organic papers

Shu-Sheng Zhang,ᵃ Min Zhang,ᵇ Jian-Hua Xu,ᵇ Xue-Mei Liᵃ and Hoong-Kun Funᶜ*

ᵃCollege of Chemistry and Molecular Engineering, Qingdao University of Science and Technology, 266042 Qingdao, Shandong, People's Republic of China, ᵇDepartment of Chemistry, Nanjing University, 210093 Nanjing, 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 σ(C–C) = 0.002 Å
R factor = 0.029
wR factor = 0.084
Data-to-parameter ratio = 15.7

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

4b,4c,9b,9c-Tetrahydro-4b,4c,9b,9c-tetrachloro-cyclobuta[1,2-a:3,4-a']diindene-5,10-dione

In the title compound, $C_{18}H_8Cl_4O_2$, a crystallographic twofold rotation axis passes through the centroid of the cyclobutane ring. The indanone moieties are each planar and each forms a dihedral angle of 66.68 (8)° with the cyclobutane ring. O1···Cl1 short contacts link symmetry-related molecules into chains along the c axis.

Comment

We have recently investigated the photoinduced dimerization reaction of 2,3-dichloroindenone, and obtained the title compound, (I), as one of the products. An X-ray crystallographic analysis was undertaken to establish the structure and configuration of (I), which turns out to be a *syn*-head-to-tail dimer.
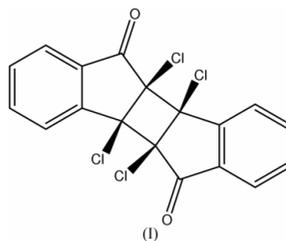
The asymmetric unit of (I) contains half the molecule, the other half being related by a crystallographic twofold axis passing through the centroid of the cyclobutane ring (C8/C9/C8ⁱ/C9ⁱ) [symmetry code: (i) $1 - x, y, \frac{1}{2} - z$]. The bond lengths and angles in (I) show normal values (Allen *et al.*, 1987), except for the C8ⁱ—C9 bond length [1.5885 (19) Å]. Owing to the presence of Cl atoms, this bond is longer than the mean Csp^3—Csp^3 distance [1.554 (21) Å] reported for cyclobutanes by Allen *et al.* (1987).

In the structure of (I), the indanone moiety is essentially planar, with atom O1 deviating from the mean plane by 0.112 (4) Å. The dihedral angle between the five-membered ring and the fused benzene ring is 1.32 (9)°, and the mean plane of the indanone moiety (C1–C9/O1) makes a dihedral angle of 66.68 (8)° with the cyclobutane ring.

The two Cl atoms attached to the indanone moiety lie on the same side of the indanone plane. The *syn* configurations of these two Cl atoms are dictated by the sp^3 character of the adjacent atoms.

In the packing of the title compound, the molecules are linked into chains along the c axis (Fig. 2) by Cl1···O1(x, $1 - y, z - \frac{1}{2}$) [3.176 (1) Å] interactions. These short contacts, together with van der Waals forces, stabilize the crystal structure.
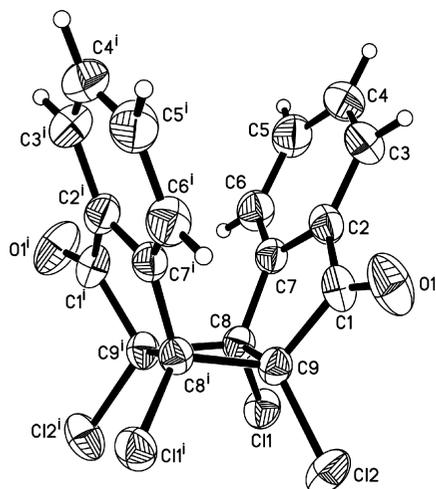
Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. [Symmetry code: (i) $1 - x$, y, $\frac{1}{2} - z$.]

