

2,3,4'-Trichlorobiphenyl

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Key indicators

Single-crystal X-ray study

$T = 173\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$

R factor = 0.026

wR factor = 0.054

Data-to-parameter ratio = 16.8

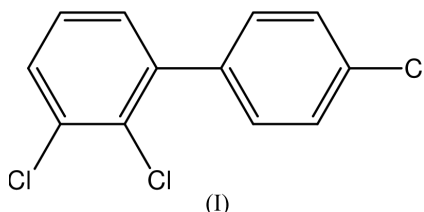
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title compound, $\text{C}_{12}\text{H}_7\text{Cl}_3$, the dihedral angle of the biphenyl moiety is $51.21(5)^\circ$.

Comment

Polychlorinated biphenyls (PCBs) are persistent and widespread environmental contaminants (Hansen, 1999). Their lipophilic character and resistance to degradation contribute to the tendency of PCBs to accumulate in the food chain, where they present an environmental and human health hazard (Hansen, 1999). The three-dimensional structure of a PCB congener is strongly correlated with its toxicity. For example, derivatives without *ortho* chlorine substituents, also referred to as 'co-planar PCBs', are known to bind to the Ah receptor, and their mechanism of toxicity is well investigated. Recently other targets with differing structural requirements have been identified as affecting several organ systems (Robertson & Hansen, 2001).

Out of 209 possible PCB congeners, only the crystal structures of seven PCB congeners have been described. The bond distances and bond angles in the title compound, (I), are similar to those found in other chlorinated biphenyls [summarized by Miao *et al.* (1997)]. The torsion angle between the two phenyl rings of PCBs appears to depend on the degree of chlorination at the *ortho* position. According to published data, mono-*ortho*, di-*ortho* and tetra-*ortho* substituted PCBs show dihedral angles of $49\text{--}52$, $58\text{--}67$ and $86\text{--}87^\circ$, respectively [summarized by Miao *et al.* (1997) and Singh *et al.* (1986)]. The dihedral angle of (I), $51.21(5)^\circ$, is the same as those published for PCB congeners with one *ortho* substituent. The theoretical torsion angles of mono-*ortho* PCBs calculated by Höfler *et al.* (1988) are larger ($60\text{--}72^\circ$) than those reported for the solid state. Thus, packing effects appear to influence the torsion angle between the two phenyl rings of PCBs in the solid state.



Experimental

The title compound, (I), was synthesized in 70% yield by the palladium-catalyzed cross coupling of 4-chlorobenzene boronic acid and 2,3-dichlorobromobenzene (Lehmler & Robertson, 2001; Bauer *et al.*, 1995). Colourless needles were obtained upon crystallization from methanol; m.p. $341\text{--}342\text{ K}$.

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Crystal data

$C_{12}H_7Cl_3$
 $M_r = 257.53$
 Monoclinic, Cc
 $a = 3.9010 (10) \text{ \AA}$
 $b = 17.398 (2) \text{ \AA}$
 $c = 15.749 (2) \text{ \AA}$
 $\beta = 94.510 (10)^\circ$
 $V = 1065.6 (3) \text{ \AA}^3$
 $Z = 4$
 $D_x = 1.605 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation
 Cell parameters from 7599 reflections
 $\theta = 1.0\text{--}27.5^\circ$
 $\mu = 0.82 \text{ mm}^{-1}$
 $T = 173 (1) \text{ K}$
 Irregular flattened needle, colourless
 $0.24 \times 0.20 \times 0.07 \text{ mm}$

Data collection

Nonius KappaCCD diffractometer
 ω scans at fixed $\chi = 55^\circ$
 4022 measured reflections
 2305 independent reflections
 2197 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.023$
 $\theta_{\text{max}} = 27.5^\circ$
 $h = -4 \rightarrow 5$
 $k = -22 \rightarrow 22$
 $l = -20 \rightarrow 20$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.026$
 $wR(F^2) = 0.054$
 $S = 1.07$
 2305 reflections
 137 parameters
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0115P)^2 + 0.3392P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.20 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.17 \text{ e \AA}^{-3}$
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.0064 (7)
 Absolute structure: (Flack, 1983),
 1076 Friedel pairs
 Flack parameter = $-0.02 (5)$

Table 1

Selected geometric parameters (\AA , $^\circ$).

| | | | |
|--------------|-------------|-------------|-------------|
| C1—C7 | 1.494 (3) | C8—C12 | 1.7401 (18) |
| C4—C1 | 1.7408 (19) | C9—C13 | 1.7323 (19) |
| C6—C1—C7—C12 | 48.0 (3) | C2—C1—C7—C8 | 54.2 (3) |

Assignment of space group Cc based on systematic absences and intensity statistics. The possibility of the centrosymmetric space group $C2/c$ was discounted by lack of a suitable solution and by the wholly satisfactory refinement in Cc . There were no substantial correlation coefficients in the variance-covariance matrix between parameters sensitive to a missing inversion centre. No formal absorption correction was applied. Face-indexed correction was not feasible because of the crystal morphology. Correction based on multiple measurements of equivalents was attempted but was deemed unsatisfactory. Anisotropic absorption is corrected in a reasonably approximate manner by the inter-frame scaling and merging of equivalents within the data-reduction program *SCALEPACK* (Otwinowski & Minor, 1997). Subsequent application of an empirical absorption correction based on ΔF^2 made no substantive difference to either data or model and so the uncorrected data were employed in refinement.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction:

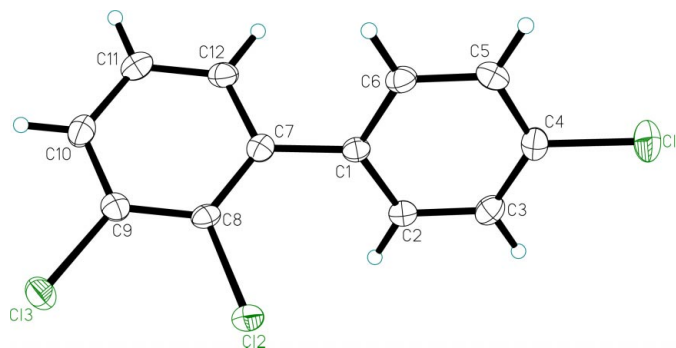


Figure 1

A view of (I) with non-H atom displacement ellipsoids drawn at the 50% probability level.

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

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