$C_{34}H_{25}NO_3$

1-Acetyl-2a′,3′,8′,8a′-tetrahydro-3′,8′-(diphenylmethylenemethano)spiro[indoline-3,2′-2′H-oxeto[3,2-b]naphthalen]-2-one

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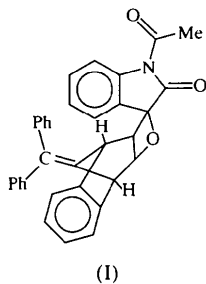
Abstract

In the crystal of the title compound, $C_{34}H_{25}NO_3$, the indole moiety is planar. The geometry of the central skeleton is distorted owing to intramolecular phenyl–phenyl repulsions. The molecules are stacked along the b axis with a distance of 3.638 (2) Å between the phenyl centroids of the inversion-related $(-x, 1 - y, -z)$ indole moiety. The planarity of the indole moiety and the orientation of the acetyl group are influenced by the C—H···O intramolecular hydrogen bonding.

Comment

Although photoinduced reactions of quinones with alkenes have received extensive investigation (Maruyama, 1988; Xu et al., 1994), no photoinduced reactions of 1-acetylindole-2,3-(2H,3H)-dione with alkenes have so far been reported. Xu et al. (1994) found that during the photolysis of chloranil with alkenes, the cycloaddition products, oxetane and/or cyclobutane derivatives, depended on the structure of the alkenes. On investigating the reactions of 1-acetylindole-2,3-(2H, 3H)-dione with alkenes under photoinduced conditions, the oxetane derivatives were obtained.

With regard to the indole moiety of the title compound, (I), the geometry is comparable with the

(I)

reported values (Ianelli et al., 1995; Seetharaman & Rajan, 1995; Chandrakantha et al., 1998; Krishna et al.,

1999). The largest discrepancy is observed for C25—C26 [1.488 (3) Å, with an average value of 1.434 (1) Å calculated for indoles in the Cambridge Structural Database (Allen et al., 1987)]. The acetyl group is twisted by 5.9 (1)° out of the plane with the indole moiety. From the torsion angles it is seen that the oxetane ring is distorted from planarity and is twisted through 87.8 (1)° with the indole moiety. After 1-acetylindole-2,3-(2H,3H)-dione was reacted with 7-diphenylmethylene-2,3-benzonorbornadiene to form the title compound, the change in the C25—O3 bond distance from former C=O double-bond distance 1.200 (3) Å [1.211 (4) Å; Zukerman-Schpector et al., 1994] to 1.457 (3) Å indicates that the C=O group is converted to an ether group. It can be seen that C24—O3 [1.465 (3) Å] and C23—C24 [1.543 (3) Å] are consistent with an ether bond and a C—C single bond, respectively. The bond lengths C1—C7 = 1.502 (3) and C7—C8 = 1.489 (3) Å are comparable with 1.51 and 1.52 Å in trans-3-(diphenylmethylene)-4-morpholinocarbonyl-5-(p-nitrophenyl)tetrahydro-2-furanone (Aupers et al., 1981). The widening of the bond angles C7—C14—C15 = 128.7 (2) and C7—C14—C22 = 134.2 (2)° may be due to the heavier substitution of the diphenyl group at C7.

The geometry of the central skeleton is distorted owing to the repulsions among C2, C9 and C27. In the title compound, the C25—C23—C22 angle [120.7 (2)°] is enlarged compared with the O3—C24—C15 angle [112.7 (2)°]. The H atoms at C15 and C22 are at equatorial orientation and the diphenylmethylene group is in a biaxial orientation with the five-membered ring (C14, C15, C22, C23 and C24). Both of the five-membered rings adopt envelope conformations and the system is cis fused.

The six-membered ring (C15, C16, C21–C24) adopts a boat conformation which makes an interplanar angle of 35.9 (2)° with the fused aromatic ring. Steric inter-
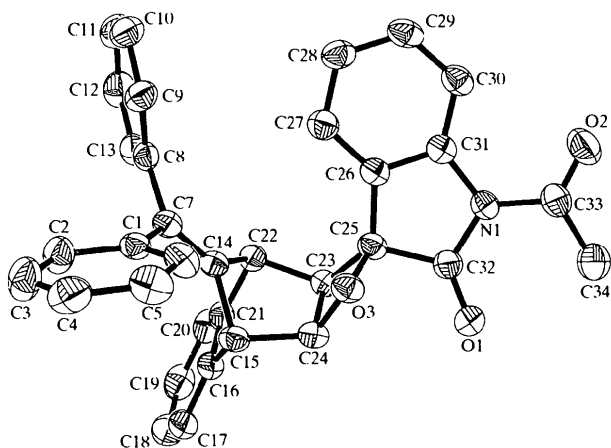

