

294 parameters

All H atoms refined

$$w = 1/[\sigma^2(F_o^2) + (0.0347P)^2 + 0.4519P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} = 0.001$$

$$\Delta\rho_{\max} = 0.358 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.205 \text{ e } \text{\AA}^{-3}$$

For both compounds, data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *CARESS* (Broach, 1978); program(s) used to solve structures: *SHELXTL* (Sheldrick 1994); program(s) used to refine structures: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SX1032). Services for accessing these data are described at the back of the journal.

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Scattering factors from

*International Tables for Crystallography* (Vol. C)

Absolute configuration:

Flack (1983)

Flack parameter = -0.02 (4)

*Acta Cryst.* (1997). **C53**, 1512–1514

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

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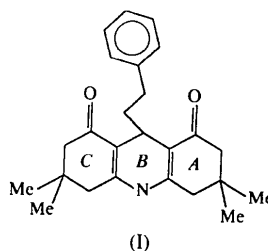
(Received 19 December 1996; accepted 9 May 1997)

## Abstract

In the title compound, C<sub>25</sub>H<sub>31</sub>NO<sub>2</sub>, the phenyl ring is approximately perpendicular to the acridine moiety, whose planarity is lost as a consequence of lack of  $\pi$ -conjugation along the ring system. Intermolecular hydrogen bonding and van der Waals interactions stabilize the molecules in the crystal.

## Comment

The fluorescence nature and the DNA binding tendency of acridinediones drew our attention towards the crystallographic study of these compounds (Sivaraman, Subramanian, Velmurugan, Subramanian & Shanmugasundaram, 1996). This work is part of our study to correlate their DNA binding ability with their structure. The observed bond angles around the N atom in



the heterocyclic ring of the title compound, (I), sum to 360° indicating  $sp^2$  hybridization. Due to the N-atom lone pair delocalization, the bond lengths N10—C1a and N10—C8a are shorter than the C—N single bond, in agreement with the value of 1.355(14) Å given by Allen *et al.* (1987) for the average  $C_{sp^2}$ — $N_{sp^2}$  planar bond. Atoms C9 and C17 are completely staggered to one another about the C15—C16 bond. The substitution at C9 is in an axial position. Mean-plane calculations show that the compound is folded by

† DCB contribution No. 856.

an angle of  $17.86(2)^\circ$  about the  $N10 \cdots C9$  direction and the phenyl ring makes a dihedral angle of  $103.92(4)^\circ$  with the plane through the acridine moiety. The total puckering amplitudes (Cremer & Pople, 1975) of the rings [ $Q_T = 0.476(1)$ ,  $0.298(1)$  and  $0.454(1)$  Å for rings *A*, *B* and *C*, respectively] give a quantitative evaluation of puckering, while the asymmetry parameters (Duax, Weeks & Rohrer, 1976) reveal a sofa conformation for the *A* and piperidine *B* rings, and a distorted half chair for the *C* ring. In addition to the van der Waals interactions, intermolecular hydrogen bonds of the  $N-H \cdots O$  [ $N10-H10 \cdots O1(x, y+1, z)$ :  $N10-H10$   $0.87(1)$ ,  $N10 \cdots O1$   $2.961(1)$ ,  $H10 \cdots O1$   $2.12(1)$  Å and  $N10-H10 \cdots O1$   $163(1)^\circ$ ] and  $C-H \cdots O$  types [ $C13-H13B \cdots O2(x, y+1, z)$ :  $C13-H13B$   $1.03(1)$ ,  $C13 \cdots O2$   $3.341(1)$ ,  $H13B \cdots O2$   $2.37(1)$  Å and  $C13-H13B \cdots O2$   $157(1)^\circ$ ] stabilize the system in the crystalline state.

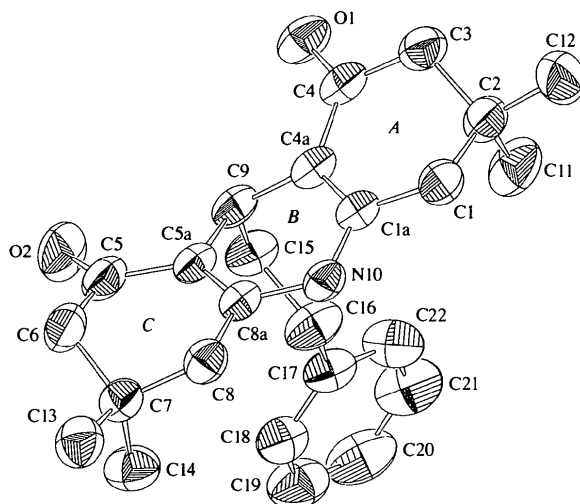


Fig. 1. The structure of the title compound showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

## Experimental

A mixture of dimedone (5 mmol) and hydrocinnamaldehyde (2.5 mmol) was refluxed in ethanol (40 ml) in the presence of ammonium hydroxide for 8 h. The separated yellow solid was filtered, dried and crystallized from ethanol.

