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1,2,3,5,6,7,8,9-Octachlorocyclopenta[*def*]-phenanthren-4-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.050
wR factor = 0.110
Data-to-parameter ratio = 14.2

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

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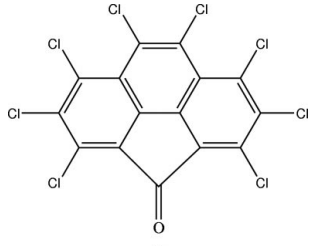
The title compound, $C_{15}Cl_8O$, was separated from the products of a solvothermal reaction of metallic sodium and carbon tetrachloride in air in a pressured autoclave. The molecule is bisected by a crystallographic mirror plane and has essentially C_{2v} symmetry.

Comment

Alkali metals and polyhalogenated alkanes under high pressure/temperature in an autoclave can undergo different reactions under different conditions and give different products, for example, diamond powders from CCl_4 (Li *et al.*, 1998), multi-wall carbon nanotubes and hollow spherical graphite from hexachlorobenzene (Jiang *et al.*, 2000), and carbon concentric spheres ('onions') from hexachloropentadiene (Li *et al.*, 2001). Long-standing interest has been focused on the fabrication of fullerenes, and various techniques, such as high-voltage electric discharge in liquid (Huang *et al.*, 1997) or vaporized (Xie *et al.*, 2001) chloroform and CCl_4, have been used to generate and trap the intermediates of fullerenes. In such a process, we have isolated perchlorinated aromatic hydrocarbons (PCAHs), which can be used as building blocks for fullerenes, and also identified a trace of C_{60} and C_{70} (Xie *et al.*, 2001). On the other hand, under solvothermal conditions, a series of perchlorinated fullerene fragments, such as $C_{26}H_8Cl_{10}$ (Peng *et al.*, 2001) and $C_{18}Cl_{12}O_2$ (Peng *et al.*, 2004), have been obtained and characterized by X-ray diffraction.



(I)

We report here the synthesis and crystal structure of the title compound, (I) (Fig. 1), a new perchlorinated compound, which was separated from the products of a solvothermal reaction. All bond lengths and angles in (I) are normal (Table 1). The molecule is bisected by a crystallographic mirror plane and has essentially C_{2v} symmetry.

Experimental

Metallic sodium (3.0 g) was added to carbon tetrachloride (25 ml) in a stainless-steel autoclave with a capacity of 50 ml. The autoclave was heated to 673 K, maintained at that temperature for 40 h and then allowed to cool to room temperature. The resulting dark powder was

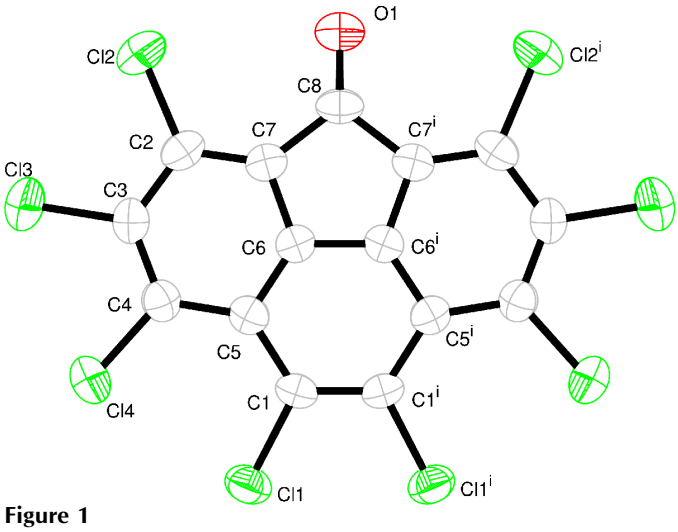


Figure 1
A view of (I), with 50% probability displacement ellipsoids. [Symmetry code: (i) $-x+1, y, z$.]

washed with water several times and dried in a vacuum at room temperature. The dried product was extracted with toluene/cyclohexane in a volume ratio of 1:1. The extract was separated by column chromatography on neutral alumina, using toluene/cyclohexane as eluant. Yellow crystals suitable for X-ray diffraction were obtained from the yellow solution upon slow evaporation of the solvent in air. The product was analyzed by mass spectrometry. The molecular peak appeared at a mass/charge ratio of 480. The isotopic distribution pattern of chlorine shows that the molecule contains eight Cl atoms.

