Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

3-Acetyl-1-(4-chlorophenyl)-2-methylbenzo[*f*]indole-4,9-dione

Hoong-Kun Fun,[a]* Suchada Chantrapromma,[b]‡ Yun Liu[c] and Jian-Hua Xu[c]

[a]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, [b]Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, and [c]Department of Chemistry, Nanjing University, Nanjing 210093, People's Republic of China

‡ Additional correspondence author, email: suchada.c@psu.ac.th.

Correspondence e-mail: hkfun@usm.my

Received 18 March 2007
Accepted 31 March 2007

In the title compound, $C_{21}H_{14}ClNO_3$, the benzo[*f*]indole unit is essentially planar. The dihedral angles between the indole unit and the planes of the chlorophenyl and acetyl groups are 84.30 (6) and 45.51 (7)°, respectively. Intramolecular C—H···O interactions generate *S*(6) and *S*(7) ring motifs. The molecules are linked *via* weak intermolecular C—H···O interactions to form double-layer chains along the *c* axis. These chains are arranged in head-to-tail and tail-to-head modes. C—H···π interactions are present in the crystal structure.

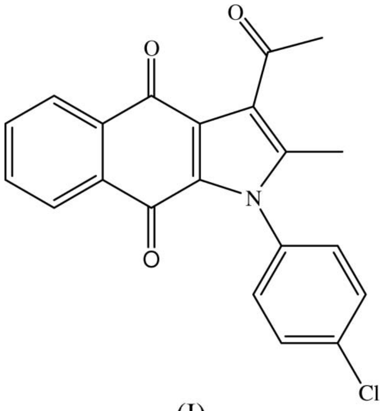
Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.040
wR factor = 0.113
Data-to-parameter ratio = 20.2

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

Comment

Naturally occurring quinones constitute an important class of natural products (Patai & Rappoport, 1988; Thomson, 1997) that have a wide range of biological activities; they include benzannulated indolequinones. The synthesis of benzo[*f*]-indole-4,9-dione derivatives has attracted much recent attention. In our recent effort to synthesize benzo[*f*]indole-4,9-diones by one-pot procedures (Hu *et al.*, 2006), we have synthesized the title compound, (I), in order to study its biological activities. An X-ray crystallographic analysis was undertaken to elucidate its three-dimensional structure.



(I)

In the asymmetric unit of (I) (Fig. 1), the benzo[*f*]indole unit (N1/C7–C18) is essentially planar, with atom C18 having the maximum deviation of 0.042 (1) Å. The chlorophenyl ring is almost perpendicularly attached at atom N1; the dihedral angle between the indole and chlorophenyl ring planes is 84.30 (6)°. The acetyl group (C19–C20/O1) is (−)-synclinally attached at atom C17 with the torsion angle C16—C17—C19—C20 = −45.7 (2)°; the dihedral angle between the indole unit and the mean plane of the acetyl group is 45.51 (7)°. The weak intramolecular interaction C20—H20*B*···O1 generates

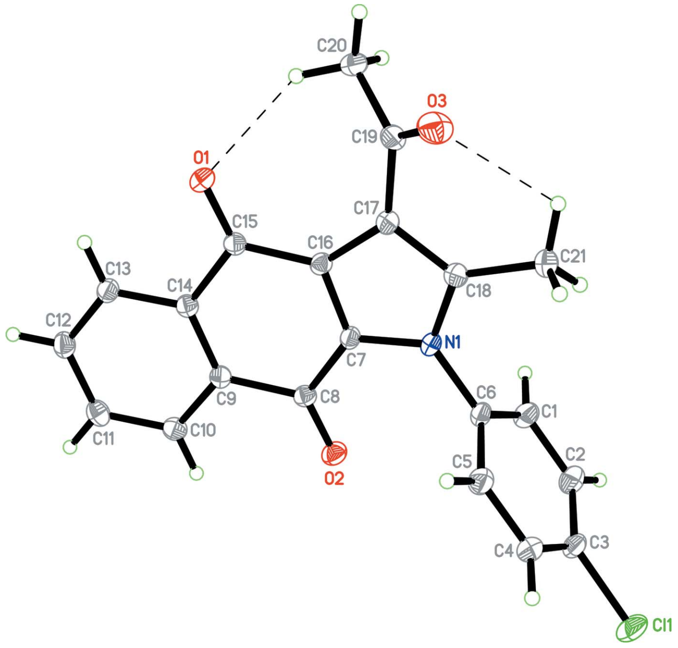


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atomic numbering. Intramolecular C—H···O weak interactions are shown as dashed lines.

