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5-(1*H*-Indol-3-yl)-1-phenylpent-4-ene-1,3-dione

Antonio F. Arrieta[a] and Arvid Mostad[b]*

[a]Institute for Organic Catalysis Research, University of Rostock, Buchbinderstraße 5-6, D-18051 Rostock, Germany, and [b]Department of Chemistry, University of Oslo, PO Box 1033 Blindern, N-0315 Oslo, Norway

Correspondence e-mail: arvidm@kjemi.uio.no

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Key indicators

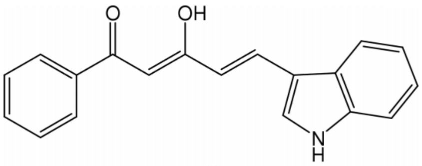
Single-crystal X-ray study
T = 105 K
Mean σ(C–C) = 0.003 Å
R factor = 0.037
wR factor = 0.097
Data-to-parameter ratio = 11.8

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

The title molecule, $C_{19}H_{15}NO_2$, exists in the enolized form and displays a strong intramolecular hydrogen bond. The structure supports the hypothesis that the conjugation in the molecule is a determining factor for the position of the enol H atom. The packing of the molecules is dominated by weak N—H···C and C—H···C interactions.

Comment

The 5-(1*H*-indol-3-yl)-1-phenyl-pent-4-ene-1,3-dione (IPPD) molecule, (I), depicted in Fig. 1, is essentially planar as a result of the conjugation throughout the pentene chain. The largest deviation (observed for atom O1) from the least-squares plane of all non-H atoms is less than 0.21 Å; the mean deviation for all non-hydrogen atoms from this plane is less than 0.06 Å. The dihedral angle formed by the planes of the phenyl and the indolyl groups is 2.7 (1)°.



(I)

The distance of 2.498 (2) Å between atoms O1 and O2 indicates a strong intramolecular hydrogen bond. Even though the enol H atom appears to be closer to O2, the difference between the distances to the two O atoms [H200—O2 1.20 (5) and H200—O1 1.37 (5) Å] is almost within experimental error. However, a statistically more significant difference in the O1—C7 and O2—C9 bond lengths [1.285 (3) and 1.320 (3) Å, respectively] points in the same direction, and is in good agreement with the observation that the enol H atom is more tightly bonded to O2 than to O1. This finding is in accordance with the principle of the longest possible conjugation in the molecule, as observed in several related compounds (Arrieta & Mostad, 2001; Arrieta & Mostad, 2002*a*; Arrieta & Mostad, 2002*b*) The details of the geometry of the intramolecular hydrogen bond and of the intermolecular short contacts (see below) are given in Table 2.

The crystal structure of the present compound is quite similar to that of 1,5-diphenylpent-4-ene-1,3-dione in that the molecules form layers normal to the longest unit cell axis (Arrieta & Mostad, 2002*b*). The contacts between the layers involve the atoms of the phenyl and indolyl rings, the shortest distances being 3.07 (3) Å for H3···C15^{v} [symmetry code (v): $\frac{3}{2} - x, y, z + \frac{1}{2}$] and 3.02 (3) Å for H16···C2^{vi} [symmetry code (vi): $2 - x, 1 - y, -\frac{1}{2} + z$]. Each molecule within the layer is in contact with six neighbours as illustrated in Fig. 3. The shortest

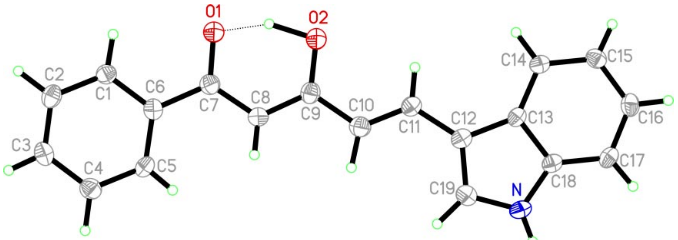


Figure 1
The IPPD molecule, showing the atom-numbering scheme, with displacement ellipsoids drawn at the 50% probability level. The intramolecular hydrogen bond is indicated by a dotted line.

