$S = 1.037$
2495 reflections
151 parameters
Methyl H atoms refined as rigid groups, others riding
$w = 1/[\sigma^2(F_o^2) + (0.0463P)^2 + 0.5608P]$
where $P = (F_o^2 + 2F_c^2)/3$

Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Si1	0.53781 (4)	0.09933 (4)	0.05492 (2)	0.02092 (13)
N1	0.44462 (11)	0.09563 (12)	−0.04499 (6)	0.0201 (3)
C1	0.43712 (16)	0.16566 (18)	0.13365 (8)	0.0319 (3)
C2	0.70274 (16)	0.20907 (17)	0.07048 (9)	0.0321 (3)
C11	0.38136 (14)	0.21337 (14)	−0.09760 (7)	0.0209 (3)
C12	0.24128 (14)	0.25562 (15)	−0.09808 (8)	0.0244 (3)
C13	0.18406 (16)	0.37400 (17)	−0.14867 (9)	0.0309 (3)
C14	0.26048 (16)	0.45051 (18)	−0.19832 (9)	0.0333 (3)
C15	0.39685 (16)	0.40832 (16)	−0.19810 (9)	0.0296 (3)
C16	0.45944 (14)	0.29083 (15)	−0.14927 (8)	0.0238 (3)
C121	0.14866 (14)	0.17733 (16)	−0.04575 (8)	0.0255 (3)
C122	0.02672 (16)	0.09664 (18)	−0.09988 (9)	0.0328 (3)
C123	0.09385 (16)	0.28845 (18)	0.01226 (9)	0.0335 (3)
C161	0.61026 (15)	0.25177 (16)	−0.15315 (9)	0.0277 (3)
C162	0.70550 (17)	0.38758 (18)	−0.12837 (11)	0.0391 (4)
C163	0.62561 (19)	0.1965 (2)	−0.23888 (10)	0.0431 (4)

Table 2. *Selected geometric parameters (Å, °)*

Si1—N1$^{\rm i}$	1.7435 (11)	Si1—C1	1.8581 (15)
Si1—N1	1.7479 (12)	N1—C11	1.4286 (16)
Si1—C2	1.8559 (16)		
N1$^{\rm i}$—Si1—N1	86.28 (5)	C11—N1—Si1$^{\rm i}$	134.60 (9)
N1$^{\rm i}$—Si1—C2	115.71 (6)	C11—N1—Si1	131.67 (9)
N1—Si1—C2	115.92 (6)	Si1$^{\rm i}$—N1—Si1	93.72 (5)
N1$^{\rm i}$—Si1—C1	116.80 (6)	C12—C11—N1	120.69 (12)
N1—Si1—C1	114.98 (6)	C16—C11—N1	120.18 (12)
C2—Si1—C1	106.59 (7)		

Symmetry code: (i) $1 - x, -y, -z$.

Data were collected according to the learnt-profile method (Clegg, 1983). Corrections for background, decay, Lorentz and polarization factors were included in the data reduction using *REDU*4 (Stoe & Cie, 1990*b*). The structure was solved by direct methods and refined by least-squares techniques. Non-H atoms were refined with anisotropic displacement parameters. The positions of the H atoms were refined using a riding model. Most calculations were conducted on an IBM *RS*6000.

Data collection: *DIF*4 (Stoe & Cie, 1990*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1990*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1992). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1138). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3,3,6,6-Tetramethyl-9-(2-nitrophenyl)-3,4,5,6,9,10-hexahydroacridine-1,8(2*H*,7*H*)-dione

Marco Brito-Arias,[a] Guadalupe Ramirez,[a] Ruth E. Rivas,[a] Elies Molins[b] and Waldemar Maniukiewicz[b]

[a]*Unidad Profesional Interdisciplinaria de Biotecnología-IPN, Av. Acueducto s/n Col. Barrio la Laguna Ticomán CP 07340, México DF, México, and* [b]*Institut de Ciència de Materials de Barcelona, Campus de la UAB, 08193 Bellaterra, Spain. E-mail: waldemar@icmab.es*

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Abstract

The title compound, $C_{23}H_{26}N_2O_4$, consists of a partially hydrogenated acridine moiety with one 2-nitrophenyl substituent on the central ring. The acridine nucleus suf-

