C8—H8A···O2^{iii}	1.01 (3)	2.48 (4)	3.481 (4)	172 (5)
C8—H8B···O1^{iv}	0.99 (4)	2.54 (3)	3.490 (6)	160 (3)
N2—H1N2···O2^{iv}	0.74 (5)	2.62 (4)	3.209 (5)	138 (2)

Symmetry codes: (i) $y, z, x-1$; (ii) $-x, -1-y, -1-z$; (iii) $1-y, 1-z, 1-x$; (iv) $-y, -z, -x$.

The title structure was solved by direct methods and refined by full-matrix least squares. 11 H atoms were located from a difference Fourier map and refined isotropically; the remaining three not found in the map were geometrically fixed and allowed to ride on their parent atoms. At this stage, the refinement converged to an R value of 0.054 (wR = 0.184). The s.u.'s of the structural parameters were high and the difference map showed peaks (0.30 to 0.45 e Å^{-3}) of almost equal interval (around 1 to 1.1 Å) on the threefold axis and origin. Refinement based on a disordered solvent model led to unstable refinement with very high displacement parameters. A search for solvent-accessible voids in the crystal using *PLATON* (Spek, 1990) showed a potential solvent volume of 309.9 Å^3 and subsequent application of *SQUEEZE* procedures (van der Sluis & Spek, 1990) showed only one relevant void (or channel) with a solvent-accessible volume of 206 Å^3. The number of electrons found in that channel is 12 and the estimated volume per atom is 143 Å^3. This indicates that the void is only partially occupied and that the original contents had probably disappeared by the time the crystal was used for data collection, without collapsing the structure. Further refinement of the structure with solvent-free reflection data obtained from the above procedure converged to an R value of 0.045 (wR = 0.125) and the accuracy of structural parameters was found to have improved. The final F_o–F_c listing was generated using the *CALC FCF* option in *PLATON*.

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXL*93 and *PLATON*.

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

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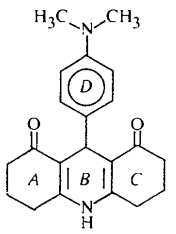
Abstract

The title compound, $C_{21}H_{24}N_2O_2$, contains an acridine moiety and a dimethylaminophenyl ring system. The side rings adopt half-chair conformations. The acridine chromophore is perpendicular to the substituted phenyl ring.

Comment

Acridines are potent DNA intercalators, with very sensitive and characteristic fluorescent properties which respond to changes in the microenvironment (Lerman, 1961). Acridines are useful for tagging molecules of interest, but their application is currently limited to covalent modification of small oligonucleotides, as no technology currently exists to attach them to larger DNAs and proteins (Selladurai *et al.*, 1990). Acridine dyes reacting with nucleic acids have received increasing interest as mutagens in micro-organisms (Sivaraman *et al.*, 1996), but relatively little attention has been given to acridine-induced mutation in higher plants, except for barley. Apart from the above, acridinediones are used as antibacterial agents for wound therapy (Acheson, 1956) and as antitumour drugs (Hempel *et al.*, 1979). In view of the above interest, we decided to analyse the conformation of the acridine moiety with respect to a dimethylaminophenyl ring system.

The *ZORTEP* (Zsolnai, 1997) plot of the title molecule, (I), with the atomic numbering scheme is shown in Fig. 1. The acridine moiety is not planar: the central



(I)

ring, *B*, adopts a boat conformation, whereas the side rings, *A* and *C*, assume half-chair conformations. The dihedral angle between rings *A* (C1–C6) and *C* (C8–C13) is 12.1 (1)°. There is considerable buckling of the acridine nucleus. The sums of the bond angles around N1 and N2 (358.6 and 359.2°) indicate sp^2 hybridization (Sivaraman *et al.*, 1996). The acridine moiety is perpendicular [91.3 (1)°] to the plane of the phenyl ring, *D*. The total puckering amplitudes for rings *A*, *B* and *C* are Q_T = 0.477 (3), 0.260 (3) and 0.459 (3) Å, respectively (Cremer & Pople, 1975).

