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Key indicators

Single-crystal X-ray study
T = 293 K
Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
R factor = 0.036
wR factor = 0.100
Data-to-parameter ratio = 9.7

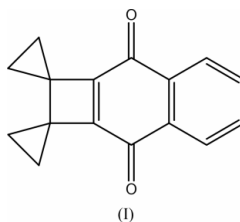
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Dispiro[cyclopropane-1,1'(2'H)-cyclobuta[b]-naphthalene-2',1''-cyclopropane]-3',8'-dione

The title compound, $\text{C}_{16}\text{H}_{12}\text{O}_2$, contains two independent molecules in the asymmetric unit. In both molecules, the benzoquinone system is essentially planar and the cyclobutene ring is coplanar with it; the cyclopropyl planes are orthogonal to the cyclobutene ring. The crystal packing is stabilized by $\text{C}-\text{H}\cdots\text{O}$ interactions as well as van der Waals forces.

Comment

We have recently investigated the photo-induced reactions of a series of quinones with bicyclopropylidene (Wang *et al.*, 2003; Usman *et al.*, 2002). During this investigation, we carried out photoreactions of 2-chloronaphthoquinone with bicyclopropylidene and obtained the title compound, (I), as one of the products. This compound was formed by the dehydrochlorination of the primary [2 + 2]-cycloadduct of these two reactants. An X-ray crystallographic analysis of (I) was undertaken to establish the structure and stereochemistry.



The structure of (I) consists of two crystallographically independent molecules *A* and *B* in the asymmetric unit of the non-centrosymmetric space group $P2_1$ (Fig. 1). The bond lengths and angles of *A* and *B* (Table 1) agree with each other and are within normal ranges (Allen *et al.*, 1987). In both molecules, the quinone ring is essentially planar, and the dihedral angles between this plane and that of the fused benzene ring are 3.95 (10) and 2.48 (12) $^\circ$ in molecules *A* and *B*, respectively. The cyclobutene plane makes dihedral angles of 3.38 (13) and 1.35 (13) $^\circ$ with the benzoquinone ring in *A* and *B*, in contrast with the corresponding value of 73.5 (2) $^\circ$ in a related structure (Wang *et al.*, 2003). This is because the $\text{C}8=\text{C}11$ double bond makes the cyclobutene ring and the benzoquinone system essentially coplanar.

In both *A* and *B*, the configurations of two cyclopropyl rings with respect to the cyclobutene are determined by the $\text{C}16\text{A}-\text{C}9\text{A}-\text{C}10\text{A}-\text{C}14\text{A}$ and $\text{C}15\text{A}-\text{C}9\text{A}-\text{C}10\text{A}-\text{C}13\text{A}$ torsion angles [-3.6 (3) and -4.5 (4) $^\circ$] in *A*, and the corresponding $\text{C}16\text{B}-\text{C}9\text{B}-\text{C}10\text{B}-\text{C}14\text{B}$ and $\text{C}15\text{B}-\text{C}9\text{B}-\text{C}10\text{B}-\text{C}13\text{B}$ torsion angles [3.9 (3) and 3.2 (3) $^\circ$] in *B*. This implies that atoms C9 and C10 are eclipsed, as observed in the related structure (Wang *et al.*, 2003). The two cyclopropyl rings are

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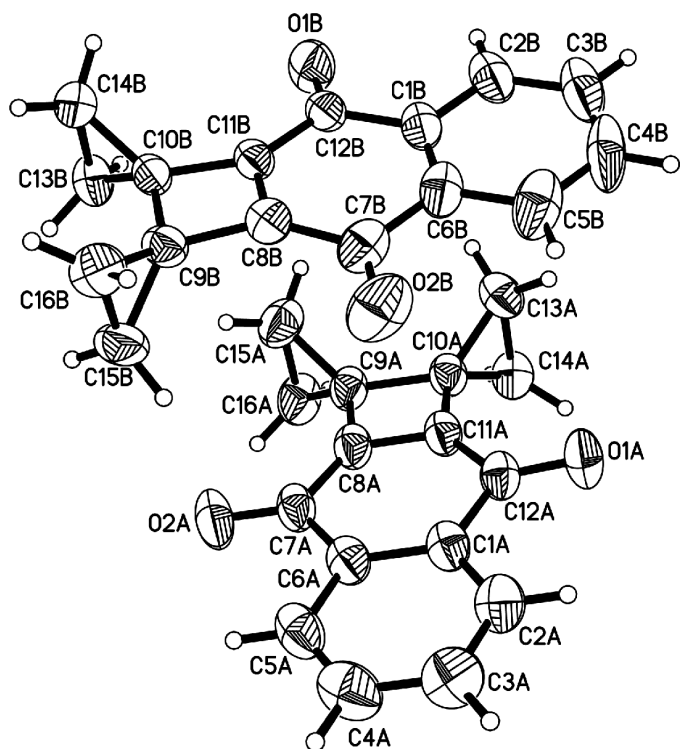


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

orthogonal to the cyclobutene ring, with dihedral angles of 89.1 (2) and 89.5 (2)° in *A*, 88.7 (2) and 89.7 (2)° in *B*. Meanwhile the two cyclopropyl rings are almost perpendicular to one another, with dihedral angles of 87.5 (3)° in *A* and 86.3 (3)° in *B*.