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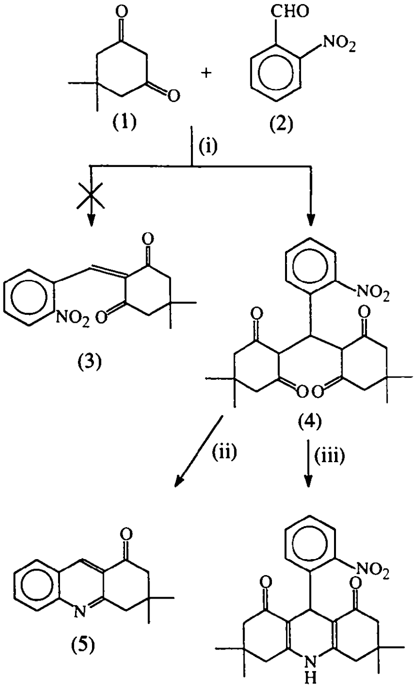
fers considerable deviations from planarity. The central ring is in a distorted boat conformation, while the two outer rings adopt almost ideal envelope conformations. The phenyl ring of the 2-nitrophenyl substituent is perpendicular to the acridine ring system. Molecules are held together by C—H···O hydrogen bonds.

Comment

Acetylcholinesterase inhibitors continue to be the therapeutic strategy of choice for patients with Alzheimer's disease (Shutske *et al.*, 1989; Schipchandler & Mattingly, 1990; Reisch & Gunaherath, 1993). Among the variety of acetylcholinesterase inhibitors, the acridine derivatives are one of the most efficient drugs, since they display potent action with high tolerance. In a previous report, we presented the synthesis and X-ray analysis of the potential anti-Alzheimer drug 3,4-dihydro-3,3-dimethyl-1(2*H*)-acridone (Martínez, Espinosa-Pérez & Brito-Arias, 1995) and as a continuation of these studies we report here the synthesis and X-ray structure analysis of the related compound 3,3,6,6-tetramethyl-9-(2-nitrophenyl)-3,4,5,6,9,10-hexahydroacridine-1,8(2*H*,7*H*)-dione, (6).



Reagents and conditions: (i) KOH/EtOH-H_2O, reflux, 2 h; (ii) Fe-AcOH/EtOH, RT, 5 h; (iii) NaN_3-HCl/THF, RT, 3 h.

A perspective view of the title molecule, together with the atomic numbering scheme, is shown in Fig. 1. Bond distances and angles of the acridine skeleton are in agreement with those of related acridine derivatives (Bundule *et al.*, 1980; Selladurai, Subramanian & Natarajan, 1989; Selladurai, Subramanian & Ramakrishnan, 1990; Sivaraman, Subramanian, Velmurugan, Subramanian & Ramakrishnan, 1994; Sivaraman, Subramanian, Velmurugan, Subramanian & Shanmugasundram, 1996). Both outer six-membered rings of the acridine moiety adopt almost ideal envelope conformations, with puckering parameters (Cremer & Pople, 1975) Q = 0.462 (2) and 0.461 (2) Å, θ = 123.5 (2) and 123.8 (2)°, and φ = 300.6 (3) and 298.5 (3)°. In contrast, the central ring is less regular. Its conformation may be best described as a distorted boat, with puckering parameters Q = 0.094 (1) Å, θ = 103.8 (1)° and φ = 352.4 (1)°. The planar phenyl ring of the 2-nitrophenyl substituent is perpendicular to the acridine moiety, with the dihedral angle between their least-squares planes being 89.04 (7)°. The mean C_{sp^2}—C_{sp^2} bond length within this ring is 1.386 (2) Å. The intra- and intermolecular C—H···O interactions (Desiraju, 1991) that contribute to the stabilization of the molecular and crystal structure are summarized in Table 3.

