2,5-Dichloro-4′-methoxybiphenyl

The dihedral angle between the benzene rings in the title compound, C₁₃H₁₀Cl₂O₁, is 59.92 (9)°.

Comment

Polychlorinated biphenyls (PCBs) are an important class of persistent environmental contaminants (Robertson & Hansen, 2001). They are metabolized in vivo to hydroxylated and other metabolites. The three dimensional structure of PCBs and their metabolites is determined by the dihedral angle between the two benzene rings. The dihedral angle is thought to be an important determinant of the binding affinity of hydroxylated PCBs to various proteins (Lehmler et al., 2002). We synthesized 2,5-dichloro-4′-methoxybiphenyl, a methylated analog of a hydroxylated PCB, as part of our ongoing research into the phase II metabolism of hydroxylated PCBs (Tampal et al., 2002; van den Hurk et al., 2002; Wang et al., 2005; Wang et al., 2006). The crystal structure of this PCB derivative showed a dihedral angles of 59.92 (9)°, which is slightly larger than the calculated dihedral angle of 57.7° in aqueous solution [calculated with MM2 using GB/SA water solvent continuum as implemented by MACROMODEL 5.0 (Still et al., 1990)].

According to our review of the literature, the experimental dihedral angles for mono-, di-, tri- and tetra-ortho CI-substituted PCB derivatives are 47–51° (Kania-Korwel et al., 2004; Lehmler et al., 2001; McKinney & Singh, 1988; Sluis et al., 1990), 59–75° (Vyas et al., 2006; Miao et al., 1997; Rissanen et al., 1988a; Romming et al., 1974; Singh et al., 1986), 82–83° (Lehmler et al., 2005; Rissanen et al., 1988b) and 84–87° (Pedersen, 1975; Shaikh et al., 2006; Singh & McKinney, 1979), respectively. As a result of crystal packing effects, the calculated dihedral angles of these PCB derivatives (viz., 57.7°, 73.0°, 89.8° and 89.9° for mono-, di-, tri- and tetra-ortho CI-substituted PCB derivatives, respectively), in contrast to the title compound, are larger than the solid state dihedral angles. Overall, the title compound and other PCB derivatives may have some conformational flexibility when interacting with proteins, a fact that may be helpful in determining three-dimensional quantitative structure–activity relationships for a variety of phase II enzymes.
Experimental

The title compound, (I), was synthesized in 75% yield by the Suzuki coupling of 4-methoxyphenylboronic acid and 2,5-dichlorobromo-benzene (Kania-Korwel et al., 2004). Colorless blocks were obtained upon crystallization from methanol.

Crystal data

\[ \text{C}_{13}\text{H}_{10}\text{Cl}_{2}\text{O} \]

\[ M_r = 253.11 \]

Monoclinic, \( P2_1/n \)

\[ a = 9.4758 (3) \, \text{Å} \]

\[ b = 14.2682 (5) \, \text{Å} \]

\[ c = 9.5562 (3) \, \text{Å} \]

\[ \beta = 116.8711 (15) \]

\[ V = 1152.52 (7) \, \text{Å}^3 \]

\[ Z = 4 \]

\[ D_r = 1.459 \, \text{Mg m}^{-3} \]

Mo Ka radiation

\[ \mu = 0.54 \, \text{mm}^{-1} \]

\[ T = 90.0 (2) \, \text{K} \]

Block, colourless

\[ 0.18 \times 0.16 \times 0.12 \, \text{mm} \]

Data collection

Nonius KappaCCD diffractometer

\( \omega \) scans

Absorption correction: multi-scan

 SCALEPACK (Otwinowski & Minor, 1997)

\[ T_{\text{max}} = 0.91, \quad T_{\text{min}} = 0.94 \]

Refinement

Refinement on \( F^2 \)

\[ R[F^2 > 2\sigma(F^2)] = 0.049 \]

\[ wR(F^2) = 0.131 \]

\[ S = 1.02 \]

5102 measured reflections

1651 reflections with \( I > 2\sigma(I) \)

\( R_{\text{ref}} = 0.052 \]

\[ \theta_{\text{max}} = 27.5^\circ \]

H atoms were positioned geometrically (C–H = 0.95–0.98 Å) and refined as riding, with \( U_{\text{iso}}(\text{H}) = 1.2 \) or 1.5 times \( U_{\text{eq}}(\text{C}) \).

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, 1994); 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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References


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