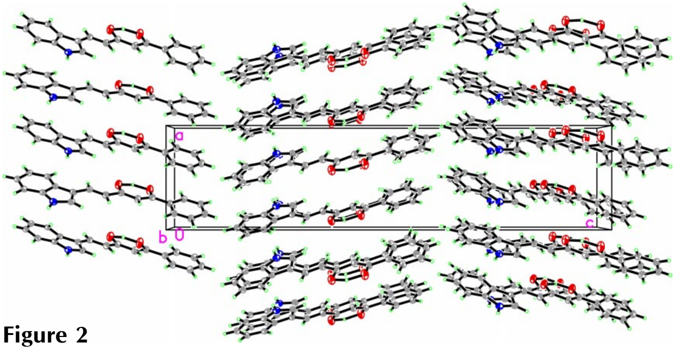


Figure 2
The packing of IPPD molecules in the crystal, viewed down the *b* axis.

intermolecular distance of 2.50 (3) Å is found between atoms H1N and C13*D* [symmetry code (*D*): $x - \frac{1}{2}$, $-y$, z]. The dihedral angles formed by the planes of the aromatic units in neighbouring molecules are 56.8 (1)° for the phenyl rings and 55.4 (1)° for the indolyl rings. This packing mode, where the neigbouring aromatic rings form angles of 70±20° with respect to each other and a C—H bond in one ring is pointing towards the π-electrons of the other, is repeatedly encountered in crystal structures of similar molecules (Mostad, 1994; Arrieta & Mostad, 2002*a*; Arrieta & Mostad, 2002*b*).

Experimental

The compound was synthesized by condensation of benzoylacetone with 1*H*-indol-3-carbaldehyde according to a known procedure (Arrieta *et al.*, 1992). M.p. 479–480 K. The crystals used in the X-ray experiments were recrystallized from a mixture of ethanol/acetone (10:1).

Crystal data

$C_{19}H_{15}NO_2$
$M_r = 289.31$
Orthorhombic, $Pca2_1$
$a = 7.6712\ (15)$ Å
$b = 5.6251\ (11)$ Å
$c = 32.773\ (7)$ Å
$V = 1414.2\ (5)$ Å^3
$Z = 4$
$D_x = 1.354$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 6355 reflections
$\theta = 2.0$–$27.0°$
$\mu = 0.09$ mm^{-1}
$T = 105\ (2)$ K
Plates, red
0.40 × 0.40 × 0.15 mm

Data collection

Bruker SMART diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
$T_{\min} = 0.876$, $T_{\max} = 0.965$
14386 measured reflections
3044 independent reflections

2508 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.052$
$\theta_{\max} = 27.0°$
$h = -9 \rightarrow 9$
$k = -7 \rightarrow 7$
$l = -41 \rightarrow 41$

