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1-Acetyl-4′,5′a-diphenyl-2′,2′a,5′,5′a-tetrahydrospiro[1*H*-indole-3,2′-oxeto[5,4-*b*]-oxazol]-2(3*H*)-one

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In the title compound, $C_{25}H_{18}N_2O_4$, the heterocyclic ring of the indole moiety is slightly distorted towards an envelope conformation. The oxetane plane is almost perpendicular to the indole plane and the dihedral angle between the oxetane and oxazole rings is 65.6 (2)°. The molecular structure is stabilized by C—H···O interactions and the crystal packing is stabilized by intermolecular C—H···O hydrogen bonds as well as dipole–dipole and van der Waals interactions.

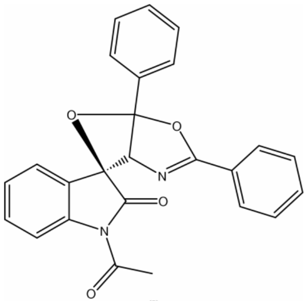
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.060
wR factor = 0.136
Data-to-parameter ratio = 10.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

In our recent investigation on photoinduced reactions of 1-acetylisatin with oxazoles, we have carried out the reaction of 1-acetylisatin with 2,5-diphenyloxazole and obtained the title compound, (I), as one of the products. This compound was formed by photo-induced [2 + 2]-cycloaddition (the Paterno–Buchi reaction) between 1-acetylisatin and the oxazole. To confirm its conformation and stereochemistry an X-ray crystallographic analysis was undertaken.



(I)

The bond lengths and angles in (I) (Table 1) show normal values (Allen *et al.*, 1987) and are comparable to the corresponding values in related compounds (Usman *et al.*, 2001, 2002). The O1—C2 bond length [1.450 (3) Å] is greater than the corresponding value in another spiroxetane structure [1.420 (3) Å; Usman *et al.*, 2002], because of the bulky phenyl substituent attached at C2.

The heterocyclic ring of the indole moiety is not strictly planar, being distorted towards an envelope conformation with atom C4 deviating by 0.145 (6) Å from the N2/C5/C10/C11 plane. The dihedral angle between the mean planes of the heterocyclic and benzene rings in the indole moiety is 3.5 (1)°. The acetyl group attached at atom N2 is slightly twisted out of the indole mean plane [dihedral angle 7.5 (1)°], showing that the acetyl group tends to be coplanar with the indole system, obviously due to the π-conjugation involving the C═O bond.

The oxetane plane is almost perpendicular to the indole mean plane [dihedral angle 87.6 (1)°], as observed in a

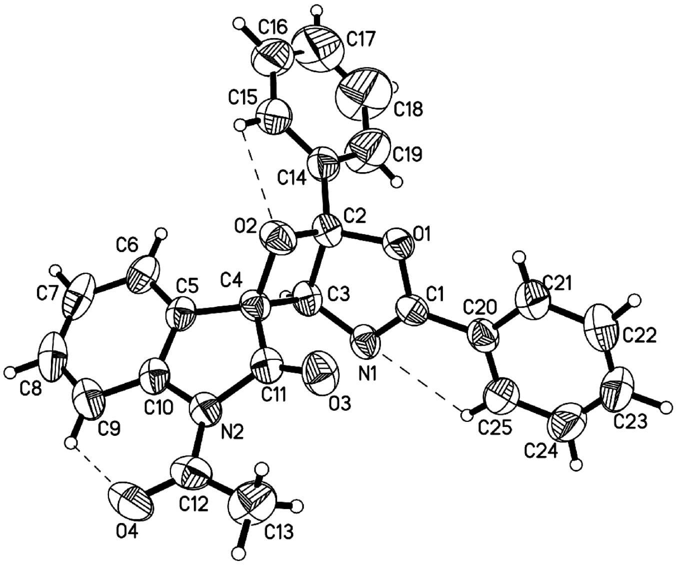


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

reported structure (Usman *et al.*, 2002). The dihedral angle between the oxetane and oxazole rings is 65.6 (2)°, which also agrees with the corresponding value [67.7 (1)°] in the related structure (Usman *et al.*, 2002). The O2—C2—C14 angle is 113.5 (2)°, implying that the phenyl ring attached at the C2 atom is pushed away from the oxetane ring. This phenyl ring forms a dihedral angle of 74.5 (2)° with the oxazole ring.

In the molecular structure of (I) (Fig. 1), there are four intramolecular interactions, forming three five-membered rings and one six-membered ring (Table 2). The C9—H9···O4 interaction supports the π-conjugation of the acetyl group with the indole ring system. In the crystal packing, the molecules are linked by intermolecular C3—H3···O4^{i} hydrogen bonds (see Table 2 for details) into chains parallel to the *b* axis. The molecular packing is stabilized by these interactions, as well as dipole–dipole and van der Waals interactions.

Experimental

The title compound was prepared by photolysis of a benzene solution of 1-acetylisatin in the presence of an excess amount of 2,5-diphenyloxazole followed by silica-gel column-chromatography separation of the reaction mixture with petroleum ether (b.p. 333–363 K)–ethyl acetate as eluant. Single crystals suitable for X-ray diffraction study were obtained by slow evaporation of a petroleum ether (b.p. 333–363 K)–ethyl acetate (6:1, *v*/*v*) solution.

Crystal data

$C_{25}H_{18}N_2O_4$
$M_r = 410.41$
Monoclinic, $P2_1/c$
$a = 16.1912\ (10)$ Å
$b = 10.3984\ (6)$ Å
$c = 12.6742\ (8)$ Å
$\beta = 107.4320\ (10)°$
$V = 2035.9\ (2)$ Å^3
$Z = 4$
$D_x = 1.339$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 4012 reflections
$\theta = 2.4$–$26.8°$
$\mu = 0.09$ mm^{-1}
$T = 293\ (2)$ K
Plate, colourless
0.40 × 0.30 × 0.10 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.964$, $T_{\max} = 0.991$
9901 measured reflections
3567 independent reflections
2683 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.027$
$\theta_{\max} = 25.0°$
$h = -19 \rightarrow 19$
$k = -11 \rightarrow 12$
$l = -15 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.136$
$S = 1.17$
3567 reflections
337 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0494P)^2 + 0.7969P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.16$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}

Table 1
Selected geometric parameters (Å).

O1—C1	1.376 (3)	N1—C3	1.438 (3)
O1—C2	1.450 (3)	C2—C3	1.539 (3)
N1—C1	1.271 (3)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O4^{i}	0.95 (2)	2.26 (2)	3.192 (3)	165 (2)
C9—H9···O4	0.93 (3)	2.28 (3)	2.834 (4)	117 (2)
C15—H15···O2	0.97 (3)	2.41 (3)	2.785 (4)	102 (2)
C21—H21···O1	0.93 (3)	2.47 (3)	2.816 (4)	102 (2)
C25—H25···N1	0.94 (3)	2.52 (3)	2.885 (4)	104 (2)

Symmetry code: (i) $2 - x, \frac{1}{2} + y, \frac{3}{2} - z$.

All H atoms were located in difference Fourier maps and were refined isotropically [C—H = 0.90 (3)–0.98 (4) Å], except for those attached to atoms C13 and C18, which were positioned geometrically and treated as riding, with C—H distances in the range 0.93–0.96 Å and $U_{\rm iso}$(H) values equal to $1.5U_{\rm eq}$(C) [$1.2U_{\rm eq}$(C) for H18]. Owing to the large fraction of weak data at higher angles, the 2θ maximum was limited to 50°.

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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