In the crystal structure of (I), molecules *A* and *B* are interconnected by C15B—H15D···O2A and C4A—H4A···O1B(1 + *x*, 1 + *y*, *z*) interactions (Fig. 2 and Table 2). The packing is stabilized by these interactions, as well as van der Waals forces.

Experimental

The title compound was prepared by photolysis of a benzene solution of 2-chloronaphthoquinone in the presence of an excess amount of bicyclopopylidene, followed by silica-gel column chromatographic separation of the reaction mixture with petroleum ether (b.p. 333–363 K)–ethyl acetate as eluants. Single crystals suitable for X-ray crystallographic analysis were grown by slow evaporation of a solution in petroleum ether (b.p. 333–363 K)–acetone (5:1, *v/v*).

Crystal data

$C_{16}H_{12}O_2$	$D_x = 1.309 \text{ Mg m}^{-3}$
$M_r = 236.26$	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 4723 reflections
$a = 8.7281$ (5) Å	$\theta = 2.4\text{--}28.2^\circ$
$b = 8.2720$ (4) Å	$\mu = 0.09 \text{ mm}^{-1}$
$c = 16.9772$ (9) Å	$T = 293$ (2) K
$\beta = 102.0473$ (9)°	Block, yellow
$V = 1198.74$ (11) Å ³	$0.70 \times 0.34 \times 0.24 \text{ mm}$
$Z = 4$	

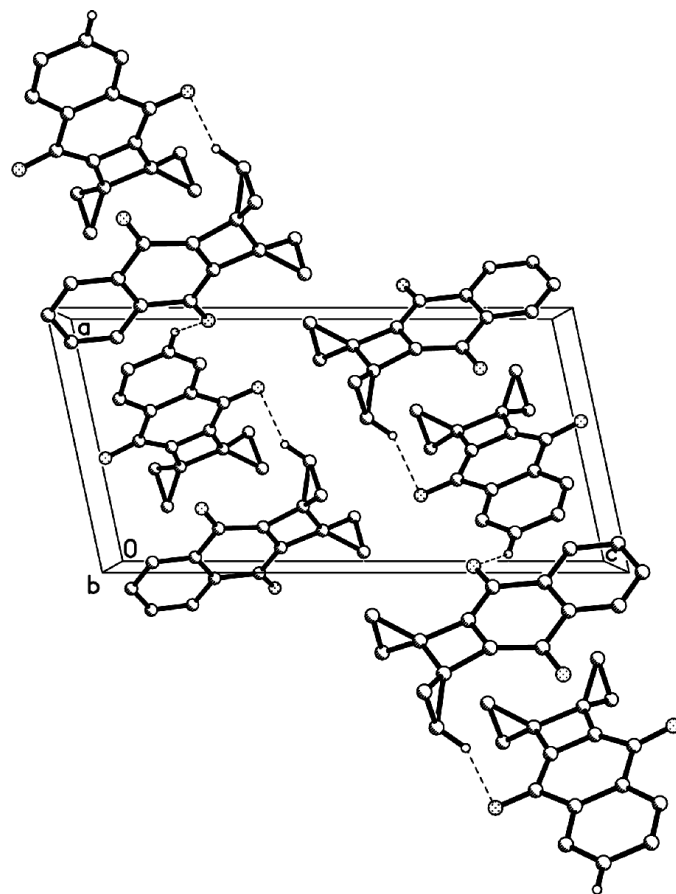


Figure 2
Packing diagram for (I), showing C—H···O interactions between molecules *A* and *B*. H atoms not involved in the interactions have been omitted.

Data collection

Siemens SMART CCD area-detector diffractometer	3147 independent reflections
ω scans	2797 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.015$
$T_{\text{min}} = 0.943$, $T_{\text{max}} = 0.980$	$\theta_{\text{max}} = 28.3^\circ$
7540 measured reflections	$h = -11 \rightarrow 11$
	$k = -8 \rightarrow 10$
	$l = -22 \rightarrow 18$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0566P)^2 + 0.1341P]$
$R[F^2 > 2\sigma(F^2)] = 0.036$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.100$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.04$	$\Delta\rho_{\text{max}} = 0.17 \text{ e \AA}^{-3}$
3147 reflections	$\Delta\rho_{\text{min}} = -0.17 \text{ e \AA}^{-3}$
324 parameters	
H-atom parameters constrained	

Table 1

Selected geometric parameters (Å).

O1A—C12A	1.224 (2)	O1B—C12B	1.219 (3)
O2A—C7A	1.225 (2)	O2B—C7B	1.215 (3)
C1A—C6A	1.424 (3)	C1B—C6B	1.401 (3)
C1A—C12A	1.500 (3)	C1B—C12B	1.501 (3)
C6A—C7A	1.494 (3)	C6B—C7B	1.516 (4)
C7A—C8A	1.463 (3)	C7B—C8B	1.460 (3)
C8A—C11A	1.362 (2)	C8B—C11B	1.343 (3)
C11A—C12A	1.459 (3)	C11B—C12B	1.460 (3)

Table 2

Geometry of C—H···O interactions (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>
C4A—H4A···O1B ⁱ	0.93	2.58	3.387 (3)	145
C15B—H15D···O2A	0.97	2.56	3.216 (3)	125

Symmetry code: (i) 1 + *x*, 1 + *y*, *z*.

All H atoms were positioned geometrically and treated as riding on their parent C atoms, with C—H = 0.93–0.97 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The Friedel reflections were merged before the final refinement because of the absence of significant anomalous scattering effects.

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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