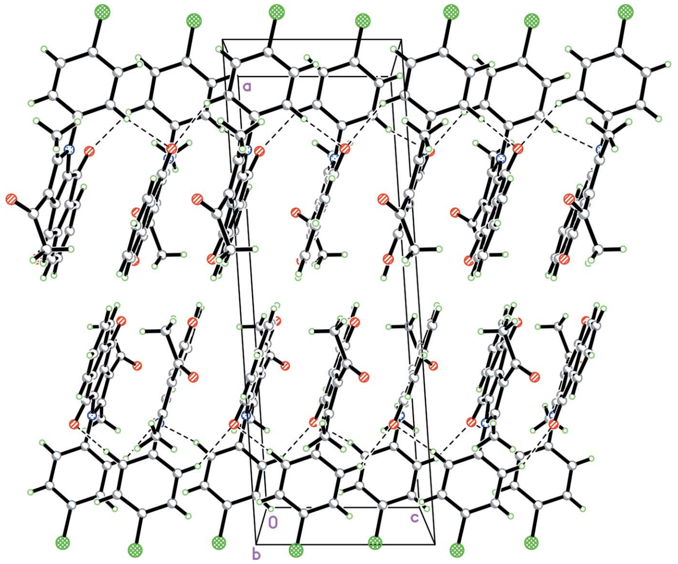


Figure 2
The crystal packing of (I), viewed down the *b* axis. Hydrogen bonds are shown as dashed lines.

an *S*(6) ring motif, and C21—H21*C*···O3 generates an *S*(7) ring motif (Bernstein *et al.*, 1995). All bond lengths and angles are in normal ranges (Allen *et al.*, 1987).

The crystal packing (Fig. 2) shows that the molecules are linked *via* weak intermolecular interactions of the type C—H···O to form double-layer chains along the *c* axis. These chains are arranged in a head-to-tail and tail-to-head manner (Fig. 2). A C—H···π interaction is present in the crystal structure (Table 2).

Experimental

Compound (I) was prepared by the reaction of 2,3-dichloro-1,4-naphthoquinone (1.1 mmol) with 4-[(4-chlorophenyl)imino]-2-pentanone (1 mmol) in the presence of sodium carbonate (2.5 mmol) in DMF (15 ml) in 92% yield. Yellow single crystals of (I) suitable for X-ray diffraction analysis were obtained by slow evaporation of the solvents from a chloroform/petroleum ether solution (1:3 *v*/*v*); m.p. 512–514 K.

Crystal data

$C_{21}H_{14}ClNO_3$
$M_r = 363.78$
Monoclinic, $P2_1/c$
$a = 20.0460$ (2) Å
$b = 11.6692$ (1) Å
$c = 7.0866$ (1) Å
$\beta = 93.8150$ (10)°

$V = 1654.03$ (3) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.25$ mm^{-1}
$T = 100.0$ (1) K
0.33 × 0.25 × 0.12 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.921$, $T_{max} = 0.971$

25984 measured reflections
4781 independent reflections
3950 reflections with $I > 2\sigma(I)$
$R_{int} = 0.033$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.113$
$S = 1.07$
4781 reflections

237 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.50$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C3	1.7401 (14)	O3—C19	1.2191 (17)
O1—C15	1.2213 (16)	C7—C16	1.3879 (17)
O2—C8	1.2242 (16)	C17—C18	1.3909 (18)
C16—C17—C19—C20	−45.7 (2)		

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

*Cg*1 is the centroid of the N1/C7/C16–C18 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*A*···O2^{i}	0.93	2.55	3.1975 (18)	127
C5—H5*A*···O2^{ii}	0.93	2.55	3.3450 (17)	144
C11—H11*A*···O3^{iii}	0.93	2.59	3.4358 (18)	151
C20—H20*B*···O1	0.96	2.27	3.0942 (17)	143
C21—H21*C*···O3	0.96	2.58	3.0508 (18)	110
C10—H10*A*···*Cg*1^{i}	0.93	3.34	3.7253 (15)	108

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

All H atoms were positioned geometrically and refined using a riding model with C—H = 0.93 Å for aromatic and 0.96 Å for CH_3; $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(C)$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

Acta Cryst. (2007). E**63**, o2314–o2316 Fun *et al.* • $C_{21}H_{14}ClNO_3$ **o2315**

This work was supported by the National Natural Science Foundation of China and the Natural Science Foundation of Jiangsu Province. The authors also thank the Malaysian Government and Universiti Sains Malaysia for the Scientific Advancement Grant Allocation (SAGA) grant No. 304/PFIZIK/653003/A118.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.

Bernstein, J., Davis, R. E., Shimoni, L. & Chamg, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Bruker (2005). *APEX2* (Version 1.27), *SAINT* (Version V7.12a) and *SADABS* (Version 2004/1). Bruker AXS Inc., Madison, Wisconsin, USA.

Hu, H.-Y., Liu, Y., Ye, M. & Xu, J. H. (2006). *Synlett*, pp. 1913–1917.

Patai, S. & Rappoport, Z. (1988). Editors. *The Chemistry of Functional Groups: The Chemistry of the Quinoid Coumpounds*. New York: Wiley.

Sheldrick, G. M. (1998). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Thomson, R. H. (1997). *Natural Occurring Quinones*, 4th ed. London/New York: Chapman & Hall.

o2316 Fun *et al.* • $C_{21}H_{14}ClNO_3$ *Acta Cryst.* (2007). E**63**, o2314–o2316