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6-(*p*-Tolyl)-6*H*-perchlorobenzo[*cd*]pyrene

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.008 Å
R factor = 0.066
wR factor = 0.124
Data-to-parameter ratio = 13.5

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

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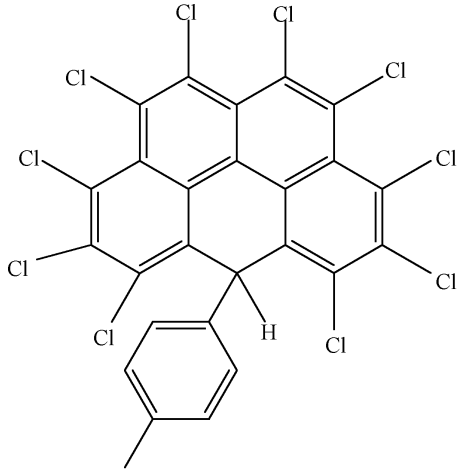
The title compound, $C_{26}H_8Cl_{10}$, was synthesized by high-pressure solvothermal reaction between sodium and carbon tetrachloride. The four fused benzene rings are twisted due to the steric repulsion of overcrowded Cl atoms.

Comment

In order to explore the formation mechanisms of fullerenes by means of trapping a macro amount of fullerene intermediates during their formation, *i.e.* fullerene fragment molecules, from a discharge system, several methods, such as discharge in liquid chloroform (Huang *et al.*, 1997), glow discharge (Xie *et al.*, 1998) and microwave discharge (Xie *et al.*, 1999), have been developed in our research group in recent years. Using these approaches, a series of perchlorinated fullerene fragments, such as perchlorofluoranthene, $C_{16}Cl_{10}$, decachlorocoranulene, $C_{20}Cl_{10}$, have been obtained. Recently, a new perchlorinated compound, (I), was synthesized by solvothermal reaction, another novel approach developed by our group.



(I)

Compound (I) is an interesting molecule as its structure is similar to perchlorobenzofluoanthene, $C_{18}Cl_{10}$, one of the fullerene fragment molecules. On the other hand, (I) is a potential candidate for the study of non-bonding molecular-orbital properties (Murata *et al.*, 1974), other examples being 6-methyl-6*H*-benzo[*cd*]pyrene, 6-methoxycarbonyl-6*H*-benzo[*cd*]pyrene and 6-mesyloxyl-6*H*-benzo[*cd*]pyrene (Hara & Yamamoto, 1980), which have been synthesized as parent hydrocarbons of the benzo[*cd*]pyrenyl system.

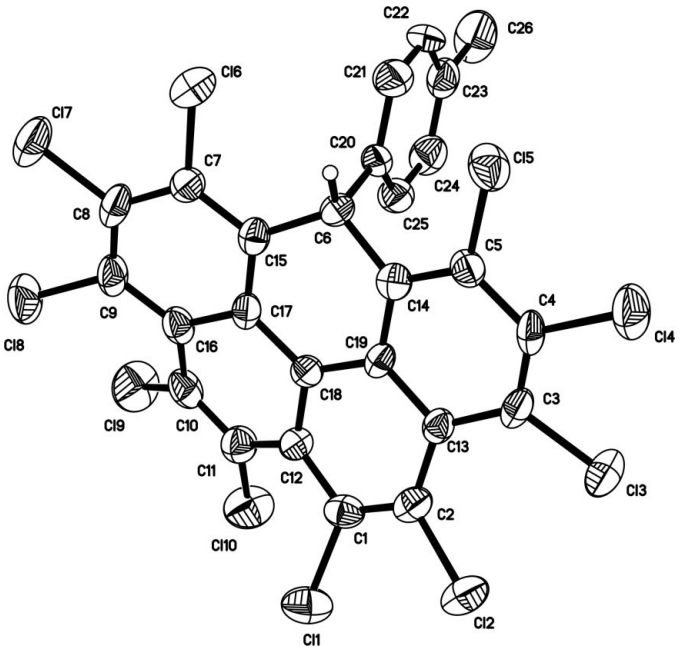


Figure 1
The molecular structure of (I) showing 50% probability displacement ellipsoids.

