

2,4,5-Trichlorobiphenyl

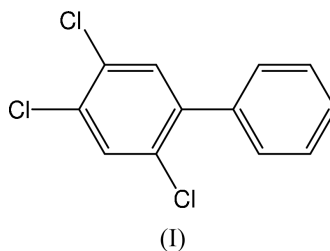
Izabela Kania-Korwel,^a Sean Parkin,^b Larry W. Robertson^{c*} and Hans-Joachim Lehmler^c^aDepartment of Environmental Chemistry and Technology, University of Silesia, Szkolna 9, 40-006 Katowice, Poland, ^bDepartment of Chemistry, University of Kentucky, Lexington, KY 40506-0055, USA, and ^cDepartment of Occupational and Environmental Health, College of Public Health, 100 Oakdale Campus #124 IREH, Iowa City, Iowa 52242-5000, USACorrespondence e-mail:
larry-robertson@uiowa.edu

Key indicators

Single-crystal X-ray study
 $T = 90$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.026
 wR factor = 0.063
Data-to-parameter ratio = 17.5For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.The dihedral angle between the benzene rings in the title compound, $\text{C}_{12}\text{H}_7\text{Cl}_3$, is $47.25(4)^\circ$.Received 20 August 2004
Accepted 25 August 2004
Online 31 August 2004

Comment

Polychlorinated biphenyls (PCBs) are a group of ubiquitous environmental pollutants (Robertson & Hansen, 2001). They are only slowly biodegraded, and they bioaccumulate and biomagnify. According to their substitution patterns, PCBs elicit a wide range of toxic responses in living organisms. Knowledge of three-dimensional structure, especially the dihedral angle which describes the deformation of the molecule from a planar conformation, helps to define the potential binding sites of PCB congeners and hence the hazards associated with their occurrence.



The title molecule, (I), has a solid-state dihedral angle of $47.25(4)^\circ$. In contrast, the solid-state torsion angle between the two benzene rings of other mono-*ortho*-chloro-substituted biphenyls is in the range 50 – 52° (Miao *et al.*, 1996; Lehmler *et al.*, 2001). In solution, the calculated dihedral angle for (I) [calculated with *MM2* using GB/SA water solvent continuum as implemented by *MACROMODEL* 5.0 (Still *et al.*, 1990)] is larger (57.7°) and is probably due to the alleviation of packing effects present in the solid state (Lehmler *et al.*, 2002). This raises the question whether the solid state or the calculated solution dihedral angle represents biologically relevant conformations of (I).

A partial answer to this question can be found in the crystallographic analysis of the binding of 4,4-hydroxy-3,3',5,5'-tetrachlorobiphenyl to the estrogen receptor (Shevtsov *et al.*, 2003). The dihedral angle of the dihydroxy PCB bound to protein was 30° , while the calculated solution and the solid-state dihedral angle of the neat dihydroxy PCB were 42 and 0° , respectively (McKinney, 1988). This implies that the biologically relevant conformations of PCBs such as the title compound can be described as the range between the calculated solution and the solid-state dihedral angles, with the understanding that these torsion angles may be greatly influenced by the protein-binding sites in which they may reside.

Experimental

The title compound, (I), was synthesized in 52% yield by the Suzuki coupling of 2,4,5-trichloriodobenzene with phenylboronic acid (Kania-Korwel *et al.*, 2004). White needles were obtained upon crystallization from methanol.

