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2a′-Chloro-3,8-dioxo-1′,2′,2a′,8a′-tetrahydrodispiro[cyclopropane-1,1′-cyclobuta[*b*]napthalene-2′,1″-cyclopropane]

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Key indicators

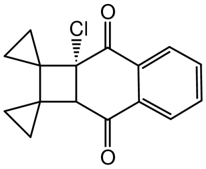
Single-crystal X-ray study
T = 213 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.065
wR factor = 0.168
Data-to-parameter ratio = 17.1

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

In the title compound, $C_{16}H_{13}ClO_2$, the tetrahydrobenzene ring adopts a boat conformation, and both cyclopropyl groups are orthogonal to the cyclobutane ring. The Cl atom shows positional disorder.

Comment

Recently, we have carried out photo-induced electron transfer (PET) reactions of bicyclopropylidene with several quinones (Wang & Xu, 2002), giving dispiro[cyclopropane] derivatives. The crystal structure of one of these derivatives has been reported (Usman *et al.*, 2002). In our continuing studies into these PET reactions, we have isolated the title compound, (I). An X-ray crystal structure analysis was undertaken to elucidate its molecular structure.



(I)

The bond lengths and angles in (I) (Fig. 1) agree with the corresponding values in related structures (Usman *et al.*, 2002; Taira *et al.*, 1993). The tetrahydrobenzene ring is non- planar, adopting a boat conformation, as observed in the related structures. Atoms C2 and C9 are displaced from the C1/C3/C8/C10 plane by 0.167 (3) and 0.199 (3) Å, respectively, and the dihedral angle between the latter plane and that of the fused benzene ring is 8.9 (1)°. The cyclobutane ring (C1/C10–C12) is slightly non-planar, with the atoms deviating from its mean plane by ±0.052 (3) Å; the dihedral angle between the C1/C10/C12 and C10/C11/C12 planes is 169.3 (2)°. The cyclobutane mean plane makes a dihedral angle of 74.0 (2)° with the C1/C8/C10/C13 plane.

The configurations of the two cyclopropyl rings (C11/C13/C14 and C12/C15/C16) with respect to the cyclobutane are characterized by the C13—C11—C12—C15 and C14—C11—C12—C16 torsion angles [10.1 (5) and 9.5 (5)°, respectively], implying that atoms C11 and C12 are eclipsed. The two cyclopropyl rings are orthogonal to the cyclobutane ring, with dihedral angles of 89.2 (3) and 89.0 (3) Å.

The Cl atom shows positional disorder, bonded to C10 or C1, with site occupancies of 0.86 and 0.14, respectively.

Experimental

The title compound was prepared by the photo-induced reaction of 2-chloronaphtho-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 by slow evaporation of a petroleum ether–ethyl acetate (20:1 *v*/*v*) solution.

Crystal data

$C_{16}H_{13}ClO_2$
$M_r = 272.71$
Monoclinic, $P2_1/n$
$a = 8.2794\ (2)$ Å
$b = 15.3344\ (2)$ Å
$c = 10.2590\ (2)$ Å
$\beta = 96.639\ (1)°$
$V = 1293.74\ (4)$ Å^3
$Z = 4$

$D_x = 1.400$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4040 reflections
$\theta = 2.7–28.4°$
$\mu = 0.29$ mm^{-1}
$T = 213\ (2)$ K
Slab, yellow
0.34 × 0.34 × 0.06 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.934$, $T_{\max} = 0.983$
7752 measured reflections

3140 independent reflections
1736 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.126$
$\theta_{\max} = 28.3°$
$h = -10 \rightarrow 11$
$k = -20 \rightarrow 16$
$l = -12 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.168$
$S = 0.93$
3133 reflections
183 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0576P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.64$ e Å^{-3}
$\Delta\rho_{\min} = -0.55$ e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.066 (9)

The H atoms were fixed geometrically and treated as riding on their parent C atoms, with C—H = 0.93–0.97 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$. The Cl atom was found to be disordered over two positions, Cl1 and Cl1*A*, bonded to C10 or C1 with occupancies of 0.864 (2) and 0.136 (2), respectively. During the refinement, similarity restraints were applied to the C10—Cl1 and C1—Cl1*A* distances and seven reflections showing a bad fit were suppressed. Owing to the poor diffraction quality of the crystal, the $R_{\rm int}$ value is high (0.126).

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, 2003).

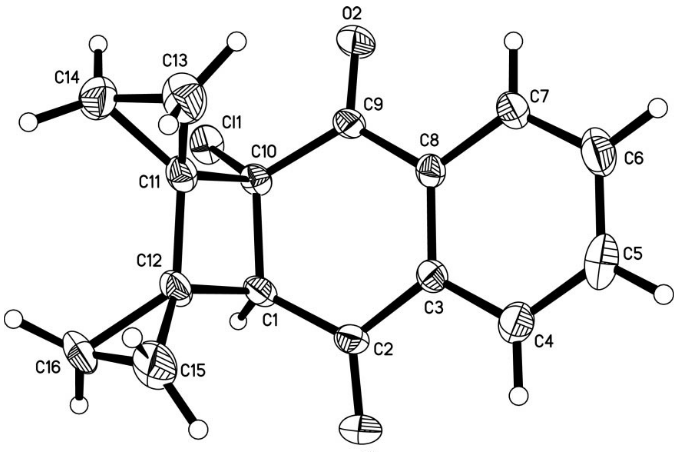


Figure 1
The structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme. For clarity, only the major component of the disordered Cl atom is shown.

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