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## Key indicators

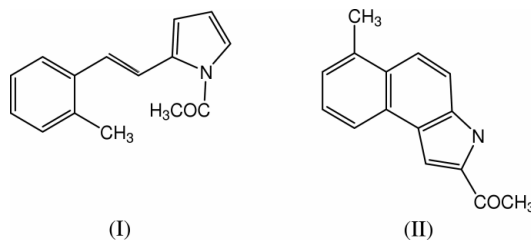
Single-crystal X-ray study  
*T* = 293 K  
Mean  $\sigma$ (C–C) = 0.005 Å  
*R* factor = 0.056  
*wR* factor = 0.168  
Data-to-parameter ratio = 8.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

## Photochemical formation of 2-acetyl-7-methylbenz[e]-indole

The title compound, C<sub>15</sub>H<sub>13</sub>NO, was obtained on irradiation of *N*-acetyl-2-(2-methylstyryl)pyrrole. It was characterized spectroscopically and by X-ray diffraction. The molecule is a planar system of three annelated rings (toluene, benzene and pyrrole). An intermolecular hydrogen bond (N–H···O) is observed, connecting molecules into a chain.

## Comment

As a part of our research on the photochemical behaviour of heterocyclic analogues of *o*-vinylstilbenes (Šindler-Kulyk & Laarhoven, 1978*a,b*, 1979), we have studied furan (Šindler-Kulyk *et al.*, 1989, 1996), annelated furans (Vujković Cvijin *et al.*, 1998; Šindler-Kulyk *et al.*, 1999; Škorić *et al.*, 2000) and pyrrole (Šindler-Kulyk *et al.*, 1995; Basarić *et al.*, 2000, 2001) analogues. While aryl derivatives undergo a [2+2] intramolecular cycloaddition reaction and formation of bicyclo[2.1.1]-cyclohexene derivatives, annelated furans react to give bicyclo[3.2.1]octadiene derivatives and cyclobutane dimers. By contrast, the pyrrole analogues give on irradiation dimeric products as a result of intra- and intermolecular addition.

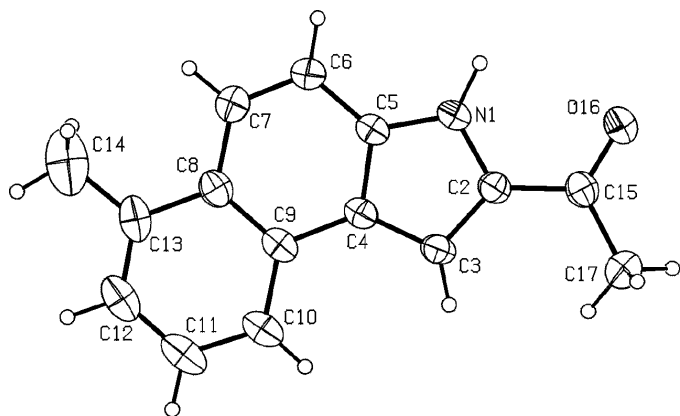


We have therefore studied the acetyl-styryl-pyrrole derivatives. On irradiation of *N*-acetyl-2-(2-methylstyryl)pyrrole, (I), along with phototransposition and *cis*–*trans* isomerization processes, the formation of benzoindeole derivative (II) was observed (see Scheme). The X-ray structure analysis has shown that a molecule of (II) is a system of three coplanar condensed rings (pyrrole, benzene and toluene) (Fig. 1). A search of the Cambridge Structural Database (Version 5.11, April 2001; Allen & Kennard, 1993) has shown that there are very few similar structures reported so far. The molecule does not include a stereogenic centre. However, molecules in the unit cell are arranged in a non-centrosymmetric fashion (space group *P*<sub>2</sub><sub>1</sub><sub>2</sub><sub>1</sub><sub>2</sub><sub>1</sub>). The acyl group at C2 reveals a small deviation from the plane of the three annelated rings [C15 0.025 (4) Å, C17 0.112 (5) Å and O16 –0.035 (3) Å]. The two *Nsp*<sup>2</sup> bonds [N1–C2 1.368 (5) Å and N1–C5 1.367 (4) Å] are in accordance with the reported average value [1.372 (16) Å; Allen *et al.*, 1987]. The other bond distances also have expected values

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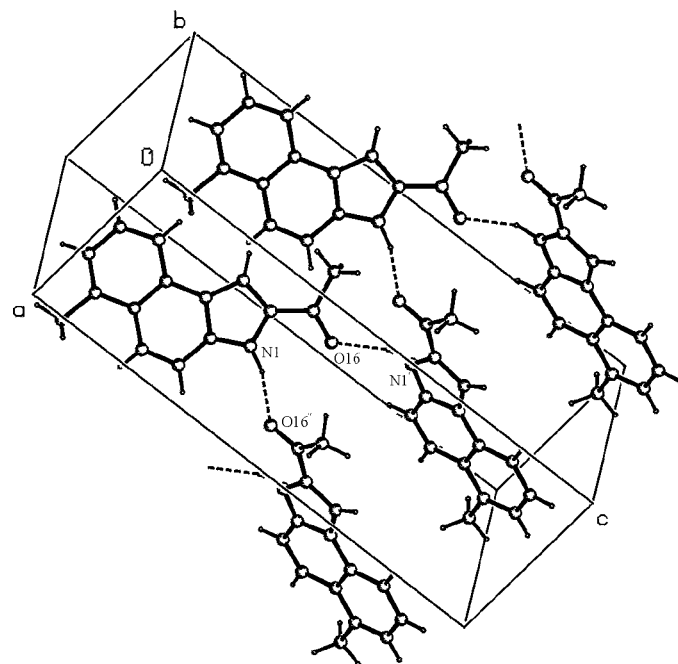