### Crystal data

$C_{25}H_{31}NO_2$   
 $M_r = 377.51$   
 Monoclinic  
 $P2_1/c$   
 $a = 16.657(1)$  Å  
 $b = 7.351(2)$  Å  
 $c = 18.400(2)$  Å  
 $\beta = 108.30(1)^\circ$   
 $V = 2139.1(6)$  Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.172$  Mg m<sup>-3</sup>  
 $D_m$  not measured

Mo  $K\alpha$  radiation  
 $\lambda = 0.71073$  Å  
 Cell parameters from 22 reflections  
 $\theta = 8-23^\circ$   
 $\mu = 0.073$  mm<sup>-1</sup>  
 $T = 293(2)$  K  
 Rectangular  
 $0.40 \times 0.30 \times 0.15$  mm  
 Pale yellow

### Data collection

Enraf-Nonius CAD-4 diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction: none  
 4475 measured reflections  
 4320 independent reflections  
 3886 reflections with  $I > 2\sigma(I)$   
 $R_{int} = 0.059$

$\theta_{max} = 26.67^\circ$   
 $h = 0 \rightarrow 21$   
 $k = 0 \rightarrow 9$   
 $l = -22 \rightarrow 21$   
 3 standard reflections every 200 reflections  
 frequency: 60 min  
 intensity decay:  $<2\%$

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.052$   
 $wR(F^2) = 0.160$   
 $S = 1.066$   
 4301 reflections  
 378 parameters  
 All H atoms refined isotropically  
 $w = 1/[\sigma^2(F_o^2) + (0.0816P)^2 + 0.3362P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.043$   
 $\Delta\rho_{max} = 0.259$  e Å<sup>-3</sup>  
 $\Delta\rho_{min} = -0.233$  e Å<sup>-3</sup>  
 Extinction correction: *SHELXL93* (Sheldrick, 1993)  
 Extinction coefficient: 0.046(2)  
 Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters (Å, °)

O1—C4	1.2334 (11)	C5—C5a	1.4573 (14)
O2—C5	1.2207 (13)	C5—C6	1.495 (2)
C1—C1a	1.4948 (14)	C5a—C8a	1.3528 (12)
C1a—C4a	1.3578 (12)	C5a—C9	1.5035 (13)
C1a—N10	1.3725 (12)	C8—C8a	1.4931 (13)
C4—C4a	1.4464 (13)	C8a—N10	1.3818 (13)
C4a—C1a—N10	120.11 (9)	C8a—C5a—C9	121.49 (9)
C4a—C1a—C1	124.08 (8)	C5—C5a—C9	119.04 (8)
N10—C1a—C1	115.78 (7)	C5a—C8a—N10	119.61 (8)
C1a—C4a—C4	119.49 (9)	C5a—C8a—C8	124.53 (9)
C1a—C4a—C9	120.57 (8)	N10—C8a—C8	115.79 (8)
C4—C4a—C9	119.94 (8)	C1a—N10—C8a	120.97 (7)
C8a—C5a—C5	119.44 (9)		
C5—C5a—C9—C15	-79.83 (10)	C9—C15—C16—C17	-179.72 (10)
C1a—C4a—C9—C15	-96.32 (9)		

Data collection: *SDP* (Frenz, 1978). Cell refinement: *SDP*. Data reduction: *SDP*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *SHELXL93* and *PARST* (Nardelli, 1983).

KG and DV thank UGC (India) for providing funds.

