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2,2′,3,4,4′,5,5′-Heptachlorobiphenyl (PCB 180)

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Key indicators

Single-crystal X-ray study
T = 90 K
Mean σ(C–C) = 0.004 Å
R factor = 0.033
wR factor = 0.087
Data-to-parameter ratio = 18.8

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

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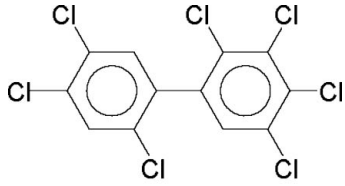
In the title compound, $C_{12}H_3Cl_7$, there are two molecules in the asymmetric unit. The dihedral angles between the benzene rings are 69.63 (7) and 68.48 (6)°.

Comment

As part of our ongoing research on the toxicity of polychlorinated biphenyls (PCBs) (Robertson & Hansen, 2001), we have synthesized 2,2′,3,4′,5,5′-heptachlorobiphenyl, (I), using the Cadogan coupling reaction. Crystallographic analysis of (I) reveals that there are two molecules in the asymmetric unit (Fig. 1). The dihedral angles, important determinants of the toxicity of PCBs, are 69.63 (7) and 68.48 (6)°. These dihedral angles are smaller than the calculated dihedral angle of 73.0° in aqueous solution.



(I)

According to our review of the literature, the experimental dihedral angles for mono-, di-, tri- and tetra-*ortho*-Cl-substi-

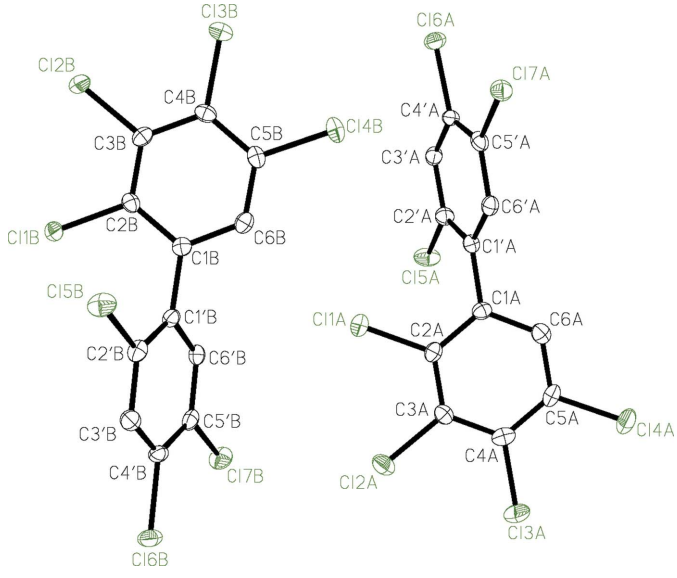


Figure 1
A view of the asymmetric unit of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity.

tuted PCB derivatives are 47–51° (Kania-Korwel *et al.*, 2004; Lehmler *et al.*, 2001; McKinney & Singh, 1988; van der Sluis *et al.*, 1990), 59–75° (Miao *et al.*, 1997; Rissanen *et al.*, 1988*a*; Rømming *et al.*, 1974; Singh *et al.*, 1986), 82–83° (Lehmler *et al.*, 2005; Rissanen *et al.*, 1988*b*) and 84–87° (Pedersen, 1975; Shaikh *et al.*, 2006; Singh & McKinney, 1979), respectively. As with the title compound, (I), the calculated dihedral angles are typically larger than the solid-state dihedral angles (*viz.* 57.7, 73.0, 89.8 and 89.9° for mono-, di-, tri- and tetra-*ortho*-Cl-substituted PCB derivatives, respectively). The difference between solid-state and calculated solution dihedral angles is probably due to crystal packing effects.

Experimental

The title compound, (I), was synthesized in 6.5% yield by the Cadogan coupling of 2,4,5-trichloroaniline with an excess of 1,2,3,4-tetrachlorobenzene (Espandiari *et al.*, 2003). The product was purified twice by column chromatography on silica gel using *n*-hexanes as eluent. Colorless plates were obtained upon crystallization from methanol.

Crystal data

$C_{12}H_3Cl_7$
$M_r = 395.29$
Triclinic, $P\overline{1}$
$a = 8.0695$ (1) Å
$b = 13.4132$ (2) Å
$c = 13.9965$ (2) Å
$\alpha = 111.1137$ (6)°
$\beta = 90.0322$ (6)°
$\gamma = 93.6854$ (7)°
$V = 1409.87$ (3) Å^3
$Z = 4$
$D_x = 1.862$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 1.39$ mm^{-1}
$T = 90.0$ (2) K
Irregular block cut from plate, colorless
0.25 × 0.20 × 0.15 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
$T_{min} = 0.723$, $T_{max} = 0.819$
12289 measured reflections
6451 independent reflections
5238 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 27.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.087$
$S = 1.05$
6451 reflections
343 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0417P)^2 + 0.9995P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.88$ e Å^{-3}
$\Delta\rho_{min} = -0.40$ e Å^{-3}

H atoms were placed in idealized positions and were refined using a riding model, with C—H = 0.95 Å and $U_{iso} = 1.2U_{eq}$(C).

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 1994); software used to prepare material for publication: *SHELX97*-2 (Sheldrick, 1997) and local procedures.

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