In addition to van der Waals interactions, the structure is also stabilized by N—H···O hydrogen bonds [N···O 2.907 (3) Å] in the solid-state conformation.

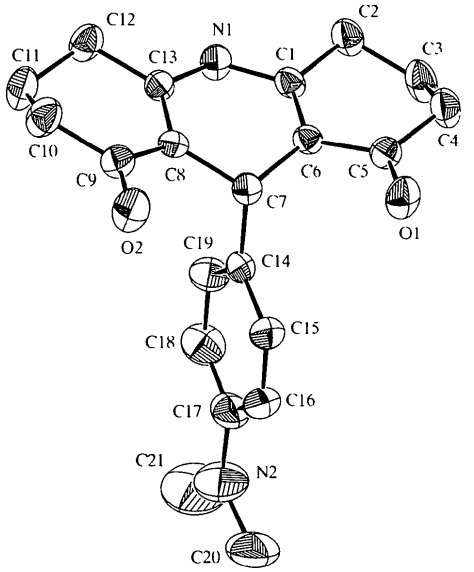


Fig. 1. *ZORTEP* (Zsolnai, 1997) diagram showing the atom-numbering scheme of the title molecule, with displacement ellipsoids at the 50% probability level.

Experimental

Small transparent pale-yellow crystals of the title compound were obtained by recrystallization from an acetone/ethanol mixture (Murugan & Ramakrishnan, 1997).

Crystal data

$C_{21}H_{24}N_2O_2$
M_r = 336.42
Monoclinic
$P2_1/n$
a = 8.574 (1) Å
b = 30.570 (9) Å
c = 7.0473 (8) Å
β = 100.53 (1)°
V = 1816.0 (6) Å^3
Z = 4
D_x = 1.230 Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 5–20°
μ = 0.079 mm^{-1}
T = 293 (2) K
Rectangular
0.35 × 0.30 × 0.27 mm
Transparent pale yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3882 measured reflections
3563 independent reflections
1820 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 25.96°
h = 0 → 10
k = 0 → 37
l = −8 → 8
3 standard reflections
every 200 reflections
intensity decay: <2%

Refinement

Refinement on F^2
$R(F)$ = 0.049
$wR(F^2)$ = 0.291
S = 1.091
3563 reflections
226 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

O1—C5	1.232 (4)	N2—C17	1.387 (6)
O2—C9	1.226 (4)	N2—C20	1.448 (8)
N1—C1	1.373 (3)	N2—C21	1.415 (8)
N1—C13	1.377 (4)		
C1—N1—C13	122.2 (3)	O2—C9—C8	121.2 (3)
C17—N2—C20	119.3 (4)	O2—C9—C10	120.4 (3)
C17—N2—C21	121.6 (4)	N1—C13—C8	119.5 (3)
C20—N2—C21	118.3 (5)	N1—C13—C12	116.9 (3)
N1—C1—C6	119.8 (3)	N2—C17—C16	121.9 (4)
O1—C5—C4	120.2 (3)	N2—C17—C18	121.4 (4)
O1—C5—C6	121.4 (3)		
C13—N1—C1—C2	−164.4 (3)	N1—C1—C6—C5	−169.6 (3)
C13—N1—C1—C6	12.5 (4)	N1—C1—C6—C7	8.7 (4)
C1—N1—C13—C8	−13.9 (4)	O1—C5—C6—C1	171.2 (3)
C1—N1—C13—C12	163.5 (3)	O1—C5—C6—C7	−7.2 (4)
C20—N2—C17—C16	−5.5 (6)	C7—C8—C9—O2	2.5 (4)
C20—N2—C17—C18	177.0 (4)	C7—C8—C13—N1	−6.2 (4)
C21—N2—C17—C16	−174.4 (5)	C9—C8—C13—N1	170.9 (3)
C21—N2—C17—C18	8.1 (7)	O2—C9—C10—C11	−155.2 (3)
N1—C1—C2—C3	−160.4 (3)	N2—C17—C18—C19	177.7 (4)

The structure was solved by direct methods. The H atoms were placed at calculated positions and refined as riding using *SHELXL*93 (Sheldrick, 1993).