Crystal data

$C_{12}H_7Cl_3$
 $M_r = 257.53$
 Triclinic, $P\bar{1}$
 $a = 3.85010$ (10) Å
 $b = 11.4482$ (3) Å
 $c = 13.0504$ (3) Å
 $\alpha = 109.8409$ (10)°
 $\beta = 92.9891$ (9)°
 $\gamma = 99.2004$ (10)°
 $V = 530.59$ (2) Å³

$Z = 2$
 $D_x = 1.612$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 2350 reflections
 $\theta = 1.0$ – 27.5 °
 $\mu = 0.82$ mm⁻¹
 $T = 90.0$ (2) K
 Block, colourless
 $0.24 \times 0.15 \times 0.10$ mm

Data collection

Nonius KappaCCD diffractometer
 ω scans
 Absorption correction: multi-scan
 (SCALEPACK; Otwinowski & Minor, 1997)
 $T_{\min} = 0.827$, $T_{\max} = 0.923$
 4668 measured reflections

2383 independent reflections
 2065 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.018$
 $\theta_{\text{max}} = 27.4$ °
 $h = -4 \rightarrow 4$
 $k = -14 \rightarrow 14$
 $l = -16 \rightarrow 16$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.026$
 $wR(F^2) = 0.063$
 $S = 1.07$
 2383 reflections
 136 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0219P)^2 + 0.2643P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.36$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.23$ e Å⁻³

Recollection of the first part of the first scan of area-detector data for this set showed no significant differences in integrated intensities. H atoms were placed in geometrically idealized positions and constrained using a riding model in which the C–H distance was fixed at 0.95 Å with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

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 Siemens SHELXTL/PC (Sheldrick, 1994); software used to prepare material for publication: SHELX97-2 (Sheldrick, 1997) and local programs.

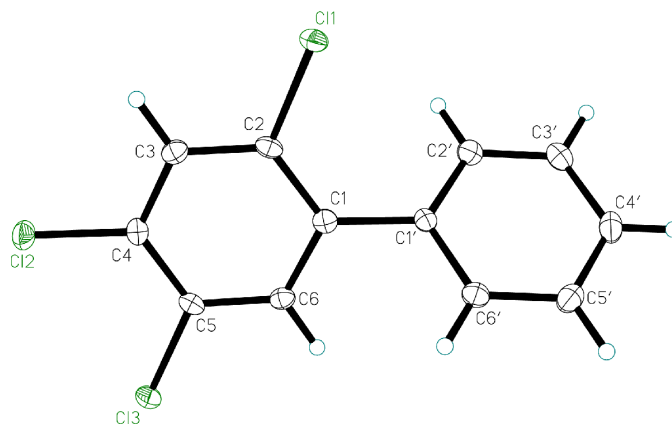


Figure 1

View of the title compound showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

This publication was made possible by a Fulbright Junior Research Grant and a Kosciuszko Foundation Grant to IKK. The project was supported by grant number P42 ES 07380 from the National Institute of Environmental Health Sciences, NIH.

References

- Kania-Korwel, I., Parkin, S., Robertson, L. W. & Lehmler, H.-J. (2004). *Chemosphere*, **56**, 735–744.
- Lehmler, H.-J., Parkin, S. & Robertson, L. W. (2001). *Acta Cryst.* **E57**, o111–o112.
- Lehmler, H.-J., Robertson, L. W., Parkin, S. & Brock, C. P. (2002). *Acta Cryst.* **B58**, 140–147.
- McKinney, J. D. (1988). *Acta Cryst.* **C44**, 558–562.
- Miao, X., Chu, S., Xu, X. & Jin, X. (1996). *Chin. Sci. Bull.* **42**, 1803–1806.
- Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
- Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr and R. M. Sweet, pp. 307–326. New York: Academic Press.
- Robertson, L. W. & Hansen, L. G. (2001). *Recent Advances in the Environmental Toxicology and Health Effects of PCBs*. Lexington: University Press of Kentucky.
- Sheldrick, G. M. (1994). *SHELXTL/PC*. Version 5. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (1997). *SHELXL97*, *SHELXS97* and *SHELX97-2*. University of Göttingen, Germany.
- Shevtsov, S., Petrotchenko, E. V., Pedersen, L. C. & Negishi, M. (2003). *Environ. Health Perspect.* **111**, 884–888.
- Still, W. C., Tempczyk, A., Hawley, R. C. & Hendrickson, T. (1990). *J. Am. Chem. Soc.* **112**, 6127–6129.