

1-Acetyl-3-[benzoyl(4-methoxyphenyl)methylene]-2,3-dihydro-1H-indol-2-one

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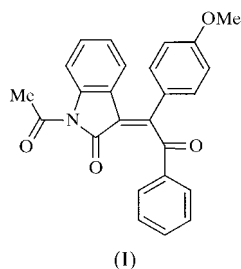
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In the title compound, C₂₅H₁₉NO₄, the indole moiety is not completely planar, the heterocyclic ring being distorted very slightly towards a half-chair conformation. The benzoyl and 4-methoxyphenyl substituents are individually almost planar and are in a bisecting and nearly perpendicular configuration, respectively, with respect to the plane of the indole moiety. The molecular and packing structures in the crystal are stabilized by intramolecular and intermolecular C—H···O interactions.

Comment

2,3-Dihydro-3-methylene-1H-indol-2-one derivatives are synthetic precursors for biologically active spirocyclohexene-indoles and certain alkaloids (Beccalli *et al.*, 1999). The stereochemistry, including the configuration and conformation, and their correlations with other spectroscopic properties are also of current research interest (Coda *et al.*, 1984). The photoreaction of 1-acetylisatin with 1,2-diphenylacetylenes is



one of the synthetic routes for obtaining such derivatives (Zhang *et al.*, 2000). In order to investigate the effect of the attached substituents of 1,2-diphenylacetylene on the regioselectivity of the photoreaction and the stereochemical properties of the product, we have synthesized 1-acetyl-3-

[benzoyl(4-methoxyphenyl)methylene]-2,3-dihydro-1H-indol-2-one, (I), which is the product of the photoreaction of 1-acetylisatin with 1-(4-methoxyphenyl)-2-phenylacetylene. As part of this research interest, we determined the X-ray crystal structure of (I) in order to confirm its conformation and stereochemistry.

The bond lengths and angles in the title compound (Table 1) have normal values (Allen *et al.*, 1987), except for the bond lengths around C11, due to the steric effects of the bulky substituents. The geometry values within the indole moiety agree with those of a related structure (Usman *et al.*, 2001).

In the title structure (Fig. 1), the indole moiety is not

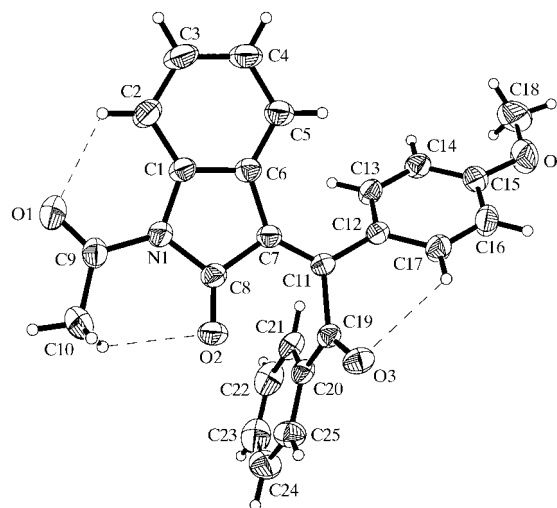


Figure 1

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

completely planar, the heterocyclic ring being distorted very slightly towards a half-chair conformation. This conformation is confirmed by its puckering parameters [$Q_2 = 0.090$ (2) Å and $\varphi_2 = 133$ (1)°; Cremer & Pople, 1975] and the deviations of C7 and C8 from the N1/C1/C6/C7/C8 mean plane are -0.052 (2) and 0.056 (2) Å, respectively. The dihedral angle between the mean planes of the heterocyclic and benzene rings of the indole moiety is 5.5 (1)°.

The benzoyl and 4-methoxyphenyl substituents attached at C11 are almost planar, although the carbonyl group of the benzoyl moiety is twisted slightly out of the plane of the benzene ring, so that O3 deviates by 0.390 (1) Å from this plane. The O3/C11/C19/C20 plane is twisted by 22.1 (1)° from that of the benzene ring. The maximum deviation from the plane of the 4-methoxyphenyl substituent is 0.022 (2) Å for atom C12. The angle between the mean planes of these two substituents is 63.6 (1)°.

Although the mean plane of the 4-methoxyphenyl group is in a bisecting configuration with respect to the plane of the indole moiety, while the benzoyl ring lies nearly perpendicular to the plane of the indole-ring system; the corresponding dihedral angles are 48.1 (1) and 85.0 (1)°, respectively. The ketone O2 atom deviates by 0.324 (1) Å from the mean plane of the indole moiety, while the acetyl group attached at N1 is twisted by 10.3 (1)° from the indole-ring plane.