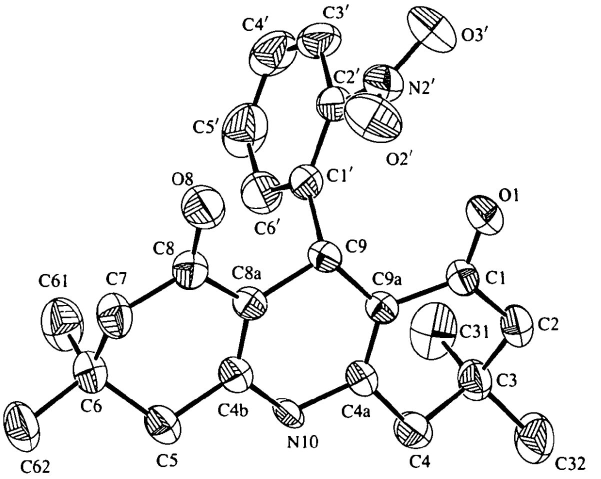


Fig. 1. The structure and labelling scheme for compound (6). Displacement ellipsoids are drawn at the 50% probability level.

Experimental

The synthetic approach used consisted of the initial condensation between dimedone, (1), and 2-nitrobenzaldehyde, (2), in a basic medium. Unexpectedly, the condensation product was not compound (3) as was reported previously (Martínez *et al.*, 1995), but the tetraketone (4). This finding was supported not only by means of ^1H NMR spectroscopy and MS spectrometry, but also by similar dimeric structures found in the literature (Greenhill, 1977; Shanmugasundram, Murugan, Ramakrishnan, Srividya & Ramamurthy, 1996). It may be noted that compound (4) is a common starting material for the preparation of compounds (5) and (6). Conversion of compound (4) to compound (5) involves the loss of one dimedone moiety as a consequence of the aromatization of the acridone ring. Compound (5) is thus readily prepared from compound (4) under reducing conditions (Fe/AcOH), while compound (6) is obtained in 40% yield when compound (4) is treated with NaN_3/HCl. For compound (6), ^1H NMR ($CDCl_3$, 293 K): δ 1.0 (6H, *s*, CH_3), 1.1 (6H, *s*, CH_3), 2.1 (4H, *s*, CH_2), 2.4 (4H, *s*, CH_2), 5.4 (1H, *s*, CH), 7.1–7.7 p.p.m. (4H, *s*, aromatic). IR ($CHCl_3$): ν 1357 (NO_2), 1529 (NO_2), 1624 cm^{-1} (CO). MS *m*/*z*: 395 (M^+), 273.

Crystal data

$C_{23}H_{26}N_2O_4$
$M_r = 394.46$
Monoclinic
$C2/c$
$a = 26.542\ (4)$ Å
$b = 10.089\ (1)$ Å
$c = 20.922\ (3)$ Å
$\beta = 133.07\ (2)°$
$V = 4092.8\ (8)$ Å^3
$Z = 8$
$D_x = 1.280$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 16.04$–$19.42°$
$\mu = 0.088$ mm^{-1}
$T = 293\ (2)$ K
Irregular
0.55 × 0.48 × 0.36 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: empirical using azimuthal (ψ) scans (North, Phillips & Mathews, 1968)
$T_{min} = 0.90$, $T_{max} = 0.99$
7010 measured reflections
5957 independent reflections

