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4-Chloro-2′,3′-dimethoxybiphenyl

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Key indicators

Single-crystal X-ray study
T = 90 K
Mean σ(C–C) = 0.002 Å
R factor = 0.042
wR factor = 0.118
Data-to-parameter ratio = 18.2

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

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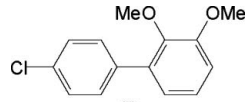
Molecules of the title compound, $C_{14}H_{13}Cl_1O_2$, crystallize as centrosymmetric dimers, connected by intermolecular C—H···O hydrogen bonds. The dihedral angle between the benzene rings is 40.05 (6)°.

Comment

As part of our ongoing research on the toxicity of polychlorinated biphenyls (PCBs), we have synthesized 4-chloro-2′,3′-dimethoxybiphenyl, (I), using the Suzuki coupling reaction. The dihydroxylated analog of this compound is a known metabolite of 4-chlorobiphenyl (McLean *et al.*, 1996) and its toxicity has been studied *in vitro* (Srinivasan *et al.*, 2001) and *in vivo* (Espandiari *et al.*, 2004).



(I)

There is some evidence that the three-dimensional structure of dihydroxylated PCB metabolites may be correlated with their reactivity towards DNA (Arif *et al.*, 2003). To explore the role of the three-dimensional structure of dihydroxylated PCB metabolites in their toxicity, we have determined the solid state structure of the related title compound, (I).

In the solid state, the dihedral angle between the benzene rings of (I) is 40.05 (6)° and, thus, is smaller than the calculated dihedral angle of 48° in aqueous solution [calculated with *MM2* using GB/SA water solvent continuum as implemented by *MACROMODEL5.0* (Still *et al.*, 1990)] as a result of crystal packing effects. Similarly, the experimental dihedral angles for *ortho* Cl-substituted PCB congeners and PCB derivatives are typically smaller than calculated values (Vyas *et al.*, 2006 and references therein). In the crystalline state, the molecules exist as centrosymmetric dimers, connected by intermolecular C—H···O hydrogen bonds (Table 1).

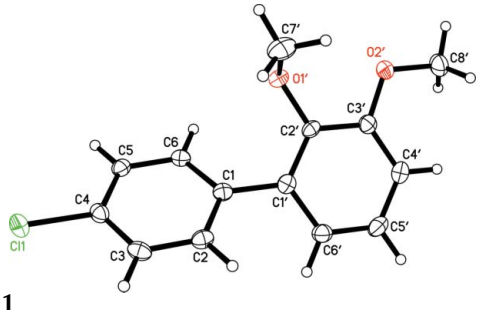


Figure 1
The molecular structure of the title compound, showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.

Experimental

Compound (I) was synthesized in 80% yield by the Suzuki coupling of 2,3-dimethoxyphenylboronic acid and *p*-bromochlorobenzene (Kania-Korwel *et al.*, 2004; Lehmler & Robertson, 2001). Colorless crystals were obtained upon crystallization from methanol.

Crystal data

$C_{14}H_{13}ClO_2$
$M_r = 248.69$
Monoclinic, $P2_1/c$
$a = 10.6579$ (2) Å
$b = 13.8592$ (3) Å
$c = 8.4098$ (3) Å
$\beta = 91.2829$ (11)°
$V = 1241.90$ (6) Å^3
$Z = 4$
$D_x = 1.330$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.29$ mm^{-1}
$T = 90.0$ (2) K
Block, colorless
0.22 × 0.20 × 0.12 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
$T_{min} = 0.938$, $T_{max} = 0.966$
5547 measured reflections
2841 independent reflections
2097 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 27.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.118$
$S = 1.06$
2841 reflections
156 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0658P)^2 + 0.1233P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.49$ e Å^{-3}
$\Delta\rho_{min} = -0.39$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···O2′^{i}	0.95	2.57	3.520 (2)	179
C6—H6···O1′	0.95	2.45	2.906 (2)	110
C6—H6···O1′^{i}	0.95	2.53	3.185 (2)	126

Symmetry code: (i) $-x+1, -y+1, -z+1$.

H atoms were found in difference Fourier maps and subsequently placed in idealized positions with constrained C—H distances of 0.98 (methyl) and 0.95 Å (aromatic). U_{iso}(H) values were set at either $1.5U_{eq}$(methyl C) or $1.2U_{eq}$(aromatic 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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