**Figure 1**  
An ORTEP (Johnson, 1976) drawing of (II) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

[C15—O16 1.236 (3) Å and C15—C17 1.500 (6) Å]. The crystal packing is characterized by weak van der Waals interactions and by an N1—H···O16 intermolecular hydrogen bond (Table 2), which connects molecules into a zigzag chain along the crystallographic *a* axis (Fig. 2). Within the zigzag chain, molecules are head-to-head oriented in such a way that every single molecule forms two hydrogen bonds with two neighbours from the opposite layer (through a donor atom O16, and through the acceptor N1). The chain is formed out of the two layers of parallel stacked molecules. No  $\pi$ – $\pi$  interactions were encountered, however, in the structure though.

## Experimental

The starting compound, (I), was prepared by acylating 2-(2-methylstyryl)pyrrole (*cis* or *trans* isomer) with acetic anhydride in the presence of triethylamine and *N,N*-dimethylaminopyridine in dichloromethane, following the procedure for the pyrrole and indole derivatives described in the literature (Nickisch *et al.*, 1980). The products, *cis*-(I) or *trans*-(I), were purified by column chromatography on silica gel using dichloromethane/petroleum ether (30%) as eluent, and characterized spectroscopically. *cis*-*N*-acetyl-2-(2-methylstyryl)pyrrole, (I): 19% yield; m.p. 336–338 K; UV (EtOH)  $\lambda_{\max}/\text{nm}$  ( $\epsilon/\text{dm}^3 \text{ mol}^{-1} \text{ cm}^{-1}$ ): 237(11942), 309(7962);  $^1\text{H}$  NMR ( $\text{C}_6\text{D}_6$ , 300 MHz)  $\delta/\text{p.p.m.}$ : 7.46 [*d*, 1H,  $J = 12.1$  Hz, ethene H (H-et)], 7.34 [*d*, 1H,  $J = 7.5$  Hz, H<sub>ar</sub>], 6.97–7.07 (*m*, 3H, H-ar), 6.49 [*d*, 1H,  $J = 12.1$  Hz, H-et], 6.37 (*m*, 1H, H-5), 5.97 (*m*, 1H, H-3), 5.75 (*m*, 1H, H-4), 2.16 (*s*, 3H, CH<sub>3</sub>), 1.71 (*s*, 3H, COCH<sub>3</sub>);  $^{13}\text{C}$  NMR ( $\text{C}_6\text{D}_6$ , 300 MHz)  $\delta/\text{p.p.m.}$ : 169.41 (*s*), 138.49 (*s*), 136.50 (*s*), 133.00 (*s*), 130.77 (*d*), 129.52 (*d*), 128.90 (*d*), 127.79 (*d*), 126.49 (*d*), 123.14 (*d*), 121.19 (*d*), 115.62 (*d*), 112.42 (*d*), 23.79 (*q*), 19.96 (*q*); *trans*-*N*-acetyl-2-(2-methylstyryl)pyrrole, (I): 65.0% yield; m.p. 339–340 K; UV (EtOH)  $\lambda_{\max}/\text{nm}$  ( $\epsilon/\text{dm}^3 \text{ mol}^{-1} \text{ cm}^{-1}$ ): 248 (12385), 328 (13830); IR (KBr)  $\nu_{\max}/\text{cm}^{-1}$ : 1716 (CO);  $^1\text{H}$  NMR ( $\text{C}_6\text{D}_6$ , 300 MHz)  $\delta/\text{p.p.m.}$ : 8.13 [*d*, 1H,  $J = 16.2$  Hz, H-et], 7.71–7.74 (*m*, 1H, H-ar), 7.22 [*d*, 1H,  $J = 16.2$  Hz, H-et], 7.00–7.05 (*m*, 3H, H-ar), 6.50 (*m*, 1H, H-3), 6.47 (*m*, 1H, H-5), 6.04 (*dd*, 1H,  $J = 3.3$  and 3.6 Hz, H-4), 2.20 (*s*, 3H, CH<sub>3</sub>), 1.71 (*s*, 3H, COCH<sub>3</sub>);  $^{13}\text{C}$  NMR ( $\text{C}_6\text{D}_6$ , 300 MHz)  $\delta/\text{p.p.m.}$ : 169.63 (*s*), 137.23 (*s*), 136.30 (*s*), 136.04 (*s*), 131.01 (*d*), 127.98 (*d*), 127.45 (*d*), 127.11 (*d*), 126.42 (*d*), 122.43 (*d*), 122.08 (*d*), 112.52 (*d*), 111.95 (*d*), 23.77 (*q*), 20.05 (*q*); MS *m/z* (%): 225 (9,  $M^+$ ), 182(7), 121(100), 91(51), 70(27);