Experimental

The title compound was synthesized by irradiation of a benzene solution of 2,3-dichloroindenone, 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. The title compound was obtained as the main product, together with a small amount of its *anti* isomer. Single crystals suitable for X-ray crystallographic analysis were grown by slow evaporation of a petroleum ether (b.p. 333–363 K)–chloroform (3:7, *v*/*v*) solution.

Crystal data

$C_{18}H_8Cl_4O_2$	$D_x = 1.634$ Mg m^{-3}
$M_r = 398.04$	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 3658
$a = 15.8948\ (10)$ Å	reflections
$b = 8.7186\ (5)$ Å	$\theta = 2.8–28.3°$
$c = 13.5633\ (9)$ Å	$\mu = 0.74$ mm^{-1}
$\beta = 120.563\ (1)°$	$T = 293\ (2)$ K
$V = 1618.47\ (17)$ Å^3	Block, colourless
$Z = 4$	$0.52 \times 0.38 \times 0.26$ mm

Data collection

Siemens SMART CCD area-detector diffractometer	1972 independent reflections
ω scans	1773 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$R_{int} = 0.016$
$T_{min} = 0.700$, $T_{max} = 0.831$	$\theta_{max} = 28.3°$
4882 measured reflections	$h = -19 \rightarrow 21$
	$k = -10 \rightarrow 11$
	$l = -17 \rightarrow 12$

Refinement

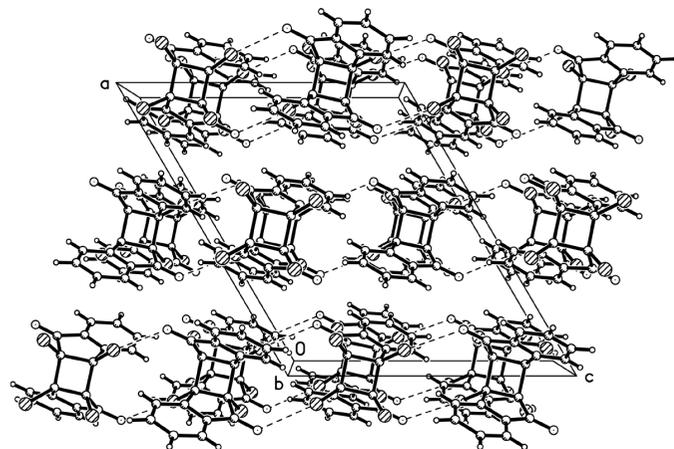|	
Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0468P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.029$	$+ 0.9124P]$
$wR(F^2) = 0.085$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} < 0.001$
1972 reflections	$\Delta\rho_{max} = 0.31$ e Å^{-3}
126 parameters	$\Delta\rho_{min} = -0.22$ e Å^{-3}
All H-atom parameters refined	Extinction correction: *SHELXTL*
	Extinction coefficient: 0.0106 (9)

Figure 2
The molecular packing of (I), showing the chains along the *c* axis. The dashed lines denote the short-contact interactions.

Table 1
Selected geometric parameters (Å, °).

Cl1—C8	1.7632 (14)	C7—C8	1.5155 (19)
Cl2—C9	1.7524 (14)	C8—C9	1.5648 (19)
C1—C9	1.540 (2)	C8—C9^i	1.5885 (19)
C7—C8—C9	103.38 (11)	C7—C8—Cl1	110.71 (10)
C7—C8—C9^i	116.42 (11)	C9—C8—Cl1	119.31 (10)
C9—C8—C9^i	90.54 (10)	C1—C9—C8	106.70 (11)
Cl1—C8—C9—C1	116.58 (11)	C7—C8—C9—Cl2	−132.06 (10)

Symmetry code: (i) $1 - x$, y, $\frac{1}{2} - z$.

All H atoms were located in difference Fourier maps and their x, y, z and U_{iso} parameters were refined. The C—H distances lie in the range 0.92 (3)–1.00 (3) Å.

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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