3965 observed reflections $[I > 2\sigma(I)]$
$R_{int} = 0.018$
$\theta_{max} = 29.98°$
$h = -37 \rightarrow 27$
$k = 0 \rightarrow 14$
$l = 0 \rightarrow 29$
3 standard reflections monitored every 100 reflections
intensity decay: 0.9%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0625$
$wR(F^2) = 0.2188$
$S = 0.996$
5076 reflections
266 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0972P)^2 + 3.6916P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = -0.025$
$\Delta\rho_{max} = 0.524$ e Å^{-3}
$\Delta\rho_{min} = -0.537$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	U_{eq}
O1	0.27280 (5)	0.79254 (12)	−0.13038 (6)	0.0572 (3)
O2′	0.15884 (6)	0.5656 (2)	−0.19088 (8)	0.0775 (5)
O3′	0.16826 (8)	0.5629 (2)	−0.28462 (7)	0.0910 (5)
O8	0.21588 (5)	0.40519 (13)	−0.01950 (7)	0.0623 (3)
N2′	0.19143 (7)	0.54248 (14)	−0.21117 (7)	0.0560 (4)
N10	0.40824 (5)	0.68628 (11)	0.16619 (5)	0.0372 (3)
C1	0.32343 (6)	0.81284 (14)	−0.05321 (7)	0.0403 (3)
C2	0.37179 (7)	0.9261 (2)	−0.02492 (9)	0.0495 (4)
C3	0.44488 (6)	0.90732 (15)	0.06343 (8)	0.0446 (3)
C31	0.48206 (8)	0.7981 (2)	0.05830 (12)	0.0721 (5)
C32	0.48544 (8)	1.0370 (2)	0.09296 (11)	0.0666 (5)
C4	0.44040 (7)	0.8688 (2)	0.12999 (8)	0.0498 (4)
C4a	0.39132 (6)	0.75805 (14)	0.09809 (7)	0.0396 (3)
C4b	0.36573 (6)	0.58449 (14)	0.14760 (7)	0.0418 (3)
C5	0.38781 (7)	0.5265 (2)	0.22900 (8)	0.0525 (4)
C6	0.36542 (6)	0.3810 (2)	0.21646 (8)	0.0465 (3)
C61	0.40640 (8)	0.2917 (2)	0.20701 (11)	0.0674 (5)
C62	0.37747 (8)	0.3357 (2)	0.29575 (10)	0.0633 (5)
C7	0.28885 (7)	0.3733 (2)	0.13395 (9)	0.0511 (4)
C8	0.26834 (6)	0.43757 (15)	0.05385 (8)	0.0435 (3)
C8a	0.31186 (6)	0.54523 (13)	0.06640 (7)	0.0373 (3)
C9	0.29276 (5)	0.60729 (13)	−0.01366 (7)	0.0353 (3)
C9a	0.33771 (5)	0.72772 (13)	0.01457 (7)	0.0354 (3)
C1′	0.30496 (6)	0.50982 (13)	−0.05780 (7)	0.0392 (3)
C2′	0.26017 (6)	0.48387 (14)	−0.14736 (8)	0.0450 (3)
C3′	0.27833 (8)	0.4007 (2)	−0.18217 (9)	0.0615 (4)
C4′	0.34165 (8)	0.3398 (2)	−0.12745 (11)	0.0715 (4)
C5′	0.38741 (8)	0.3644 (2)	−0.03918 (12)	0.0705 (5)
C6′	0.36894 (7)	0.4484 (2)	−0.00549 (9)	0.0531 (4)

Table 2. *Selected geometric parameters* (Å, °)

O1—C1	1.2212 (14)	C4—C4a	1.484 (2)
O2′—N2′	1.218 (2)	C4a—C9a	1.333 (2)
O3′—N2′	1.221 (2)	C4b—C8a	1.330 (2)
O8—C8	1.217 (2)	C4b—C5	1.491 (2)
N2′—C2′	1.461 (2)	C5—C6	1.538 (2)
N10—C4b	1.371 (2)	C6—C61	1.526 (3)
N10—C4a	1.372 (2)	C6—C7	1.525 (2)
C1—C9a	1.469 (2)	C6—C62	1.529 (2)
C1—C2	1.505 (2)	C7—C8	1.510 (2)
C2—C3	1.523 (2)	C8—C8a	1.473 (2)
C3—C31	1.531 (3)	C8a—C9	1.515 (2)
C3—C4	1.527 (2)	C9a—C9	1.512 (2)
C3—C32	1.531 (2)	C9—C1′	1.527 (2)
O2′—N2′—O3′	122.60 (15)	C61—C6—C7	109.90 (13)
O2′—N2′—C2′	120.00 (13)	C61—C6—C62	108.96 (14)
O3′—N2′—C2′	117.4 (2)	C7—C6—C62	110.04 (13)
C4b—N10—C4a	118.58 (9)	C61—C6—C5	111.07 (14)
O1—C1—C9a	120.25 (13)	C7—C6—C5	107.61 (12)
O1—C1—C2	121.34 (13)	C62—C6—C5	109.24 (13)
C9a—C1—C2	118.40 (10)	C8—C7—C6	115.25 (13)
C1—C2—C3	114.86 (12)	O8—C8—C8a	120.34 (13)
C2—C3—C31	110.63 (13)	O8—C8—C7	121.28 (14)
C2—C3—C4	108.10 (12)	C8a—C8—C7	118.32 (11)
C31—C3—C4	109.64 (13)	C4b—C8a—C8	118.39 (12)
C2—C3—C32	110.22 (12)	C4b—C8a—C9	123.04 (12)
C31—C3—C32	109.50 (14)	C8—C8a—C9	118.57 (10)
C4—C3—C32	108.72 (13)	C4a—C9a—C1	118.17 (12)
C4a—C4—C3	111.88 (11)	C4a—C9a—C9	123.23 (12)
C9a—C4a—N10	122.66 (12)	C1—C9a—C9	118.56 (9)
C9a—C4a—C4	125.87 (12)	C9a—C9—C8a	108.73 (9)
N10—C4a—C4	111.47 (10)	C9a—C9—C1′	108.49 (11)
C8a—C4b—N10	122.96 (12)	C8a—C9—C1′	110.69 (11)
C8a—C4b—C5	125.67 (13)	C6′—C1′—C9	117.45 (11)
N10—C4b—C5	111.37 (10)	C2′—C1′—C9	126.45 (11)
C4b—C5—C6	112.02 (12)		