As shown in Fig. 1, four fused benzene rings are twisted due to the steric repulsion of overcrowded Cl atoms. The tolyl group is almost perpendicular to the skeleton plane of the compound. This perpendicular tolyl helps to pack the molecules in the crystals.

Experimental

An appropriate amount of sodium was placed in a stainless-steel autoclave and the autoclave was filled with carbon tetrachloride, up to 75% of the total volume. The autoclave was maintained at 573 K for over 40 h and then allowed to cool to room temperature. A dark solid product was collected and extracted with toluene. The extract was washed with 1 *M* HCl and then water. The solution was separated with neutral Al_2O_3 column chromatography, using cyclohexane as eluant. The first yellow fraction was collected and analyzed by mass spectrometry. The molecular peak appears at a mass/charge ratio of 670. It can be concluded that the molecule contains ten Cl atoms from the chlorine isotope distribution pattern of the molecular peak. Crystals of (I) were obtained from the fraction solution. The melting point is 541 K. The other characterizations of (I): mass spectrum for molecular ion peak, *m*/*z*: 670; ^{1}H NMR spectral data (500 MHz, CD_3Cl): 7.021, 7.005 (*d*, aromatic protons, 2H); 6.931, 6.915 (*d*, aromatic protons, 2H); 6.553 (*s*, methenyl, 1H); 2.202 (*s*, methyl, 3H).

Crystal data

$C_{26}H_8Cl_{10}$
M_r = 674.82
Tetragonal, $I4_1/a$
a = 27.0286 (4) Å
c = 13.6117 (5) Å
V = 9944.0 (4) Å^3
Z = 16
D_x = 1.803 Mg m^{-3}
Mo Kα radiation
Cell parameters from 25 reflections
θ = 7.5–15°
μ = 1.14 mm^{-1}
T = 293 (2) K
Prism, yellow
0.42 × 0.32 × 0.30 mm

Data collection

SMART CCD area-detector diffractometer
ω–2θ scans
Absorption correction: multi-scan (*SADABS*; Blessing, 1995)
T_{min} = 0.489, T_{max} = 0.710
11 159 measured reflections
4384 independent reflections
2147 reflections with $I > 2\sigma(I)$
R_{int} = 0.101
θ_{max} = 25.0°
$h = -18 \rightarrow 32$
$k = -13 \rightarrow 32$
$l = -12 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.125
S = 1.05
4384 reflections
325 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0278P)^2 + 1.2293P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C1	1.725 (6)	C2—C13	1.441 (7)
Cl2—C2	1.727 (6)	C3—C13	1.420 (8)
Cl3—C3	1.736 (6)	C6—C15	1.498 (7)
Cl4—C4	1.718 (6)	C6—C14	1.513 (7)
Cl5—C5	1.725 (6)	C6—C20	1.530 (7)
Cl6—C7	1.742 (6)	C11—C12	1.428 (8)
Cl7—C8	1.720 (6)	C12—C18	1.410 (7)
Cl8—C9	1.722 (6)	C13—C19	1.426 (7)
Cl9—C10	1.730 (6)	C17—C18	1.432 (7)
Cl10—C11	1.732 (6)	C18—C19	1.456 (7)
C1—C12	1.441 (8)		
C2—C1—C12	122.0 (5)	C14—C6—C20	109.5 (4)
C2—C1—Cl1	119.2 (5)	C18—C12—C11	117.3 (5)
C12—C1—Cl1	118.0 (5)	C18—C12—C1	117.6 (6)
C1—C2—C13	121.4 (5)	C11—C12—C1	125.1 (6)
C1—C2—Cl2	117.4 (5)	C5—C14—C19	118.9 (5)
C13—C2—Cl2	120.7 (5)	C5—C14—C6	120.1 (5)
C15—C6—C14	113.6 (5)	C19—C14—C6	120.7 (5)
C15—C6—C20	109.0 (4)		

Data collection: *SMART*1000 (Bruker, 1998); cell refinement: *SMART*1000; data reduction: *SMART*1000; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SMART*1000; software used to prepare material for publication: *SMART*1000.

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