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1-Acetyl-7a′-phenylspiro[3*H*-indole-3,2′(2a′*H*)-oxeto[2,3-*b*]benzofuran]-2(1*H*)-one

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In the title compound, $C_{24}H_{17}NO_4$, the oxetane ring deviates from planarity. The benzofuran and indole moieties are in synclinal configurations, and the attached phenyl ring forms a dihedral angle of 68.0 (2)° with respect to the oxetane ring. Intramolecular C—H···O interactions stabilize the molecular structure.

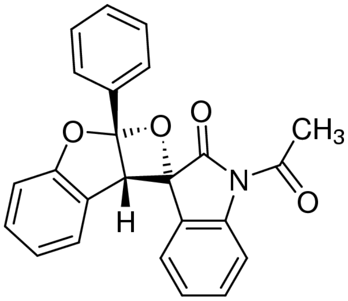
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.064
wR factor = 0.169
Data-to-parameter ratio = 12.2

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

Comment

The photoinduced reactions of 1*H*-indole-2,3-dione (isatin) derivatives, which are compounds of interest mainly due to their biological activities (Bieck *et al.*, 1993) and their widespread use as synthetic precursors of many natural and unnatural products (Popp, 1975; Shvekhgeimer, 1996), have been investigated intensively (Xue *et al.*, 2000, 2001) in order to extend the scope of the photoinduced reactions of carbonyl compounds. The title compound, (I), was one of the two diastereomers obtained in the photoinduced reaction of 1-acetylisatin with 2-phenylbenzofuran. To confirm its conformation and stereochemistry an X-ray structure determination of (I) was undertaken (Fig. 1).



(I)

The bond lengths and angles observed in the title compound (Table 1) show normal values (Allen *et al.*, 1987). These values within the indole and oxetane moieties agree with those of the corresponding related structures previously studied (Zukerman-Schpector *et al.*, 1992; Prakash *et al.*, 1997). The oxetane ring shows a slight puckering and therefore deviates from planarity, with a maximum deviation of −0.025 (3) Å and a dihedral angle of 174.6 (3)° between the O3/C8/C16 and C8/C9/C16 planes. Non-planar oxetanes also are observed in other derivatives (Morris *et al.*, 1987; Khan *et al.*, 1991; Prakash *et al.*, 1997). The benzofuran and indole moieties are almost planar and make dihedral angles of 65.5 (2) and 89.0 (2)° with the mean plane through the oxetane. This corresponds to the synclinal configuration for the two moieties with respect to the

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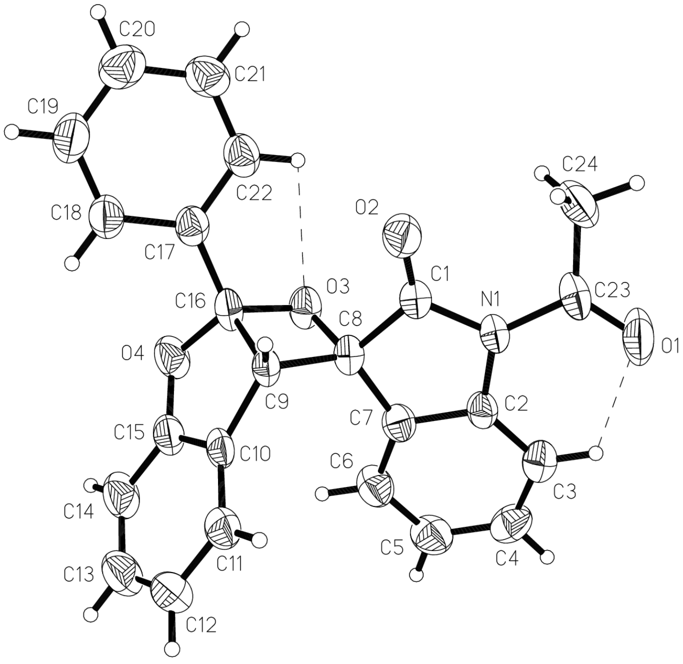

