

4-[ $\alpha$ -Benzoyl- $\alpha$ -(2-phenylethynyl)methylene]isoquinoline-1,3-dioneAnwar Usman,<sup>a</sup> Ibrahim Abdul Razak,<sup>a</sup> Hoong-Kun Fun,<sup>a\*</sup> Suchada Chantrapromma,<sup>a†</sup> Yan Zhang<sup>b</sup> and Jian-Hua Xu<sup>b</sup><sup>a</sup>X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and <sup>b</sup>Department of Chemistry, Nanjing University, Nanjing 210093, People's Republic of China

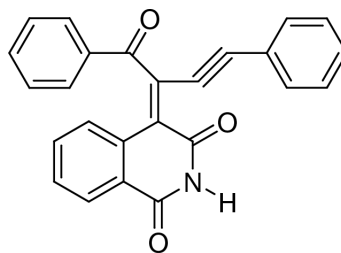
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## Key indicators

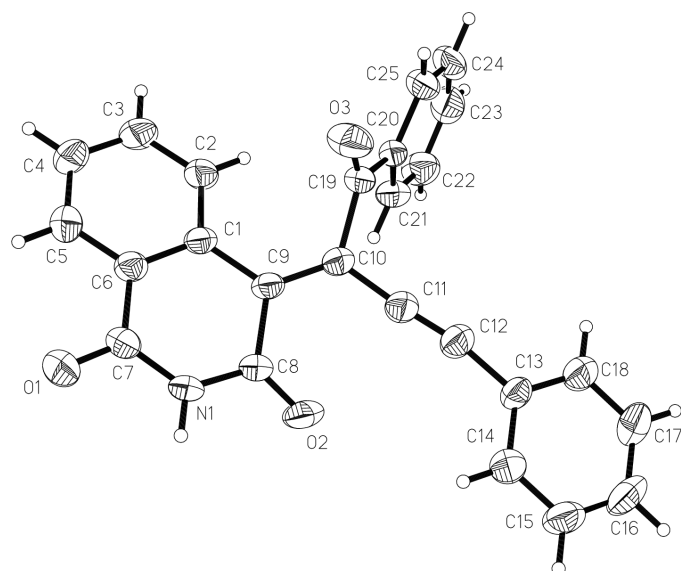
Single-crystal X-ray study  
 $T = 293$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å  
 $R$  factor = 0.074  
 $wR$  factor = 0.195  
Data-to-parameter ratio = 11.6For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.In the title compound,  $\text{C}_{25}\text{H}_{15}\text{NO}_3$ , the isoquinoline moiety is distorted from planarity. In the solid state, the molecules exist as centrosymmetrically  $\text{N}-\text{H}\cdots\text{O}$  hydrogen-bonded dimers.

## Comment

The photoinduced reaction of isoquinoline-1,3-diones with diphenylacetylenes has been intensively investigated (Zhang *et al.*, 2000). As an extension of the photoreactions of quinoline-1,3-dione derivatives with alkynes, we have synthesized the title compound, (I). An X-ray analysis of (I) was undertaken in order to elucidate its molecular conformation.The bond lengths and angles in the title compound (Table 1) show normal values (Allen *et al.*, 1987) except those around atom C10, due to the steric effect of the bulky substituents. The C10–C11 and C12–C13 bond distances are longer than that of the typical  $\text{Csp}^2-\text{Csp}^1$  distance and the C10–C19 bond distance is longer than that of the typical  $\text{Csp}^2-\text{Csp}^2$  distance. The bond angles around C10 and the C10–C11–C12–C13 torsion angle of  $145(3)^\circ$  indicate that both the bulky benzoyl and phenylethynyl substituents are pushed slightly away from the isoquinoline moiety.

(I)

The heterocyclic ring of the isoquinoline moiety is distorted from planarity with  $Q_T = 0.184(3)$ ,  $q_2 = 0.176(3)$ ,  $q_3 = 0.055(3)$  Å and  $\varphi_2 = -253.2(10)^\circ$  (Cremer & Pople, 1975) and the mean plane through that ring forms a dihedral angle of  $8.3(2)^\circ$  with the benzene ring. Atoms O1 and O2 deviate from the isoquinoline moiety by  $-0.202(2)$  and  $0.422(3)$  Å, respectively. The phenyl ring of the phenylethynyl substituent is nearly coplanar with the plane through the isoquinoline moiety, with a dihedral angle of  $5.9(1)^\circ$  between them. The dihedral angle between the phenyl rings of the benzoyl and phenylethynyl substituents is  $66.5(2)^\circ$ . In the crystal, the molecules form centrosymmetrically  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonded dimers (Table 2) which are stacked along the  $a$  cell axis.