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *CAD*-4 *Software*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93. Molecular graphics: *ZORTEP* (Zsolnai, 1997). Software used to prepare material for publication: *PARST* (Nardelli, 1983, 1995) and *PARSTCIF* (Nardelli, 1991).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1361). Services for accessing these data are described at the back of the journal.

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1,3,4,8-Tetraphenyl-7-oxa-1,2-diazaspiro-[4.4]nona-2,8-dien-6-one†

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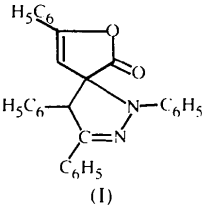
Abstract

In the title compound, $C_{30}H_{22}N_2O_2$, the pyrazoline ring conformation deviates slightly from an ideal envelope conformation. It is substituted by three planar phenyl rings, inclined to it at angles of 89.8 (1), 14.1 (1) and 7.3 (1)°. The substituted phenyl rings are in equatorial and axial positions with respect to the pyrazoline ring. The lactone ring is essentially planar, but the keto group O atom deviates from the least-squares plane through the ring atoms by −0.130 (1) Å. The lactone ring has one phenyl substituent, which adopts an axial position and is inclined at an angle of 11.3 (1)°. The dihedral angle between the pyrazoline and lactone rings is 87.6 (1)°. The crystal structure is stabilized by weak intermolecular hydrogen bonds.

Comment

Pyrazoline compounds have many important pharmacological properties, finding use as, for example, anti-inflammatory agents, herbicides, analgetic agents, antibacterial agents, moderate non-toxic local anaesthetics and antifungal agents (Gusar *et al.*, 1995; Sharma *et al.*, 1993; Ankhiwala & Hathi, 1996). They are also effective scintillation solutes and lubricating oil antioxidants (Beher *et al.*, 1967). Lactones serve as starting materials for the synthesis of natural products (Rao, 1976). The lactone derivatives α- and β-angelica lactones are cardiovascular agents, whereas the γ-lactone is used in the perfume industry (Rao, 1964; Jenkins & Hartung, 1950). Furthermore, lactones find use in the preparation of pyrrolidone (Lakhrissi & Chapleur, 1994). In view of the above importance of such compounds and to confirm the structure assignments and relative stereochemistries, a structure determination of the title spiro pyrazoline–lactone compound, (I), was carried out.



(I)

In the pyrazoline–lactone ring system (Fig. 1), the pyrazoline ring deviates slightly from an ideal envelope conformation [Q_2 = 0.238 (2) Å and Φ_2 = 3.2 (4)°; Cremer & Pople, 1975]. This is also confirmed by the sum of the bond angles within the pyrazoline ring [534.1 (11)°]. The pyrazoline and lactone rings are nearly orthogonal to each other [87.6 (1)°]. The bond lengths and angles of the pyrazoline ring differ slightly from the values found for acetone 4,4-dimethyl-5-oxo-2-pyrazolin-3-ylhydrazone (Meyers *et al.*, 1996). The three phenyl rings, *A*, *C* and *D*, attached to the pyrazoline ring at C7, N4 and C6, subtend angles of 89.8 (1), 14.1 (1) and 7.3 (1)°, respectively. Phenyl rings *A* and *C* are disposed equatorially, while ring *D* is in an axial position with respect to the pyrazoline ring. The planar lactone ring is inclined at an angle of 11.3 (1)° to the substituted phenyl ring *B*, which adopts an axial

† DCB contribution No. 882.