Acta Crystallographica Section C
**Crystal Structure Communications**
ISSN 0108-2701

# 1,1′-Diacetyl-3-hydroxy-2,2′,3,3′-tetrahydro-3,3′-bi(1*H*-indole)-2,2′-dione

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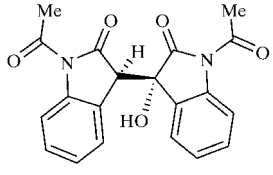
Received 19 July 2001
Accepted 22 October 2001
Online 22 December 2001

In the title compound, $C_{20}H_{16}N_2O_5$, both of the 1-acetylisatin (1-acetyl-1*H*-indole-2,3-dione) moieties are planar and form a dihedral angle of 74.1 (1)°. Weak intermolecular hydrogen bonds and C—H···$\pi$ interactions stabilize the packing in the crystal.

## Comment

Derivatives of 1*H*-indole-2,3-dione (isatin) are of interest due to their biological activity (Bieck *et al.*, 1993) and widespread use as synthetic precursors (Popp, 1975; Shvekhgeimer, 1996). Xu's group has intensively investigated the photoinduced reactions of isatin derivatives in order to explore further the scope of the photochemistry of these compounds (Xue *et al.*, 2000, 2001). The title compound, (I), is one of the products of the photoreaction of 1-acetylisatin and dibenzoylmethane. We have undertaken an X-ray crystal structure analysis of (I) (Fig. 1) to confirm the biindole structure and to establish the steric configuration of the two chiral C atoms.




(I)

The bond lengths and angles observed in (I) are within normal ranges (Table 1). The values for the two 1-acetylisatin moieties agree with each other and are comparable with those of a previously studied structure (Zukerman-Schpector *et al.*, 1992). Each of the 1-acetylisatin moieties is almost planar, with maximum deviations of 0.028 (2) (C7) and −0.066 (1) Å (C9). The dihedral angles between the heterocyclic and benzene rings are 1.7 (1) and 4.1 (1)°. The two acetyl groups are twisted by 8.3 (1) and 14.7 (1)° from their attached isatin planes, and the two 1-acetylisatin moieties form a dihedral angle of 74.1 (1)°. The O2 and O3 ketone atoms are essentially coplanar with their attached isatin groups, with maximum deviations of 0.032 (1) and 0.127 (1) Å, respectively. Atoms C8 and C9 are chiral centers.

Although intramolecular interactions between the acetyl O1 and O4 atoms and the indole-H atoms [H5*A*···O4 2.33 Å, C5···O4 2.863 (2) Å and C5*A*—H5*A*···O4 116°; H12*A*···O1 2.31 Å, C12···O1 2.855 (2) Å and C12—H12*A*···O1 117°] can be viewed as closed six-membered rings of O4—C17—N1—C6—C5—H5*A* and O1—C19—N2—C11—C12—H12*A*, the planarity of both of the 1-acetylisatin moieties is mostly due to conjugation of the N—C and C═O bonds. Atom O4 is also involved in a weak intermolecular O—H···O hydrogen bond, and atom O2 facilitates a weak bifurcated intermolecular

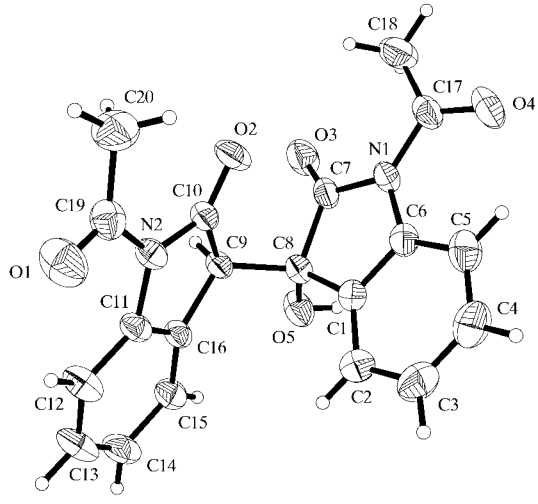



**Figure 1**
The structure of (I) showing 50% probability displacement ellipsoids and the atom-numbering scheme.

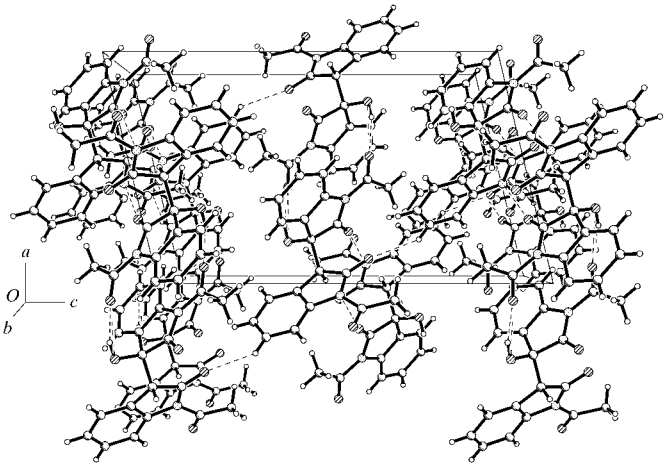



**Figure 2**
Packing diagram of (I) viewed down the *b* axis. The dashed lines denote weak intermolecular hydrogen bonds.