Table 3. *Hydrogen-bonding geometry* (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3′—H3′···O1^{i}	0.93	2.51	3.338 (2)	147.8 (2)
C4′—H4′···O3′^{ii}	0.93	2.45	3.255 (3)	144.6 (3)
C9—H9···O2′	0.98	2.21	2.926 (1)	128.8 (1)

Symmetry code: (i) $\frac{1}{2} - x, y - \frac{1}{2}, -\frac{1}{2} - z$.

The structure of the title compound was solved by direct methods. Refinement was based on full-matrix least-squares methods, with anisotropic displacement parameters for all non-

H atoms and isotropic displacement parameters for H atoms. All H atoms were included at calculated positions and refined using a riding model, each with an isotropic displacement parameter equal to 1.5 times U_{eq} of the attached C or N atom.

Data collection: *CAD*-4 *Software*. Cell refinement: *CAD*-4 *Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *PLATON* (Spek, 1996*a*) and *PLUTON* (Spek, 1996*b*). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1390). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Two C-Unsubstituted Enaminals

ALEXANDER J. BLAKE,[a,b]* HAMISH MCNAB,[b] LILIAN C. MONAHAN,[b] SIMON PARSONS[b] AND ELIZABETH STEVENSON[b]

[a]*Department of Chemistry, The University of Nottingham, University Park, Nottingham NG7 2RD, England, and*
[b]*Department of Chemistry, The University of Edinburgh, West Mains Road, Edinburgh EH9 3JJ, Scotland. E-mail: a.j.blake@nott.ac.uk*

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Abstract

In both 3-(*N*,*N*-diisopropylamino)-2-propenal, $C_9H_{17}NO$, (3), and 3-(1,2,3,4-tetrahydro-1-quinolinyl)-2-propenal, $C_{12}H_{13}NO$, (4), the entire enaminal system (O1—C1—C2—C3—N4) is approximately planar. The angles around the N atoms in (3) and (4) sum to values near 360°, indicating planarity in both molecules. One of the two crystallographically independent molecules of (3) exhibits disorder in its isopropyl groups.

Comment

The enaminone structural unit (1) (Greenhill, 1977) is a classic example of a 'push–pull' substituted alkene, yet surprisingly little crystallographic information is available for simple derivatives of this conjugated system. Most enaminone structures which have been reported involve derivatives which have substituents either in the 1- (Carugo, Castellani & Rizzi, 1990) or 2-position (Mague, De & Krogstad, 1995; Niederhauser, Sterchi & Neuenschwander, 1976). Structures of 1,2-disubstituted (Arriortua *et al.*, 1992; Peralta *et al.*, 1995) and 1,3-disubstituted (Emsley, Freeman, Parker, Kuroda & Overill, 1987) derivatives are known, as are those in which the system is incorporated in a heterocyclic ring (*e.g.* Hickson *et al.*, 1986; Blake, McNab & Monahan, 1988). The only published crystal structure of a C-unsubstituted derivative, however, is that of the β-naphthyl compound (2) (Chunli, Zhongheng, Heng & Youqi, 1984), but full details of this determination are not readily available. We therefore report here the structures of a typical *N*,*N*-dialkyl enaminal and an *N*-alkyl *N*-aryl derivative; simple enaminones are liquids, so for convenience we have selected the *N*,*N*-diisopropyl compound (3) and the tetrahydroquinoline structure (4). In order to avoid possible complications due to hydrogen bonding at this stage, we have specifically focused on *N*,*N*-disubstituted examples for these determinations.

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