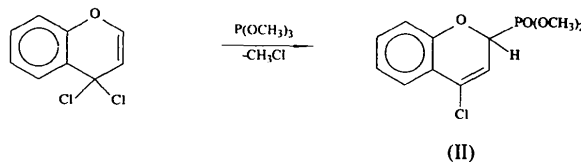
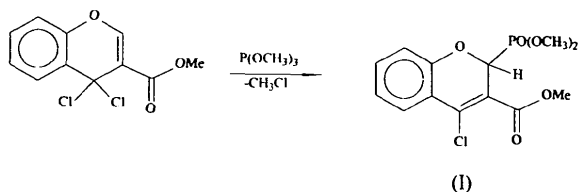
Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1284). Services for accessing these data are described at the back of the journal.

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show biological activity (Farkas, Kallay, Gabor & Wagner, 1982; Gabor, 1988; Yudelevich, Komarov & Ionin, 1985). The title compounds exhibit either a myocardial nutritional circulation effect in rabbits and a spasmolytic effect on isolated rabbit intestine (Kostka, Modranka, Szadowska, Graczyk & Orszulak, 1994) or are expected to exhibit spasmolytic properties.



*Acta Cryst.* (1997). C53, 1514–1516

## 2H-1-Benzopyrans. II. 4-Chloro-3-methoxy-carbonyl-2-dimethoxyphosphoryl-2H-1-benzopyran, (I), and 4-Chloro-2-dimethoxyphosphoryl-2H-1-benzopyran, (II)†

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### Abstract

The pyranoid rings are in a half-boat conformation in compound (I), C<sub>13</sub>H<sub>14</sub>ClO<sub>6</sub>P, and in a deformed half-boat-towards-half-chair conformation in compound (II), C<sub>11</sub>H<sub>12</sub>ClO<sub>4</sub>P. Fusion with the pyranoid ring has no significant influence on the planarity of the benzene ring in either compound. In both structures, the dimethoxyphosphoryl group is attached axially. The presence of the methoxycarbonyl substituent in (I) seems responsible for the disorder observed in the dimethoxyphosphoryl group.

### Comment

This paper is the continuation of the structure determinations of new derivatives of benzopyran (Olszak *et al.*, 1994). It is known that 2H-1-benzopyran derivatives

† Alternative names: methyl 4-chloro-2-dimethoxyphosphoryl-2H-1-benzopyran-3-carboxylate, (I), and dimethyl (4-chloro-2H-1-benzopyran-2-yl)phosphonate, (II).

The condensed ring system in (I), excepting atom C1, is almost planar. The best plane calculated through the benzene ring and that through the five atoms of the pyranoid ring form a dihedral angle of 0.50 (7)°. The benzene ring is planar to within experimental error. The pyranoid ring has a half-boat conformation. The puckering parameters (Cremer & Pople, 1975) corresponding to the sequence O1—C1—C2—C3—C9—C8 are  $Q = 0.364$  (2) Å,  $\varphi = 41.8$  (4)° and  $\theta_2 = 64.6$  (4)°, with C1 at the apex. A pseudosymmetry twofold axis runs through the midpoints of the O1—C1 and C3—C9 bonds [asymmetry parameter  $\Delta_2(\text{C1—O1}) = 0.039$  (1); Nardelli, 1983]. The dimethoxyphosphoryl group is attached axially to the ring with torsion angles C8—O1—C1—P10 and C3—C2—C1—P10 of 81.2 (2) and  $-89.1$  (2)°, respectively. The group is disordered, with atoms O13 and O23 split over two sites with occupation factors  $k_A = 0.709$  (7) and  $k_B = 0.291$  (7).

The pyranoid ring in compound (II) is in a half-chair conformation that is slightly deformed towards a half-boat conformation, with the C1 atom at the apex. The skeleton of the pyranoid ring itself has a twofold pseudosymmetry axis running through the midpoints of the C1—O1 and C3—C9 bonds. The puckering parameters (Cremer & Pople, 1975) corresponding to the sequence C1—C2—C3—C9—C8—O1 are  $Q = 0.194$  (2) Å,  $\varphi_2 = 151.2$  (9)° and  $\theta_2 = 116.0$  (7)°, and the asymmetry parameter (Nardelli, 1983)  $\Delta_2(\text{C1—O1})$  is 0.002 (1)°. The dimethoxyphosphoryl group is attached axially to the ring. The torsion angles C8—O1—C1—P10 and C3—C2—C1—P10 are 102.2 (2) and  $-110.6$  (3)°, respectively.

Compound (II) is devoid of a methoxycarbonyl group in position 3 in comparison with (I). This absence seems to influence the conformation of the dimethoxy-