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1,2,3,4,4b,4c,6,7,8,9,9b,9c-Dodecachloro-4b,4c,9b,9c-tetrahydrocyclobuta[1,2-*a*;3,4-*a*′]-diindene-5,10-dione

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Key indicators

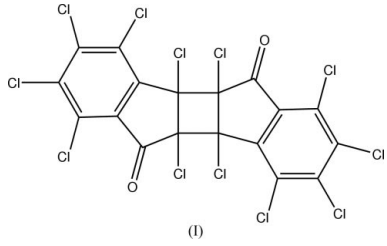
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.032
wR factor = 0.090
Data-to-parameter ratio = 17.5

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

The title compound, $C_{18}Cl_{12}O_2$, was synthesized by a solvothermal reaction between sodium and carbon tetrachloride; the oxygen may come from air. The molecule has crystallographic C_2 symmetry and contains a four-membered ring, two five-membered rings and two six-membered rings. The five-membered rings are attached to opposite sides of the cyclobutane group and the six-membered rings are fused to the five-membered rings. In the solid state, the molecule adopts a boat form.

Comment

The solvothermal method was used for the first time to synthesize an inorganic complex by Salta *et al.* (1994). Since then, it has been mainly applied to synthesizing nanometer materials and coordination complex polymers (Wang et al., 1999). The first example of the synthesis of an organic compound using this method was carried out by Peng et al. (2001). We report here the synthesis and crystal structure of a new compound synthesized by the solvothermal method. In the title compound, (I), the structure comprises a four-membered ring, two five-membered rings and two six-membered rings. The five-membered rings are attached to opposite sides of the cyclobutane group, and the six-membered rings are fused to the five-membered rings. The molecule lies on a crystallographic twofold axis, which passes through the center of, and is perpendicular to, the cyclobutane ring. In the solid state, the molecule has a boat form.



(I)

Experimental

Metallic sodium (3.0 g) was put into a stainless-steel autoclave filled with carbon tetrachloride (25 ml). The autoclave volume was 40 ml and was about 75% filled, and so contained air, which is most likely the source of oxygen in the product. The autoclave was heated to 573 K, maintained at this temperature for 40 h and then allowed to cool to room temperature. The resulting dark powder product was washed with water many times and dried in a vacuum at room temperature. The dried product was extracted with cyclohexane. The extract was separated with column chromatography on active alumina, using cyclohexane as the eluant. Yellow crystals were

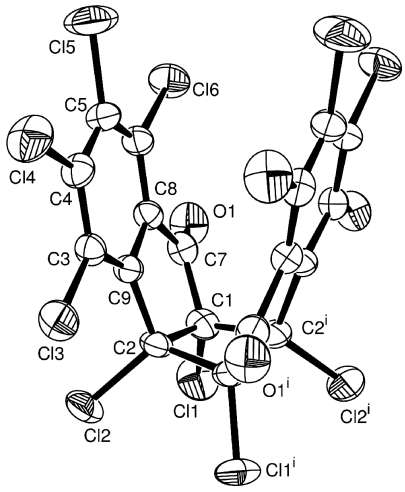


Figure 1
*ORTEP*II (Johnson, 1976) plot of (I). Displacement ellipsoids are drawn at the 50% probability level. [Symmetry code: (i) $1-x, y, \frac{1}{2}-z$.]

obtained from the first yellow component upon slow evaporation of cyclohexane in air. The crystals were analyzed by mass spectrometry. The molecular peak appeared at a mass/charge ratio of 674. The isotopic distribution pattern of chlorine shows that the molecule contains 12 Cl atoms.

Crystal data

$C_{18}Cl_{12}O_2$
$M_r = 673.58$
Orthorhombic, *Pbcn*
$a = 9.0245\ (18)$ Å
$b = 18.407\ (4)$ Å
$c = 13.516\ (3)$ Å
$V = 2245.2\ (8)$ Å^3
$Z = 4$
$D_x = 1.993$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1526 reflections
$\theta = 3.3$–26.3°
$\mu = 1.50$ mm^{-1}
$T = 298\ (2)$ K
Prism, yellow
0.32 × 0.12 × 0.10 mm

Data collection

Bruker SMART APEX 2000 diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.646$, $T_{max} = 0.865$
20 533 measured reflections
2562 independent reflections
2109 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 27.5^\circ$
$h = -11 \rightarrow 11$
$k = -23 \rightarrow 21$
$l = -15 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.090$
$S = 1.05$
2562 reflections
146 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0481P)^2 + 0.8462P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.46$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0023 (3)

Table 1
Selected geometric parameters (Å, °).

Cl1—C1	1.739 (2)	C1—C2^{i}	1.594 (3)
Cl2—C2	1.752 (2)	C2—C9	1.518 (3)
Cl3—C3	1.706 (2)	C3—C9	1.384 (3)
Cl4—C4	1.712 (2)	C3—C4	1.399 (3)
Cl5—C5	1.710 (2)	C4—C5	1.399 (3)
Cl6—C6	1.713 (2)	C5—C6	1.386 (3)
O1—C7	1.199 (3)	C6—C8	1.389 (3)
C1—C7	1.524 (3)	C7—C8	1.485 (3)
C1—C2	1.565 (3)	C8—C9	1.391 (3)
C7—C1—C2	105.63 (16)	C5—C4—Cl4	119.21 (16)
C7—C1—C2^{i}	116.03 (17)	C6—C5—C4	119.85 (18)
C2—C1—C2^{i}	88.02 (15)	C6—C5—Cl5	119.93 (17)
C7—C1—Cl1	112.43 (15)	C4—C5—Cl5	120.21 (17)
C2—C1—Cl1	117.71 (15)	C5—C6—C8	119.10 (19)
C2^{i}—C1—Cl1	114.71 (14)	C5—C6—Cl6	120.83 (17)
C9—C2—C1	103.44 (16)	C8—C6—Cl6	119.98 (18)
C9—C2—C1^{i}	113.42 (16)	O1—C7—C8	127.8 (2)
C1—C2—C1^{i}	90.49 (16)	O1—C7—C1	126.2 (2)
C9—C2—Cl2	112.21 (14)	C8—C7—C1	105.92 (17)
C1—C2—Cl2	116.83 (15)	C6—C8—C9	121.08 (19)
C1^{i}—C2—Cl2	117.92 (14)	C6—C8—C7	128.55 (19)
C9—C3—C4	118.66 (18)	C9—C8—C7	110.28 (18)
C9—C3—Cl3	121.33 (16)	C3—C9—C8	120.31 (17)
C4—C3—Cl3	120.00 (16)	C3—C9—C2	128.19 (18)
C3—C4—C5	120.88 (19)	C8—C9—C2	111.31 (17)
C3—C4—Cl4	119.88 (17)		

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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References

Bruker (2001). *SAINT* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Peng, Y., Xie, S. Y., Huang, R. B. & Zheng, L. S. (2001). *Acta Cryst.* E**57**, o617–o618.
Salta, J., Chang, Y. D. & Zubieta, J. (1994). *J. Chem. Soc. Chem. Commun.* pp. 1039–1040.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Wang, R. Z., Li, Y. F., Qiao, X. G., Yang, G. Y., Xing, Y. H., Zeng, Q. X., Xu, J. Q., Lin, Y. H., Xing, Y. & Jia, H. Q. (1999). *Chem. J. Chin. Univ.* **20**, 1186–1188.