

Hou-Guang Shi,^a Xue-Mei Li,^b
Jian-Hua Xu,^a Shu-Sheng Zhang^b
and Hoong-Kun Fun^{c*}

^aDepartment of Chemistry, Nanjing University, 210093 Nanjing, People's Republic of China, ^bCollege of Chemistry and Molecular Engineering, Qingdao University of Science and Technology, 266042 Qingdao, Shandong, People's Republic of China, and ^cX-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia

Correspondence e-mail: hkfun@usm.my

Key indicators

Single-crystal X-ray study
T = 293 K
Mean $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$
R factor = 0.047
wR factor = 0.128
Data-to-parameter ratio = 10.4

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

2a,8a-Dichloro-2a,8a-dihydro-1-phenyl-dichlorocyclobuta[b]naphthalene-3,8-dione

In the title compound, $\text{C}_{18}\text{H}_{10}\text{Cl}_2\text{O}_2$, the tetrahydrobenzene ring adopts a boat conformation, and the cyclobutene ring is almost coplanar with the attached phenyl ring. There are two intramolecular $\text{C}-\text{H}\cdots\text{O}$ interactions, each forming a five-membered ring. The crystal structure is stabilized by intermolecular $\text{C}-\text{H}\cdots\text{O}$ and $\pi-\pi$ interactions.

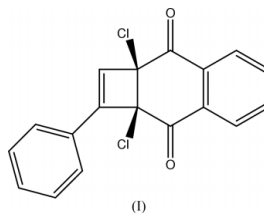
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Comment

In our recent investigations of photoinduced reactions of α -dicarbonyl compounds with alkynes, we have studied the reaction of 2,3-dichloro-1,4-naphthoquinone with phenylacetylene. The title compound, (I), the main product in this reaction, is formed by [2 + 2]-cycloaddition of the triplet-excited 2,3-dichloronaphthoquinone to phenylacetylene. An X-ray crystallographic analysis was undertaken to study the stereochemistry of (I).



The bond lengths and angles in (I) are within normal ranges (Allen *et al.*, 1987) and agree with the corresponding values in related structures, except for the C8—C11 bond length [1.602 (4) Å]. This is longer than the corresponding value in the related structure 2a',8a'-dichloro-3,8-dioxo-1',2',2a',-8a'-tetrahydrodispiro[cyclopropane-1,1'-cyclobuta[b]naphthalene-2',1''-cyclopropane] (Usman *et al.*, 2002), due to the repulsion of the lone pairs of electrons on the two attached chlorine substituents. The C8—C9 bond length is greater than the corresponding C10—C11 bond length (Table 1), due to π -conjugation of the phenyl ring attached at C9.

The tetrahydrobenzene ring adopts a boat conformation, as observed in the related structure (Usman *et al.*, 2002), with atoms C7 and C12 deviating from the C1/C6/C8/C11 plane by 0.254 (4) and 0.305 (7) Å, respectively. The cyclobutene ring (C8—C11) is almost coplanar with the attached phenyl ring (C13—C18), the dihedral angle being 2.9 (2)°. The cyclobutene ring also makes a dihedral angle of 87.9 (2)° with the C1—C6 benzene ring.

In the title structure, there are two intramolecular hydrogen-bond interactions, *viz.* C2—H2 \cdots O1 and C5—H5 \cdots O2 (Fig. 1), forming two closed five-membered rings. In the crystal structure, the molecules are linked by weak C10—H10 \cdots O2ⁱ [symmetry code: (i) $x - 1, y, z$] interactions into

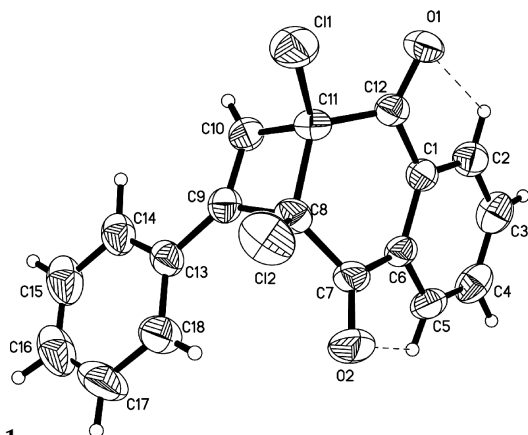


Figure 1

The structure of compound (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. Intramolecular interactions are indicated by dashed lines.

ribbons parallel to the *a* axis (Table 2 and Fig. 2). The packing is also stabilized by π - π interactions involving the centroids of the benzene rings: (a) $Cg4(x, y, z) \cdots Cg4(-x, 3-y, -z)$ at 3.626 Å (*Cg4* is the centroid of ring C13–C18) and (b) $Cg2(x, y, z) \cdots Cg2(-x, 3-y, 1-z)$ at 3.759 Å (*Cg2* is the centroid of ring C1–C6).