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  DOI: 10.1107/S0108270101017875 

C—H···O hydrogen bond (Table 2). These intermolecular hydrogen bonds interconnect the molecules into a three-dimensional molecular arrangement (Fig. 2). Intermolecular C—H···$\pi$ interactions are observed for C18—H18$A$···$\pi$($C_A$)$^{i}$ [H···$\pi$ 2.79 Å, C···$\pi$ 3.570 (2) Å and C—H···$\pi$ 139°; symmetry code: (i) $2-x$, $2-y$, $-z$] and C18—H18$A$···$\pi$($C_B$)$^{ii}$ [H···$\pi$ 3.19 Å, C···$\pi$ 3.846 (2) Å and C—H···$\pi$ 127°; symmetry code: (ii) $1-x$, $2-y$, $-z$], where $C_A$ and $C_B$ denote the centers of gravity of benzine rings C1–C6 and C11–C16, respectively, of the isatin moieties. The intermolecular hydrogen bonds and C—H···$\pi$ interactions stabilize the molecular packing.

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

| | | | |
|---|---|---|---|
| O2—C10 | 1.2072 (16) | C1—C6 | 1.394 (2) |
| O3—C7 | 1.1978 (19) | C1—C8 | 1.502 (2) |
| O5—C8 | 1.4197 (18) | C7—C8 | 1.551 (2) |
| N1—C7 | 1.4169 (19) | C8—C9 | 1.537 (2) |
| N1—C6 | 1.436 (2) | C9—C16 | 1.5040 (19) |
| N2—C10 | 1.4059 (19) | C9—C10 | 1.521 (2) |
| N2—C11 | 1.4419 (18) | | |
| | | | |
| C7—N1—C6 | 108.95 (12) | C1—C8—C9 | 116.05 (13) |
| C10—N2—C11 | 109.06 (12) | O5—C8—C7 | 110.15 (12) |
| | | | |
| C1—C8—C9—C16 | −65.88 (17) | | |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O5—H5$A$···O4$^{i}$ | 0.82 | 2.00 | 2.799 (2) | 164 |
| C9—H9$A$···O2$^{ii}$ | 0.98 | 2.59 | 3.564 (2) | 176 |
| C13—H13$A$···O2$^{iii}$ | 0.93 | 2.49 | 3.143 (2) | 128 |

Symmetry codes: (i) $2-x, 2-y, -z$; (ii) $1-x, 2-y, -z$; (iii) $x-\frac{1}{2}, \frac{3}{2}-y, z-\frac{1}{2}$.

## Experimental

The title compound was prepared by the photoinduced reaction of a benzene solution (40 ml) of 1-acetylisatin (0.05 *M*) in an excess of dibenzoylmethane with Pyrex-filtered light from a medium-pressure mercury lamp. The reaction was carried out under a constant nitrogen purge. After irradiation, the solvent was removed *in vacuo* and the residue was separated by column chromatography with petroleum spirit/ethyl acetate as eluent. The title compound was obtained as the sole product and was recrystallized from an acetone/petroleum ether mixture.

*Crystal data*

| | |
|---|---|
| $C_{20}H_{16}N_2O_5$ | $D_x$ = 1.383 Mg m$^{-3}$ |
| $M_r$ = 364.35 | Mo $K\alpha$ radiation |
| Monoclinic, $P2_1/n$ | Cell parameters from 8056 reflections |
| $a$ = 10.2447 (1) Å | $\theta$ = 2.1–28.3° |
| $b$ = 10.3416 (1) Å | $\mu$ = 0.10 mm$^{-1}$ |
| $c$ = 16.9848 (2) Å | $T$ = 293 (2) K |
| $\beta$ = 103.482 (1)° | Block, colorless |
| $V$ = 1749.89 (3) Å$^3$ | 0.40 × 0.36 × 0.32 mm |
| $Z$ = 4 | |

*Data collection*

| | |
|---|---|
| Siemens SMART CCD area-detector diffractometer | 4186 independent reflections |
| | 2872 reflections with $I > 2\sigma(I)$ |
| $\omega$ scans | $R_{int}$ = 0.071 |
| Absorption correction: empirical (*SADABS*; Sheldrick, 1996) | $\theta_{max}$ = 28.2° |
| | $h = -13 \rightarrow 13$ |
| $T_{min}$ = 0.961, $T_{max}$ = 0.968 | $k = -13 \rightarrow 10$ |
| 11 650 measured reflections | $l = -22 \rightarrow 22$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | H-atom parameters constrained |
| $R[F^2 > 2\sigma(F^2)]$ = 0.053 | $w = 1/[\sigma^2(F_o^2) + (0.0647P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2)$ = 0.138 | |
| $S$ = 0.92 | $(\Delta/\sigma)_{max} < 0.001$ |
| 4186 reflections | $\Delta\rho_{max}$ = 0.21 e Å$^{-3}$ |
| 246 parameters | $\Delta\rho_{min}$ = −0.30 e Å$^{-3}$ |

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

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).

The authors would like to thank the Malaysian Government and Universiti Sains Malaysia for research grant R&D No. 305/PFIZIK/610961. AU thanks Universiti Sains Malaysia for a Visiting Postdoctoral Fellowship.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: DA1206). Services for accessing these data are described at the back of the journal.

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