Crystal data

$C_{15}Cl_8O$
$M_r = 479.75$
Orthorhombic, *Cmca*
$a = 22.979\ (5)$ Å
$b = 8.7180\ (17)$ Å
$c = 15.697\ (3)$ Å
$V = 3144.6\ (11)$ Å^3
$Z = 8$
$D_x = 2.027$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 8.0$–$15.0°$
$\mu = 1.43$ mm^{-1}
$T = 293\ (2)$ K
Prism, yellow
0.32 × 0.26 × 0.18 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.657$, $T_{max} = 0.783$
1588 measured reflections
1588 independent reflections
1078 reflections with $I > 2\sigma(I)$
$\theta_{max} = 26.0°$
$h = -28 \rightarrow 0$
$k = 0 \rightarrow 10$
$l = -19 \rightarrow 0$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.110$
$S = 1.04$
1588 reflections
112 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0421P)^2 + 1.1559P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C1	1.717 (4)	C3—C4	1.382 (6)
Cl2—C2	1.713 (4)	C4—C5	1.432 (5)
Cl3—C3	1.724 (4)	C5—C6	1.379 (5)
Cl4—C4	1.706 (4)	C6—C7	1.398 (5)
C1—C1^i	1.392 (8)	C6—C6^i	1.429 (8)
C1—C5	1.450 (5)	C7—C8	1.498 (5)
C2—C7	1.365 (5)	C8—O1	1.206 (7)
C2—C3	1.413 (6)	C8—C7^i	1.498 (5)
C1^i—C1—C5	122.2 (2)	C6—C5—C4	113.8 (4)
C1^i—C1—Cl1	117.36 (13)	C6—C5—C1	114.5 (3)
C5—C1—Cl1	120.4 (3)	C4—C5—C1	131.6 (4)
C7—C2—C3	118.1 (4)	C5—C6—C7	127.0 (4)
C7—C2—Cl2	121.1 (3)	C5—C6—C6^i	123.2 (2)
C3—C2—Cl2	120.8 (3)	C7—C6—C6^i	109.8 (2)
C4—C3—C2	122.9 (4)	C2—C7—C6	118.0 (4)
C4—C3—Cl3	119.8 (3)	C2—C7—C8	134.3 (4)
C2—C3—Cl3	117.3 (3)	C6—C7—C8	107.7 (4)
C3—C4—C5	120.2 (4)	O1—C8—C7	127.5 (2)
C3—C4—Cl4	117.3 (3)	O1—C8—C7^i	127.5 (2)
C5—C4—Cl4	122.4 (3)	C7—C8—C7^i	105.0 (5)

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

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1988); cell refinement: *CAD*-4 *Software*; data reduction: *XCAD*4 (Harms, 1997); 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

Enraf–Nonius (1988). *CAD*-4 *Software*. Enraf–Nonius, Delft, The Netherlands.
Harms, K. (1997). *XCAD*4. University of Marburg, Germany.
Huang, R.-B., Huang, W.-J., Wang, Y.-H., Tang, Z.-C. & Zheng, L.-S. (1997). *J. Am. Chem. Soc.* **117**, 5954–5955.
Jiang, Y., Wu, Y., Zhang, S.-Y., Xu, C.-Y., Yu, W.-C., Xie, Y. & Qian, Y.-T. (2000). *J. Am. Chem. Soc.* **122**, 12383–12384.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Oak Ridge, Tennessee, USA.
Li, C.-Y., Chiu, H.-T., Peng, C.-W., Yen, M.-Y., Chang, Y.-H. & Liu, C.-S. (2001). *Adv. Mater.* **13**, 1105–1107.
Li, Y.-D., Qian, Y.-T., Liao, H.-W., Ding, Y., Yang, L., Xu, C.-Y., Li, F.-Q. & Zhou, G.-E. (1998). *Science*, **281**, 246–247.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Peng, Y., Xie, S.-Y., Huang, R.-B. & Zheng, L.-S. (2001). *Acta Cryst.* E**57**, o617–o618.
Peng, Y., Xie, S.-Y., Long, L.-S., Huang, R.-B. & Zheng, L. S. (2004). *Acta Cryst.* E**60**, o762–o763.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Xie, S.-Y., Deng, S.-L., Yu, L.-J., Huang, R.-B. & Zheng, L.-S. (2001). *J. Phys. Chem. B*, **105**, 1734–1738.