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

## Experimental

The title compound, (I), was prepared by the photolysis of a benzene solution of *N*-methylisoquinoline-1,3-dione (0.05 *M*) in an excess amount of 1,4-diphenylbuta-1,3-diyne with light of wavelength longer than 400 nm and under constant nitrogen purging. The slow evaporation of a solution of the product in a mixture of petroleum ether and acetone (2:3) gave single crystals suitable for X-ray study.

### Crystal data

$C_{25}H_{15}NO_3$   
 $M_r = 377.38$   
Triclinic,  $P\bar{1}$   
 $a = 7.5566$  (5) Å  
 $b = 10.3679$  (7) Å  
 $c = 12.7275$  (8) Å  
 $\alpha = 102.273$  (1)°  
 $\beta = 100.562$  (1)°  
 $\gamma = 100.703$  (1)°  
 $V = 931.28$  (11) Å<sup>3</sup>

$Z = 2$   
 $D_x = 1.346$  Mg m<sup>-3</sup>  
Mo  $K\alpha$  radiation  
Cell parameters from 3718 reflections  
 $\theta = 1.7\text{--}29.6^\circ$   
 $\mu = 0.09$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
Block, yellow  
 $0.34 \times 0.22 \times 0.18$  mm

### Data collection

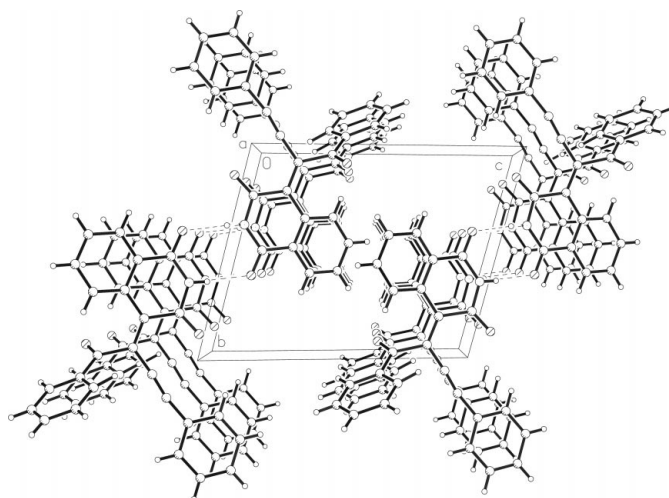
Siemens SMART CCD area-detector diffractometer  
 $\omega$  scans  
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)  
 $T_{\min} = 0.970$ ,  $T_{\max} = 0.984$   
5135 measured reflections

3151 independent reflections  
1918 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.067$   
 $\theta_{\text{max}} = 25.0^\circ$   
 $h = -8 \rightarrow 8$   
 $k = -7 \rightarrow 12$   
 $l = -15 \rightarrow 15$

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.074$   
 $wR(F^2) = 0.195$   
 $S = 0.91$   
3151 reflections  
271 parameters  
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0735P)^2]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.35$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.33$  e Å<sup>-3</sup>  
Extinction correction: *SHELXTL*  
Extinction coefficient: 0.077 (10)



**Figure 2**  
Packing of the molecules viewed down the *a* axis.

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

N1—C7	1.356 (4)	C10—C11	1.418 (4)
N1—C8	1.378 (4)	C10—C19	1.535 (4)
C1—C6	1.399 (4)	C11—C12	1.186 (4)
C1—C9	1.476 (4)	C12—C13	1.435 (4)
C8—C9	1.508 (4)	C19—C20	1.472 (4)
C9—C10	1.350 (4)		
C9—C10—C11	124.8 (2)	C12—C11—C10	175.2 (3)
C9—C10—C19	123.9 (2)	C11—C12—C13	170.3 (3)
C11—C10—C19	110.8 (2)		

**Table 2**  
Hydrogen-bonding 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>
N1—H1A...O1 <sup>i</sup>	0.79 (4)	2.14 (3)	2.928 (3)	170 (3)

Symmetry code: (i)  $-1 - x, -1 - y, -z$ .

After checking their presence in a difference map, the H atoms were geometrically fixed and allowed to ride on the parent atoms and refined isotropically. Atoms H1A and H21A, attached to atoms N1 and C21, respectively, were freely refined.

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.

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