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In the title compound, three intramolecular C—H···O interactions form closed rings, *viz.* O2/C8/N1/C9/C10/H10A, O1/C9/N1/C1/C2/H2A and O3/C19/C11/C12/C17/H17A. The molecules are linked by an intermolecular C23—H23A···O3 interaction into columns along the *a* axis, and another intermolecular C10—H10B···O3 interaction (Table 2) inter-

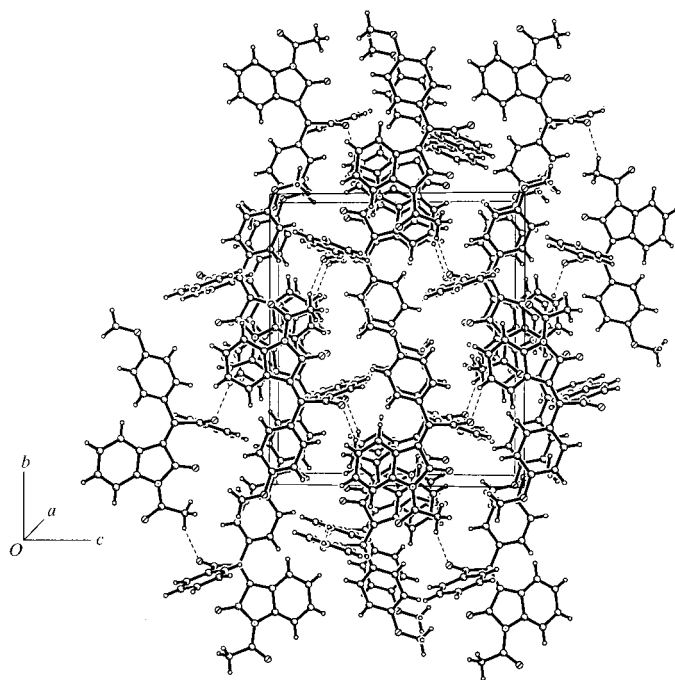


Figure 2
Packing of the title compound viewed down the *a* axis.

connects the columns into a three-dimensional framework. These interactions, together with van der Waals interactions, stabilize the molecular and packing structures in the crystal (Fig. 2).

Experimental

The title compound, (I), was prepared by a photoinduced reaction of 1-acetylisatin with 1-(4-methoxyphenyl)-2-phenylacetylene (Zhang *et al.*, 2000). The primary product in the photoreaction is supposed to be a spiroxetene which rearranges under the reaction conditions to the *Z* and *E* isomers of 1-acetyl-3-[benzoyl(4-methoxyphenyl)methylene]-2,3-dihydro-1*H*-indol-2-one, (I). Single crystals of (I) suitable for X-ray diffraction analysis were obtained by slow evaporation of a petroleum ether–ethyl acetate solution.

Crystal data

C₂₅H₁₉NO₄
M_r = 397.41
 Monoclinic, *P*2₁/*c*
a = 8.0395 (3) Å
b = 16.7881 (5) Å
c = 14.8438 (5) Å
 β = 96.954 (1)°
V = 1988.7 (1) Å³
Z = 4

D_x = 1.327 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 5444 reflections
 θ = 1.8–28.4°
 μ = 0.09 mm⁻¹
T = 293 (2) K
 Block, yellow
 0.34 × 0.26 × 0.22 mm

Data collection

Siemens SMART CCD area-detector diffractometer
 ω scans
 Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
 T_{\min} = 0.970, T_{\max} = 0.980
 13 734 measured reflections

4857 independent reflections
 2603 reflections with $I > 2\sigma(I)$
 R_{int} = 0.069
 θ_{max} = 28.3°
 h = -9 → 10
 k = -19 → 22
 l = -19 → 19

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)]$ = 0.052
 $wR(F^2)$ = 0.135
 S = 0.91
 4857 reflections
 274 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.058P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.18 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.0066 (12)

Table 1
Selected geometric parameters (Å, °).

N1—C8	1.409 (2)	C7—C11	1.349 (2)
N1—C1	1.427 (2)	C11—C12	1.485 (2)
C1—C6	1.403 (2)	C11—C19	1.522 (2)
C6—C7	1.464 (2)	C19—C20	1.476 (3)
C7—C8	1.486 (2)		
C7—C11—C12	125.92 (16)	O3—C19—C11	118.77 (15)
C7—C11—C19	119.41 (15)	O3—C19—C20	122.30 (16)
C12—C11—C19	114.66 (13)		
C8—N1—C9—O1	-173.37 (19)	C18—O4—C15—C16	-179.1 (2)
C1—N1—C9—O1	6.2 (3)	O3—C19—C20—C21	-157.28 (17)
C18—O4—C15—C14	1.6 (3)	O3—C19—C20—C25	24.0 (2)

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

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
C2—H2A···O1	0.93	2.29	2.835 (3)	117
C10—H10A···O2	0.96	2.38	2.762 (3)	103
C10—H10B···O3 ⁱ	0.96	2.43	3.328 (3)	155
C17—H17A···O3	0.93	2.57	3.149 (2)	121
C23—H23A···O3 ⁱⁱ	0.93	2.43	3.251 (3)	148

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

After checking their presence in a difference Fourier synthesis, the positions of all H atoms were geometrically idealized and allowed to ride on their parent C atoms, with C—H distances of 0.93 and 0.96 Å for aromatic and methyl H atoms, respectively, and fixed displacement parameters defined by $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}_{\text{Ar}})$ or $1.5U_{\text{eq}}(\text{C}_{\text{Me}})$.

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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Supplementary data for this paper are available from the IUCr electronic archives (Reference: LN1121). Services for accessing these data are described at the back of the journal.

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