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2,2′,5,5′-Tetrachlorobiphenyl (PCB 52)

Xiu-Sheng Miao,[a] Shao-Gang Chu,[a] Xiao-Bai Xu[a]* and Xiang-Lin Jin[b]

[a]*Research Center for Eco-Environmental Sciences, Chinese Academy of Sciences, Beijing 100085, People's Republic of China, and* [b]*Institute of Physical Chemistry, Beijing University, Beijing 100871, People's Republic of China*

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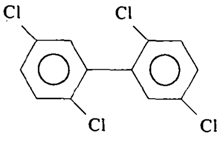
Abstract

The title compound, $C_{12}H_6Cl_4$, 2,2′,5,5′-tetrachlorobiphenyl, crystallized in the monoclinic space group *C*2/*c*. The dihedral angle and bond length between the two phenyl rings are 59.0° and 1.467 (14) Å, respectively. The *ortho*- and *meta*-C—Cl bond lengths, C(6)—Cl(2) and C(3)—Cl(1), are 1.746 (8) and 1.746 (9) Å, respectively.

Comment

Polychlorinated biphenyls (PCBs) are recognized as significant environmental contaminants. A total of 209 different congeners are theoretically possible; these vary significantly in their toxicity, biological activity and degradation as a result of the different number and/or substitution position of the Cl atoms. Previous work (Safe, 1992) has suggested that the congeners having a coplanar structure show higher toxicity. The dihedral angle between the two phenyl rings may thus play a dominant role in determining biological activity. Our aim is to study the structure–activity relationships of a series of environmentally important PCB congeners and as part of this work, the structure of 2,2′,5,5′-tetrachlorobiphenyl, (I), was determined.



(I)

The molecule of the title compound has a twofold rotation axis normal to the biphenyl bridge and bisecting the dihedral angle between the two phenyl rings (Fig. 1). The C—C bond distances in the two phenyl rings are normal, with values in the range 1.364 (11) to 1.416 (10) Å and a mean of 1.388 (11) Å. The carbon skeleton of each phenyl ring is planar, but the Cl(1) and Cl(2) atoms deviate from planarity by 0.0152 and 0.0616 Å, respectively. Atoms C1, C2, C6 and C1′ are coplanar within 0.0024 Å and the mean deviation of these atoms from the plane is 0.0012 Å. The *ortho*- and *meta*-C—Cl bond lengths, C(6)—Cl(2) and C(3)—Cl(1), are 1.746 (8) and 1.746 (9) Å, respectively. The inter-ring C(1)—C(1′) bond length of the title compound is 1.467 (14) Å. The distances of the Cl(1) atom from atoms H(2) and H(4) are 2.789 and 2.808 Å, and the distances of the Cl(2) atom from atoms H(2′) and H(5) are 2.967 and 2.743 Å, respectively. The dihedral angle between the two phenyl rings in the title compound is 59.0° while those in 2,4′-dichlorobiphenyl (van der Sluis, Moes, Behm, Smykalla, Beurskens & Lenstra, 1990), 2,2′,4,4′,5,5′-hexachlorobiphenyl (Singh, Pedersen & McKinney, 1986) and 2,2′,4,4′,6,6′-hexachlorobiphenyl (Singh & McKinney, 1979) are 49.6, 58.3, and 87.3°, respectively. It may be suggested that the dihedral angle should be affected principally by the *ortho* chlorine substituents.

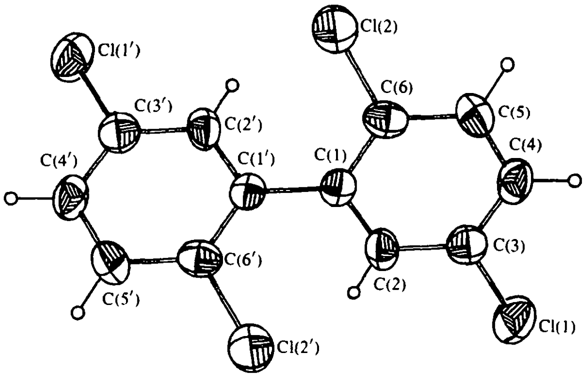


Fig. 1. The molecular structure and atom-numbering scheme of the title compound. Displacement ellipsoids are plotted at the 50% probability level.

The molecules are packed in layers parallel to the *bc* plane at $x = 0$ and $x = \frac{1}{2}$. There are van der Waals interactions between molecules and weak Cl-atom interactions between layers. The distance between atoms Cl(1) and Cl(2)($\frac{1}{2} + x$, $\frac{1}{2} - y$, $z - \frac{1}{2}$) is 3.416 Å, which is shorter than the sum of their van der Waals radii (3.6 Å).

