 = 0.057$ (2), revealing a half-chair conformation for this ring with a local pseudo-twofold axis running through the midpoint of the C7—C8 bond. The asymmetry parameter for ring A, $\Delta C_2(C2—C3) = 0.058$ (1), reveals the conformation of ring A as a half-chair with a local twofold axis running through the midpoint of the C2—C3 bond. The dihedral angle between the two mean planes passing through C5/C6a/C6/C7/C8/C9/C9a/N10 and C5/C4a/C4/C3/C2/C1/C10/N10 is 5.7 (1)°, indicating that the acridine moiety is slightly folded about a line passing through N10 and C5, as observed in related structures (Gunasekaran et al., 1996; Sankaranarayanan et al., 1998). The dihedral angle between rings A and C is 6.5 (2)°.

In addition to van der Waals interactions, an intermolecular C—H···O hydrogen bond stabilizes the molecular packing: C14···O6^i = 3.519 (4) and
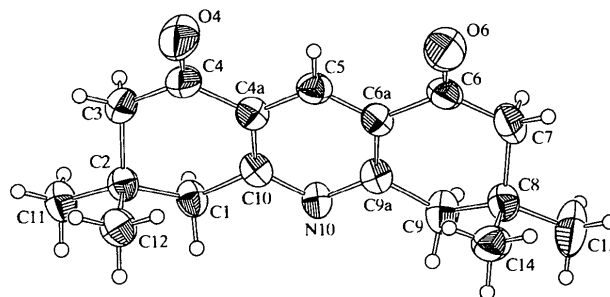

Fig. 1. The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. H atoms are drawn as spheres of arbitrary radii.

$H14A \cdots O6^i = 2.641$ Å, and $C14—H14A \cdots O6^i = 152.2°$ [symmetry code: (i) $x - 1, y, z$].

Experimental

3,3,6,6-Tetramethyl-3,4,6,7,9,10-hexahydro-1,8($2H,5H$)-acridinedione (10 mmol) was dissolved in chloroform (100 ml) and stirred at room temperature with active MnO_2 (5 g) for 8 h. The MnO_2 was filtered off and the filtrate concentrated; the solid obtained was filtered and recrystallized from methanol.

Crystal data

$C_{17}H_{21}NO_2$	Mo $K\alpha$ radiation
$M_r = 271.35$	$\lambda = 0.71073$ Å
Orthorhombic	Cell parameters from 39
$P2_12_12_1$	reflections
$a = 6.9887 (2)$ Å	$\theta = 5.38–12.51°$
$b = 10.7123 (2)$ Å	$\mu = 0.077$ mm^{-1}
$c = 20.4198 (4)$ Å	$T = 293 (2)$ K
$V = 1528.73 (6)$ Å^3	Rectangular prism
$Z = 4$	$0.45 \times 0.38 \times 0.29$ mm
$D_x = 1.179$ Mg m^{-3}	Pale yellow
D_m not measured	

Data collection

Siemens $P4$ diffractometer	$\theta_{max} = 27.49°$
$\omega/2\theta$ scans	$h = -8 \rightarrow 9$
Absorption correction: none	$k = -13 \rightarrow 13$
9982 measured reflections	$l = -20 \rightarrow 26$
2024 independent reflections	3 standard reflections
1501 reflections with	every 97 reflections
$I > 2\sigma(I)$	frequency: 60 min
$R_{int} = 0.047$	intensity decay: $<3\%$

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} < 0.001$
$R[F^2 > 2\sigma(F^2)] = 0.051$	$\Delta\rho_{max} = 0.14$ e Å^{-3}
$wR(F^2) = 0.118$	$\Delta\rho_{min} = -0.17$ e Å^{-3}
$S = 1.094$	Extinction correction: none
2024 reflections	Scattering factors from
181 parameters	*International Tables for*
H atoms constrained	*Crystallography* (Vol. C)
$w = 1/[\sigma^2(F_o^2) + (0.0394P)^2$	
$+ 0.3749P]$	
where $P = (F_o^2 + 2F_c^2)/3$	

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

C1—C10	1.502 (4)	C6—C6a	1.490 (4)
C4—C4a	1.488 (4)	C6a—C9a	1.400 (4)
C4a—C5	1.396 (4)	C9—C9a	1.503 (4)
C5—C6a	1.383 (4)	C9a—N10	1.347 (3)
C6a—C5—C4a	119.5 (3)	C9a—N10—C10	118.1 (2)
C12—C2—C3—C4	64.8 (3)	C6—C7—C8—C14	-63.6 (3)
C11—C2—C3—C4	-175.7 (2)	C6—C7—C8—C13	176.7 (3)

All H atoms were geometrically fixed and allowed to ride on the corresponding non-H atoms, with C—H = 0.96–0.97 Å, and $U_{iso} = 1.5U_{eq}$ of the attached C atom for methyl-H atoms and $1.2U_{eq}$ for other H atoms. Because of the presence of only very weak anomalous scatterers such as O, N and C, the absolute structure cannot be determined reliably [the $F(hkl)$ and $F(\bar{h}kl)$ reflections have been merged using the command *MERG3* in *SHELXL97* (Sheldrick, 1997a)].

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997b). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a). Molecular graphics: *ZORTEP* (Zsolnai, 1997). Software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1983b, 1995).

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