

# 1-Acetyl-1,2-dihydro-2-oxotrispiro[indole-3,2'-oxetane-3',1''; 4',1'''-dicyclopropane]

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## Key indicators

Single-crystal X-ray study

$T = 293$  K

Mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å

$R$  factor = 0.062

$wR$  factor = 0.176

Data-to-parameter ratio = 12.8

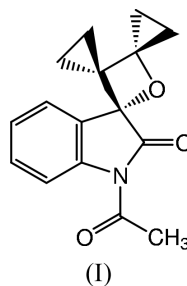
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title compound,  $\text{C}_{16}\text{H}_{15}\text{NO}_3$ , the oxetane ring shows slight deviations from planarity. Its mean plane is approximately orthogonal to the mean plane of the indole system as well as to both cyclopropyl planes.

## Comment

The photoinduced electron transfer (PET) reactions of bicyclopropylidene and its derivatives, so far, have not been thoroughly investigated. In the course of our systematic studies on the PET reactions of various electron-acceptor compounds, we have carried out the photoinduced reactions of bicyclopropylidene with 1-acetylisatin (Wang & Xu, 2002), isolated the title compound, (I), and undertaken its single-crystal X-ray diffraction study.

The bond lengths and angles in (I) (Fig. 1) are within normal ranges (Allen *et al.*, 1987). The geometrical parameters of the indole moiety (C1–C8/N1) are comparable to those of the related structures reported earlier (Usman *et al.*, 2002). Although both atoms C2 and C10 in the oxetane ring (O3/C2/C9/C10) are in the  $sp^3$  hybridization state, their different environments cause slight differences in the O3–C2 and O3–C10 bond lengths, and the O3–C2–C1, O3–C2–C3, O3–C10–C11 and O3–C10–C12 angles (Table 1).



The oxetane ring, as is usually observed (Usman *et al.*, 2002), is not exactly planar. The atoms deviate by  $\pm 0.033$  (2) Å from their mean plane, with the dihedral angle between the C2/O3/C10 and C2/C9/C10 planes being  $172.7$  (2)°.

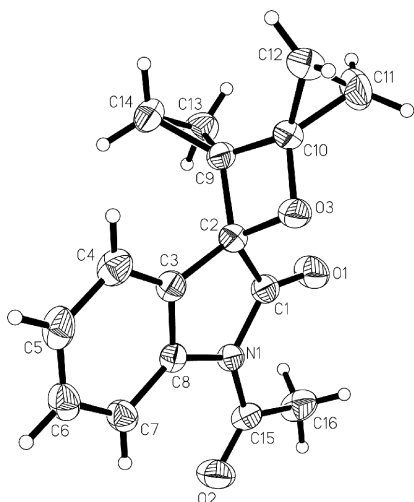
The indole moiety is essentially planar, mainly due to the C=O and C–N conjugation, with the dihedral angle between the planes of its heterocyclic and benzene rings being  $1.6$  (1)°. The acetyl group (O2/C15/C16) bonded to atom N1 is twisted out of the indole plane by a small angle of  $4.8$  (1)°, thus indicating that the acetyl group tends to be coplanar with the indole system (Usman *et al.*, 2002), obviously due to the  $\pi$ -conjugation involving the acetyl C=O bond.

The mean plane of the oxetane ring is almost perpendicular to that of the indole moiety, corresponding to a dihedral angle

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**Figure 1**  
The structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme.

of 88.5 (1)°. The oxetane ring is also perpendicular to both cyclopropyl rings (C9/C13/C14 and C10/C11/C12); this is not unexpected, taking into account the  $sp^3$  hybridization state of atoms C2, C9 and C10.

The reference molecule comprising the asymmetric unit and depicted in Fig. 1 shows the *S* configuration of the chiral center at atom C2. However, the centrosymmetric space group indicates that the crystal is a racemic mixture of both enantiomers of the title compound.

## Experimental

The title compound was prepared by photoinduced reactions of 1-acetylisatin ( $0.05 \text{ mol l}^{-1}$ ) with an excess of bicycpropylidene in benzene solution, and isolated by column chromatography on silica gel (Wang & Xu, 2002). Single crystals suitable for X-ray diffraction analysis were obtained by slow evaporation from an acetone–petroleum ether (1:5 *v/v*) solution.

### Crystal data

$\text{C}_{16}\text{H}_{15}\text{NO}_3$   
 $M_r = 269.29$   
Monoclinic,  $P2_1/c$   
 $a = 10.4584$  (1) Å  
 $b = 9.2661$  (2) Å  
 $c = 13.7923$  (3) Å  
 $\beta = 90.329$  (1)°  
 $V = 1336.57$  (4) Å<sup>3</sup>  
 $Z = 4$

$D_x = 1.338 \text{ Mg m}^{-3}$   
Mo  $K\alpha$  radiation  
Cell parameters from 5107 reflections  
 $\theta = 2.6\text{--}28.3^\circ$   
 $\mu = 0.09 \text{ mm}^{-1}$   
 $T = 293$  (2) K  
Block, colorless  
 $0.46 \times 0.32 \times 0.22 \text{ mm}$

### Data collection

Siemens SMART CCD area-detector diffractometer  
 $\omega$  scans  
6266 measured reflections  
2346 independent reflections  
1656 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.116$   
 $\theta_{\text{max}} = 25.0^\circ$   
 $h = -12 \rightarrow 5$   
 $k = -10 \rightarrow 11$   
 $l = -16 \rightarrow 16$

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.062$   
 $wR(F^2) = 0.176$   
 $S = 0.99$   
2346 reflections  
183 parameters  
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0865P)^2]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.30 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.40 \text{ e \AA}^{-3}$   
Extinction correction: *SHELXTL*  
Extinction coefficient: 0.130 (16)

**Table 1**

Selected geometric parameters (Å, °).

O3—C2	1.471 (3)	C2—C9	1.533 (3)
O3—C10	1.438 (3)	C9—C10	1.494 (3)
O3—C2—C3	114.16 (18)	O3—C10—C11	121.9 (2)
O3—C2—C1	110.05 (17)	O3—C10—C12	120.8 (2)

The H atoms were fixed geometrically and treated as riding on their parent C atoms, with C—H distances = 0.93–0.97 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  [ $1.5U_{\text{eq}}(\text{C})$  for the H atoms of the methyl group].

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT* and *SADABS* (Sheldrick, 1996); 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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## References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.  
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.  
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.  
Sheldrick, G. M. (1997). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.  
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.  
Spek, A. L. (1990). *Acta Cryst.* **A46**, C-34.  
Usman, A., Razak, I. A., Fun, H.-K., Chantrapromma, S., Zhang, Y. & Xu, J.-H. (2002). *Acta Cryst.* **C58**, o287–o288.  
Wang, L. & Xu, J.-H. (2002). In preparation.