It has been suggested that the dihedral angle between the pair of phenyl rings in PCB congeners plays a dominant rôle in determining their toxicities and biological activities. The dihedral angle in the title compound is 59.0°, which is in contrast with the coplanarity of high toxicity PCB congeners. This is consistent with the fact that it has neither been classified as a highly toxic congener nor given the TEFs (Toxic Equivalency Factors) (Safe, 1992).

Experimental

The synthesis and purification of (I) were carried out according to Mullin, Pochini, McCrindle, Romkes, Safe & Safe (1984). The crude product was separated by adsorption chromatography on a silica gel column and eluted with petroleum ether. The elute was concentrated and further purified by repeated thin layer chromatography. Single crystals were obtained from ethanol solution and data were collected on the best crystal available.

Crystal data

$C_{12}H_6Cl_4$
$M_r = 292.0$
Monoclinic
$C2/c$
$a = 12.909\,(3)$ Å
$b = 8.174\,(2)$ Å
$c = 11.302\,(2)$ Å
$\beta = 94.82\,(3)°$
$V = 1188.4\,(5)$ Å^3
$Z = 4$
$D_x = 1.632$ Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 7.5–10°$
$\mu = 0.967$ mm^{-1}
$T = 296$ K
Thin plate
0.50 × 0.30 × 0.07 mm
Colourless

Data collection

Rigaku AFC-6*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scans (*SHELXTL-Plus*; Sheldrick, 1990)
$T_{\min} = 0.5731$, $T_{\max} = 1.000$
884 measured reflections
773 independent reflections
550 observed reflections $[F > 4\sigma(F)]$
$R_{\rm int} = 0.0880$
$\theta_{\max} = 22.5°$
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 8$
$l = -12 \rightarrow 12$
3 standard reflections monitored every 150 reflections intensity decay: 0.3%

Refinement

Refinement on F
$R = 0.077$
$wR = 0.094$
$S = 0.97$
550 reflections
73 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F) + 0.0135F^2]$
$(\Delta/\sigma)_{\max} = 0.003$
$\Delta\rho_{\max} = 0.70$ e Å^{-3}
$\Delta\rho_{\min} = -0.38$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Cl(1)	0.6341 (2)	0.1181 (3)	0.4071 (2)	0.063 (1)
Cl(2)	0.3101 (2)	0.4524 (4)	0.7035 (2)	0.063 (1)
C(1)	0.4905 (5)	0.2829 (10)	0.6851 (6)	0.036 (2)
C(2)	0.5611 (6)	0.2119 (10)	0.6140 (6)	0.040 (2)
C(3)	0.5430 (6)	0.2096 (11)	0.4918 (8)	0.047 (3)
C(4)	0.4540 (6)	0.2756 (12)	0.4331 (7)	0.055 (3)
C(5)	0.3829 (6)	0.3475 (11)	0.5028 (7)	0.049 (3)
C(6)	0.4017 (5)	0.3541 (10)	0.6233 (7)	0.041 (3)

Table 2. *Selected geometric parameters (Å, °)*

Cl(1)—C(3)	1.746 (9)	Cl(2)—C(6)	1.746 (8)
C(1)—C(2)	1.392 (10)	C(1)—C(6)	1.416 (10)
C(1)—C(1′)	1.467 (14)	C(2)—C(3)	1.382 (11)
C(3)—C(4)	1.386 (11)	C(4)—C(5)	1.389 (12)
C(5)—C(6)	1.364 (11)		
C(2)—C(1)—C(6)	115.4 (6)	C(2)—C(1)—C(1′)	121.0 (7)
C(6)—C(1)—C(1′)	123.6 (7)	C(1)—C(2)—C(3)	121.2 (7)
Cl(1)—C(3)—C(2)	119.2 (6)	Cl(1)—C(3)—C(4)	118.3 (7)
C(2)—C(3)—C(4)	122.5 (8)	C(3)—C(4)—C(5)	117.0 (7)
C(4)—C(5)—C(6)	120.8 (7)	Cl(2)—C(6)—C(1)	119.2 (6)
Cl(2)—C(6)—C(5)	117.7 (6)	C(1)—C(6)—C(5)	123.0 (7)

Symmetry code: (′) $1 - x, y, \frac{3}{2} - z$.

Crystals of good quality were difficult to obtain and this was the main reason for the large *R* values. H atoms were not refined. They were located at calculated positions and assigned isotropic displacement parameters. Data collection and refinement used *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). The structure solution and refinement used *SHELXTL-Plus* (Sheldrick, 1990), which was also used for data reduction, molecular graphics and the preparation of material for publication.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KH1067). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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α,α,α,2,3,4,5,6-Octafluoroacetanilide

ABDELAZIZ MAHOUI, JACQUES LAPASSET* AND JACQUES MORET

GDPC, cc 026, Université Montpellier II, Place E. Bataillon, 34095 Montpellier CEDEX 5, France. E-mail: lapas@gdpc.univ-montp2.fr

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Abstract

Acetanilide and its derivatives have been the subject of some interest because of certain anomalous properties

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