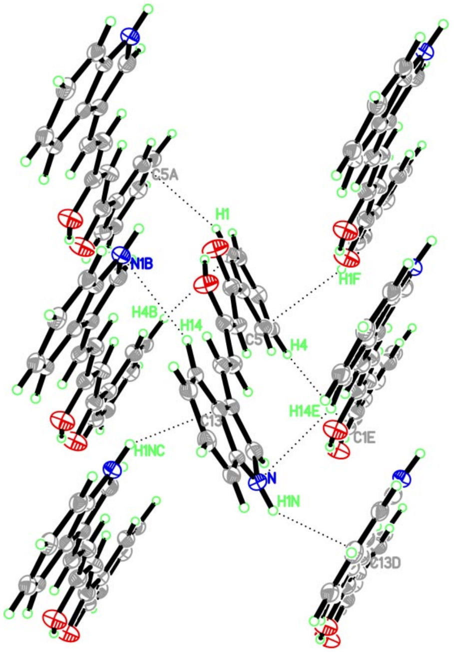


Figure 3
The interaction between neighbouring molecules in the layers of the crystal structure of IPPD. Short contacts are indicated by dotted lines. [Symmetry codes: (*A*) $\frac{1}{2} + x, 2 - y, z$; (*B*) $\frac{1}{2} + x, 1 - y, z$; (*C*) $\frac{1}{2} + x, -y, z$; (*D*) $x - \frac{1}{2}, -y, z$; (*E*) $x - \frac{1}{2}, 1 - y, z$; (*F*) $x - \frac{1}{2}, 2 - y, z$.]

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.097$
$S = 1.09$
3044 reflections
259 parameters

All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0532P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.16$ e Å^{-3}
$\Delta\rho_{\min} = -0.19$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C7	1.285 (3)	C9—C10	1.455 (3)
O2—C9	1.320 (3)	C10—C11	1.339 (3)
N—C18	1.371 (3)	C11—C12	1.452 (3)
N—C19	1.367 (3)	C12—C13	1.449 (3)
C6—C7	1.486 (3)	C12—C19	1.384 (3)
C7—C8	1.415 (3)	C13—C18	1.422 (3)
C8—C9	1.387 (3)		
C19—N—C18	109.81 (18)	C11—C10—C9	123.8 (2)
C1—C6—C7	118.92 (19)	C10—C11—C12	125.6 (2)
C5—C6—C7	122.3 (2)	C19—C12—C13	106.10 (19)
O1—C7—C8	120.3 (2)	C19—C12—C11	127.7 (2)
O1—C7—C6	117.98 (19)	C13—C12—C11	126.21 (19)
C8—C7—C6	121.73 (19)	C14—C13—C12	134.62 (19)
C9—C8—C7	121.2 (2)	C18—C13—C12	106.62 (19)
O2—C9—C8	121.02 (19)	N—C18—C17	130.31 (19)
O2—C9—C10	118.20 (19)	N—C18—C13	107.56 (19)
C8—C9—C10	120.8 (2)	N—C19—C12	109.9 (2)
C2—C1—C6—C7	−178.96 (19)	C7—C8—C9—O2	2.2 (3)
C4—C5—C6—C7	178.7 (2)	C7—C8—C9—C10	−177.3 (2)
C1—C6—C7—O1	−7.5 (3)	O2—C9—C10—C11	1.0 (3)
C5—C6—C7—O1	173.4 (2)	C8—C9—C10—C11	−179.5 (2)
C1—C6—C7—C8	170.7 (2)	C9—C10—C11—C12	−176.7 (2)
C5—C6—C7—C8	−8.4 (3)	C10—C11—C12—C19	0.4 (4)
O1—C7—C8—C9	0.2 (3)	C10—C11—C12—C13	178.7 (2)
C6—C7—C8—C9	−177.9 (2)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···C5^{i}	0.98 (3)	2.96 (3)	3.872 (3)	155.4 (19)
C4—H4···C1^{ii}	1.02 (2)	2.95 (2)	3.812 (3)	142.4 (15)
C14—H14···N^{iii}	0.97 (3)	2.87 (3)	3.459 (3)	120.1 (19)
N—H1N···C13^{iv}	0.85 (3)	2.50 (3)	3.225 (3)	143 (2)
C3—H3···C15^{v}	0.92 (2)	3.07 (3)	3.939 (3)	159.7 (18)
C16—H16···C2^{vi}	0.94 (3)	3.02 (3)	3.877 (3)	150.9 (19)
O2—H200···O1	1.20 (5)	1.37 (5)	2.498 (2)	154 (4)

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

All H atoms were located from a difference Fourier map and refined isotropically. C—H distances are in the range 0.87–1.02 Å; N—H is 0.85 (5) Å.

As one would expect, due to the lack of anomalous scatterers, the Flack (1983) parameter is indeterminate.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXL*97; software used to prepare material for publication: *SHELXL*97.

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