Experimental

The title compound was prepared by the photolysis of an acetonitrile solution of 2,3-dichloro-1,4-naphthoquinone, in the presence of an excess amount of phenylacetylene, followed by chromatographic separation of the reaction mixture on a silica-gel column with petroleum ether (b.p. 333–363 K)–ethyl acetate as eluants. A single crystal suitable for X-ray crystallographic analysis was prepared by slow evaporation of solvents from a solution of (I) in petroleum ether (b.p. 333–363 K)–acetone (10:1, v/v).

Crystal data

$C_{18}H_{10}Cl_2O_2$	$Z = 2$
$M_r = 329.16$	$D_x = 1.508 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 7.1114 (7) \text{ \AA}$	Cell parameters from 2166 reflections
$b = 8.3052 (8) \text{ \AA}$	$\theta = 2.7\text{--}28.3^\circ$
$c = 13.8250 (13) \text{ \AA}$	$\mu = 0.45 \text{ mm}^{-1}$
$\alpha = 77.338 (2)^\circ$	$T = 293 (2) \text{ K}$
$\beta = 81.395 (2)^\circ$	Block, colourless
$\gamma = 65.793 (2)^\circ$	$0.42 \times 0.40 \times 0.06 \text{ mm}$
$V = 724.98 (12) \text{ \AA}^3$	

Data collection

Siemens SMART CCD area-detector diffractometer	2482 independent reflections
ω scans	2009 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.017$
$T_{\text{min}} = 0.833$, $T_{\text{max}} = 0.974$	$\theta_{\text{max}} = 25.0^\circ$
3542 measured reflections	$h = -8 \rightarrow 8$
	$k = -9 \rightarrow 9$
	$l = -16 \rightarrow 13$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.055P)^2 + 0.345P]$
$R[F^2 > 2\sigma(F^2)] = 0.047$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.128$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.15$	$\Delta\rho_{\text{max}} = 0.28 \text{ e \AA}^{-3}$
2482 reflections	$\Delta\rho_{\text{min}} = -0.24 \text{ e \AA}^{-3}$
239 parameters	
All H-atom parameters refined	

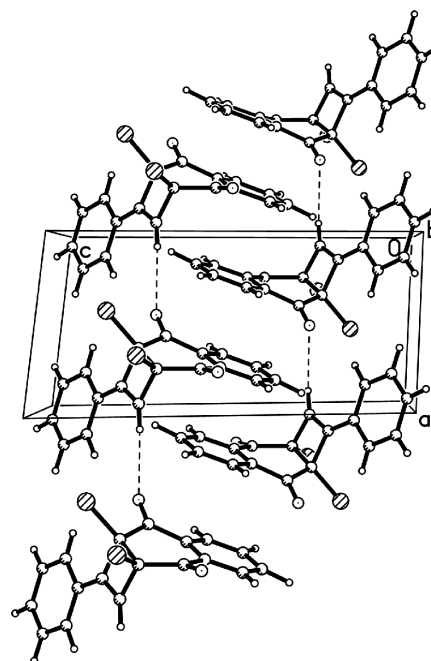


Figure 2

A packing diagram of compound (I), showing ribbons in the *a*-axis direction. Dashed lines denote C–H \cdots O intermolecular interactions.

Table 1

Selected geometric parameters (Å, °).

C8–C9	1.534 (4)	C9–C10	1.328 (4)
C8–C11	1.602 (4)	C10–C11	1.491 (4)
C12–C8–C11–C12	122.6 (2)		

Table 2

Hydrogen-bonding geometry (Å, °).

<i>D</i> –H \cdots <i>A</i>	<i>D</i> –H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> –H \cdots <i>A</i>
C2–H2 \cdots O1	0.91 (3)	2.51 (3)	2.822 (4)	100 (2)
C5–H5 \cdots O2	0.88 (4)	2.52 (3)	2.822 (5)	101 (3)
C10–H10 \cdots O2 ⁱ	0.94 (4)	2.50 (4)	3.379 (5)	156 (3)

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

All H atoms were located in difference Fourier maps and were refined isotropically. As a result of the large fraction of weak data at higher angles, the 2θ maximum was limited to 50° .

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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