Figure 1
The structure of (I) showing 50% probability displacement ellipsoids and the atom-numbering scheme.

oxetane. The angle C9—C16—C17 is 123.4 (2)°, implying that the attached phenyl ring is pushed slightly away from the benzofuran and indole moieties, and it forms a dihedral angle of 68.0 (2)° with the oxetane ring. Atom O2 deviates by −0.092 (2) Å from the mean plane of the indole moiety, while the acetyl group attached at N1 is twisted by 13.9 (2)° from the plane. The O1 atom of the acetyl group and the O3 atom show weak intramolecular hydrogen-bond interactions (Table 2) forming closed rings O1—C23—N1—C2—C3—H3*A* and O3—C16—C17—C22—H22*A*. These interactions stabilize the molecular structure.

Experimental

The title compound, (I), was prepared by the photoinduced reaction (Peterno–Büchi reaction; Arnold, 1968) of a benzene solution (40 ml) of 1-acetylisatin (0.05 *M*) with an excess amount of 2-phenylbenzofuran with a light of a medium-pressure mercury lamp (500 W, λ > 400 nm). The reaction was carried out under constant nitrogen purging. After irradiation, the solvent was removed *in vacuo* and the residue was separated by column chromatography. Compound (I) was obtained as the main diastereomer of the product and was recrystallized from a mixture of petroleum ether and ethyl acetate.

Crystal data

$C_{24}H_{17}NO_4$
$M_r = 383.39$
Triclinic, $P\overline{1}$
$a = 9.2136$ (2) Å
$b = 10.2904$ (3) Å
$c = 10.9603$ (3) Å
$\alpha = 68.119$ (1)°
$\beta = 80.830$ (1)°
$\gamma = 75.083$ (1)°
$V = 929.47$ (4) Å^3

$Z = 2$
$D_x = 1.370$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 3227 reflections
$\theta = 2.0$–28.3°
$\mu = 0.09$ mm^{-1}
$T = 293$ (2) K
Slab, colourless
0.36 × 0.28 × 0.18 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
5322 measured reflections
3231 independent reflections
2148 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.048$
$\theta_{\max} = 25.0$°
$h = -10 \rightarrow 9$
$k = -12 \rightarrow 8$
$l = -13 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.064$
$wR(F^2) = 0.169$
$S = 0.94$
3231 reflections
264 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0957P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.29$ e Å^{-3}
$\Delta\rho_{\min} = -0.31$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.081 (8)

Table 1
Selected geometric parameters (Å, °).

O3—C16	1.439 (3)	C2—C7	1.396 (3)
O3—C8	1.462 (3)	C7—C8	1.495 (3)
O4—C15	1.378 (3)	C8—C9	1.553 (3)
O4—C16	1.442 (3)	C9—C10	1.493 (3)
N1—C1	1.403 (3)	C9—C16	1.555 (3)
N1—C2	1.437 (3)	C10—C15	1.391 (4)
C1—C8	1.541 (3)	C16—C17	1.497 (4)
C16—O3—C8	93.09 (16)	O3—C8—C9	90.29 (16)
C15—O4—C16	107.99 (19)	O3—C16—C9	91.08 (17)
C1—N1—C2	109.43 (18)	O4—C16—C9	107.13 (19)
N1—C1—C8	108.1 (2)	C17—C16—C9	123.4 (2)
C7—C8—C1	102.86 (19)		
C1—N1—C2—C3	−179.5 (2)	C16—C9—C10—C11	−178.3 (3)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3*A*···O1	0.93	2.29	2.839 (4)	117
C22—H22*A*···O3	0.93	2.53	2.857 (3)	100

After checking their presence in the difference map, all H atoms were geometrically fixed and allowed to ride on the parent C atoms and refined isotropically. Due to a 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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