Fig. 1. A 50% probability displacement ellipsoid plot (SHELXTL; Sheldrick, 1997) of (I).

action between the phenyl rings of the dimethylene is avoided as the phenyl rings are perpendicular to each other [dihedral angle 88.3 (1)°] which is comparable with 93.8 (2)° in *cis*-1,5-diphenyl-3-(diphenylmethyl-ene)-4-morpholinocarbonyl-2-pyrrolidinone (Baydar *et al.*, 1984).

The molecules are stacked along the *b* axis with a distance of 3.638 (2) Å between the phenyl centroids of the inversion-related $(-x, 1-y, -z)$ indole moiety. The planarity of the indole moiety and the orientation of the acetyl group are influenced by the $C—H \cdots O$ intramolecular hydrogen bonding (Table 2).

Experimental

A solution of 1-acetylindole-2,3-$(2H,3H)$-dione (0.57 g, 3.0 mmol) and 7-diphenylmethylene-2,3-benzonorbornadiene (1.10 g, 3.6 mmol) in benzene (60 ml) was photolysed with light ($\geq$400 nm) under constant argon purging for 18 h. The photolysate was concentrated *in vacuo* and the residue was chromatographically separated using petroleum ether (333–363 K) and ethyl acetate (10:1) as eluents. The title compound (0.51 g, yield 34.3%) was obtained. Clear crystals suitable for X-ray diffraction analysis were obtained from the ethyl acetate solution.

Crystal data

$C_{34}H_{25}NO_3$	Mo $K\alpha$ radiation
$M_r = 495.55$	$\lambda = 0.71073$ Å
Orthorhombic	Cell parameters from 8192
$Pbca$	reflections
$a = 9.0605$ (2) Å	$\theta = 2.59–33.20°$
$b = 17.8725$ (1) Å	$\mu = 0.082$ mm^{-1}
$c = 31.4066$ (6) Å	$T = 293$ (2) K
$V = 5085.79$ (15) Å^3	Block
$Z = 8$	$0.44 \times 0.34 \times 0.20$ mm
$D_x = 1.294$ Mg m^{-3}	Colourless
D_m not measured	

Data collection

Siemens SMART CCD area-detector diffractometer	3270 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.067$
Absorption correction: none	$\theta_{max} = 27.5°$
30 518 measured reflections	$h = -11 \rightarrow 11$
5829 independent reflections	$k = -23 \rightarrow 23$
	$l = -19 \rightarrow 40$

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} < 0.001$
$R[F^2 > 2\sigma(F^2)] = 0.067$	$\Delta\rho_{max} = 0.225$ e Å^{-3}
$wR(F^2) = 0.158$	$\Delta\rho_{min} = -0.216$ e Å^{-3}
$S = 1.083$	Extinction correction: none
5829 reflections	Scattering factors from
343 parameters	*International Tables for*
H atoms constrained	*Crystallography* (Vol. C)
$w = 1/[\sigma^2(F_o^2) + (0.0481P)^2$	
$+ 1.8828P]$	
where $P = (F_o^2 + 2F_c^2)/3$	

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

N1—C33	1.416 (3)	C23—C24	1.543 (3)
O1—C32	1.200 (3)	C23—C25	1.549 (3)
O2—C33	1.200 (3)	C33—C34	1.486 (4)
C7—C14	1.334 (3)		
C32—N1—C33	126.0 (2)	C14—C7—C8	123.0 (2)
C32—N1—C31	108.8 (2)	C14—C7—C1	119.3 (2)
C33—N1—C31	124.3 (2)	C8—C7—C1	117.6 (2)
C25—O3—C24	91.1 (2)	O2—C33—C34	122.0 (3)
C25—O3—C24—C23	-12.4 (2)	C24—O3—C25—C23	12.3 (2)
C25—C23—C24—O3	11.7 (2)	C24—C23—C25—O3	-11.7 (2)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H$\cdots$A	D—H	H$\cdots$A	D$\cdots$A	D—H$\cdots$A
C30—H30A$\cdots$O2	0.93	2.29	2.833 (4)	117
C34—H34A$\cdots$O1	0.96	1.98	2.776 (4)	139

Data collection: *SMART* (Siemens, 1996). Cell refinement: *SAINT* (Siemens, 1996). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 1990).

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