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

Ying Peng,[a] Su-Yuan Xie,[a] Shun-Liu Deng,[a] Rong-Bin Huang[a]* and Lan-Sun Zheng[b]

[a]Department of Chemistry, Xiamen University, Xiamen 361005, People's Republic of China, and [b]State Key Laboratory for Physical Chemistry of Solid Surfaces, Department of Chemistry, Xiamen University, Xiamen 361005, People's Republic of China

Correspondence e-mail: rbhuang@xmu.edu.cn

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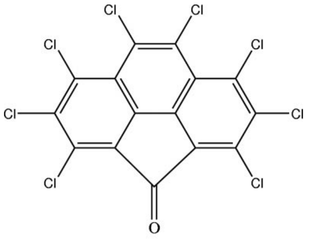
The title compound, $C_{15}Cl_8O$, was separated from the products of a solvothermal reaction of metallic sodium and carbon tetrachloride in air in a pressured autoclave. The molecule is bisected by a crystallographic mirror plane and has essentially C_{2v} symmetry.

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

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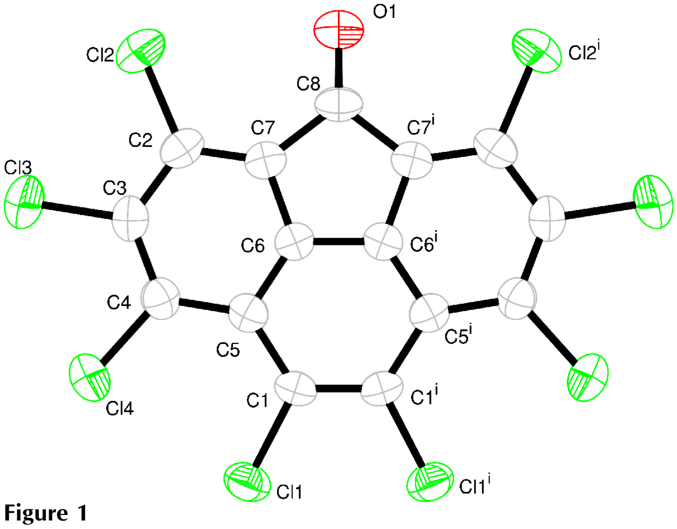

Figure 1
A view of (I), with 50% probability displacement ellipsoids. [Symmetry code: (i) $-x + 1, y, z$.]

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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