2a′,8a′-Dichloro-3,8-dioxo-1′,2′,2a′,8a′-tetra-hydrodispiro[cyclopropane-1,1′-cyclobuta[b]-naphthalene-2′,1″-cyclopropane]

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In the title compound, $C_{16}H_{12}Cl_2O_2$, the tetrahydrobenzene ring adopts a boat conformation, and both cyclopropyl groups are orthogonal to the cyclobutane ring. The cyclobutane and tetrahydrobenzene rings share one C—C bond, and are folded about this bond relative to each other.

Comment

Photo-induced electron transfer (PET) reactions of the electron donor bicyclopropylidene and its derivatives have not been widely investigated. With our continuing studies into the PET reactions of electron-rich compounds, we have carried out PET reactions of bicyclopropylidene with several quinones (Wang & Xu, 2002). In this study, we have isolated the title compound, (I), and undertaken its X-ray crystal structure analysis in order to elucidate its configuration and conformation.

(I)
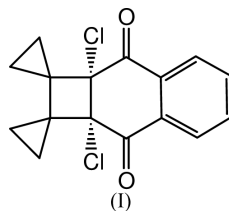
The bond lengths and angles in (I) (Fig. 1) are within normal ranges (Allen *et al.*, 1987). Those within the dioxotetrahydrocyclobuta[b]naphthalene moiety (O1/O2/C1–C12) agree with the corresponding values in a related structure (Taira *et al.*, 1993), except for the C1—C10 bond length [1.587 (3) Å], which is slightly longer than the corresponding value in the related structure, mainly due to the repulsions of the lone pair of electrons of the two attached chlorine substituents.

In (I), the benzo group is not coplanar with the tetrahydrobenzene ring, which is distorted slightly towards a boat conformation. Atoms C2 and C9 are displaced from the C1/C3/C8/C10 plane by 0.339 (2) and 0.202 (2) Å, respectively, and the dihedral angle between the tetrahydrobenzene and benzene rings is 9.5 (1)°.

The cyclobutane moiety (C1/C10–C12) is slightly out of planarity, with the atoms deviating from its mean plane by ±0.042 (3); the dihedral angle of the C1/C10/C12 and C10/C11/C12 planes is 171.2 (2)°. The cyclobutane mean plane makes a dihedral angle of 78.7 (1)° with the tetrahydrobenzene ring, as a result of the sp^3 hybridization of the atoms C1 and C10.

The configurations of the two cyclopropyl rings (C11/C13/C14 and C12/C15/C16) with respect to the cyclobutane are determined by the C13—C11—C12—C15 and C14—C11—
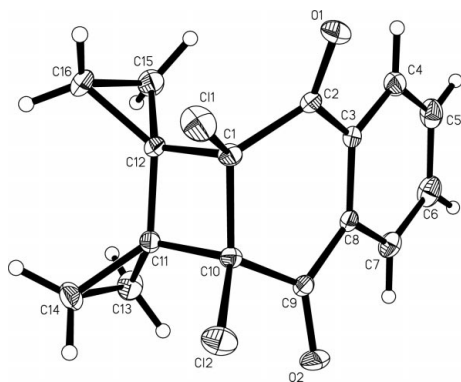
Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

C12—C16 torsion angles [both are 9.2 (2)°], implying that atoms C11 and C12 are eclipsed. The Cl1—C1—C10—Cl2 torsion angle is 9.1 (2)°, also indicating an eclipsed configuration around atoms C1 and C10. The two cyclopropane rings are orthogonal to the cyclobutane ring, with dihedral angles of 89.4 (2) and 89.5 (2)°. In the crystal, molecules are linked into extended chains by weak intermolecular C—H···O hydrogen bonds, *via* unit-cell translations in the **a** direction (see Fig. 2 and Table 2).

Experimental

The title compound was prepared by the photo-induced reaction of 2,3-dichloronaphtho-1,4-quinone with an excess of bicyclopropylidene in benzene solution, and was isolated by column chromatography on silica gel. Single crystals suitable for X-ray diffraction study were obtained from slow evaporation of a petroleum ether–ethyl acetate (15:1 *v/v*) solution.

Crystal data

$C_{16}H_{12}Cl_2O_2$
$M_r = 307.16$
Triclinic, $P\bar{1}$
$a = 7.2430$ (1) Å
$b = 10.0455$ (2) Å
$c = 10.0831$ (2) Å
$\alpha = 85.4658$ (8)°
$\beta = 73.306$ (1)°
$\gamma = 69.401$ (1)°
$V = 657.59$ (2) Å^3

$Z = 2$
$D_x = 1.551$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3828 reflections
$\theta = 3.1–28.3°$
$\mu = 0.49$ mm^{-1}
$T = 213$ (2) K
Block, colorless
$0.50 \times 0.46 \times 0.40$ mm

Data collection

Siemens SMART CCD area-detector
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.792$, $T_{max} = 0.828$
3750 measured reflections

2753 independent reflections
2464 reflections with $I > 2\sigma(I)$
$R_{int} = 0.066$
$\theta_{max} = 27.0°$
$h = -8 \rightarrow 9$
$k = -12 \rightarrow 11$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.078$
$wR(F^2) = 0.199$
$S = 1.07$
2753 reflections
182 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.125P)^2]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.97$ e Å^{-3}
$\Delta\rho_{min} = -1.22$ e Å^{-3}
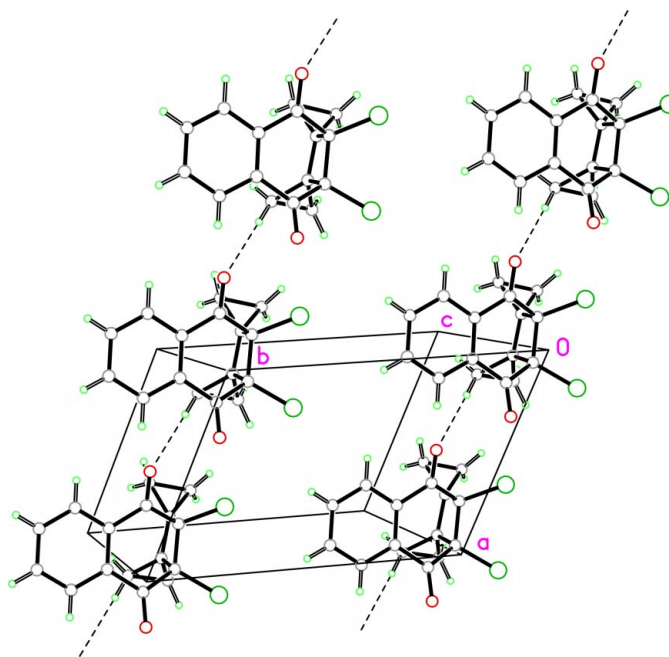Extinction correction: *SHELXTL*
Extinction coefficient: 0.68 (5)

Figure 2
Packing diagram of compound (I), showing extended chains in the **a** direction. Dashed lines denote C—H···O intermolecular interactions.

Table 1
Selected geometric parameters (Å).

C1—C12	1.543 (3)	C11—C12	1.505 (3)
C10—C11	1.542 (3)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C15—H15B···O2^i	0.97	2.42	3.384 (4)	172

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

The H atoms were fixed geometrically and treated as riding on the parent C atoms, with C—H = 0.93–0.97 Å and U_{iso}(H) = 1.2 U_{eq}(C). The maximum and minimum electron-density peaks are 0.06 Å from Cl1 and 0.92 Å from Cl2, respectively.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT* and *SADABS* (Sheldrick, 1996); 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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