**Figure 2**  
Crystal packing of (II) with the hydrogen bonds shown dashed.

elemental analysis: calculated for  $\text{C}_{15}\text{H}_{15}\text{NO}$ : C 79.97, H 6.71, N 6.22%; found: C 79.93, H 6.51%. On irradiation of *cis*- and *trans*-*N*-acetyl-2-(2-methylstyryl)pyrrole, (I), in benzene (concentration  $10^{-3}$  M) at 350 nm and under anaerobic conditions, a mixture of many photoproducts was obtained. After repeated purification by column chromatography on silica gel, the electrocyclic product 2-acetyl-7-methylbenz[*e*]indole, (II), was isolated in crystalline form (yield 1%) and identified by spectroscopic methods: m.p. 516 K;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz)  $\delta/\text{p.p.m.}$ : 9.39 (*bs*, 1H, NH), 8.13 (*d*, 1H,  $J = 7.8$  Hz, H-4), 7.94 (*d*, 1H,  $J = 9.3$  Hz, H-9), 7.72 (*s*, 1H, H-3), 7.54 (*d*, 1H,  $J = 9.3$  Hz, H-8), 7.51 (*t*, 1H,  $J = 7.8$  Hz, H-5), 7.33 (*d*, 1H,  $J = 7.2$  Hz, H-6), 2.74 (*s*, 3H, COCH<sub>3</sub>/CH<sub>3</sub>), 2.66 (*s*, 3H, COCH<sub>3</sub>/CH<sub>3</sub>); MS *m/z* (%): 223 ( $M^+$ , 100), 209(73), 194(55), 180(45), 153(57), 139(52). Suitable crystals for X-ray diffraction were obtained by recrystallization of (II) from  $\text{CDCl}_3$  at room temperature.

## Crystal data

$\text{C}_{15}\text{H}_{13}\text{NO}$   
 $M_r = 223.26$   
Orthorhombic,  $P2_12_12_1$   
 $a = 6.8710$  (6) Å  
 $b = 8.8350$  (10) Å  
 $c = 19.042$  (2) Å  
 $V = 1156.0$  (2) Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.283$  Mg m<sup>-3</sup>

Cu  $K\alpha$  radiation  
Cell parameters from 25 reflections  
 $\theta = 9.5$ – $21.9^\circ$   
 $\mu = 0.63$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
Prismatic, colourless  
 $0.14 \times 0.12 \times 0.09$  mm  
Crystal source: see text

## Data collection

Enraf-Nonius CAD-4 diffractometer  
 $\theta/2\theta$  scans  
Absorption correction: none  
1398 measured reflections  
1398 independent reflections  
1030 reflections with  $I > 2\sigma(I)$

$\theta_{\max} = 74.9^\circ$   
 $h = -8 \rightarrow 0$   
 $k = -11 \rightarrow 0$   
 $l = 0 \rightarrow 23$   
3 standard reflections  
frequency: 120 min  
intensity decay: none

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.056$   
 $wR(F^2) = 0.168$   
 $S = 1.08$   
 1398 reflections  
 161 parameters  
 H atoms treated by a mixture of  
 independent and constrained  
 refinement

$w = 1/[\sigma^2(F_o^2) + (0.1015P)^2 + 0.0534P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.31 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\min} = -0.15 \text{ e } \text{Å}^{-3}$   
 Extinction correction: *SHELXL97*  
 Extinction coefficient: 0.012 (2)

Table 1

Selected geometric parameters (Å, °).

O16—C15	1.236 (3)	N1—C5	1.367 (4)
N1—C2	1.368 (5)		
C2—N1—C5	109.1 (2)	N1—C5—C4	108.0 (3)
N1—C2—C15	120.4 (2)	O16—C15—C2	119.9 (4)
N1—C2—C3	108.4 (3)	O16—C15—C17	121.6 (3)
N1—C5—C6	128.9 (3)		

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N1—H1 $\cdots$ O16 <sup>i</sup>	0.96 (4)	1.99 (4)	2.894 (3)	156 (4)

Symmetry code: (i)  $\frac{1}{2} + x, \frac{1}{2} - y, 1 - z$ .

H atoms were included in geometrically calculated positions and refined with a riding model, except for the N—H H1 atom, which was located in a difference map and refined freely. High displacement parameters may indicate unresolved disorder for the methyl group at C14, and possibly lesser disorder in the neighbouring C11—C13 atoms. Friedel pairs were not measured, since anomalous dispersion effects are negligible.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *SET4* and *CELDIM* in *CAD-4 Software*; data reduction:

*HELENA* (Spek, 1997); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *PLATON* (Spek, 1990).

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