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An asymmetric biphenyl compound: 3-chloro-4,4′-dimethoxybiphenyl

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Key indicators

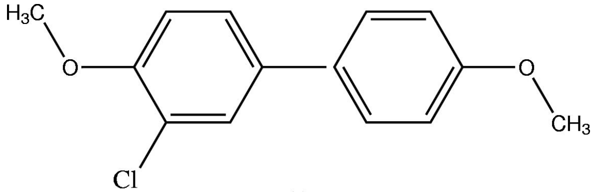
Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.006 Å
R factor = 0.063
wR factor = 0.131
Data-to-parameter ratio = 13.4

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

The asymmetric biphenyl title compound, $C_{14}H_{13}ClO_2$, exhibits a significant twist of *ca* 30° about the central C—C bond.

Comment

Enantiopure biphenyl carbinols are valuable intermediates in the preparation of new ligands (Longmire *et al.*, 1997; Imai *et al.*, 2000; Mikami *et al.*, 2002) and are useful models for understanding the biosynthesis and stereochemistry of naturally occurring compounds which possess the biphenyl fragment (Uchida *et al.*, 1985; Tene *et al.*, 2000). Recently, significant progress has been made in the study of conformationally flexible hydroxylated biphenyls, including investigating their application in stoichiometric and catalytic asymmetric reactions (Bartels & Helmchen, 1999; Superchi *et al.*, 2001; Pàmies *et al.*, 2000; Mikami *et al.*, 2002). This interest reflects the importance of the biphenyl unit in stereochemical control (Mikami & Yamanaka, 2003) and the efforts devoted to the design of new chiral and configurationally flexible biphenyls. In spite of the importance of biphenyl carbinols, an efficient method to synthesize these compounds in high yields and in an optically pure form is still required. Prompted by the above, the title compound, (I) (Fig. 1), was examined.



(I)

There are no unusual bond lengths and angles in (I). There is a significant twist about the central C4—C10 bond, as manifested by the C5—C4—C10—C9 torsion angle of −150.5 (4)°. The length of this bond, 1.494 (5) Å, suggests

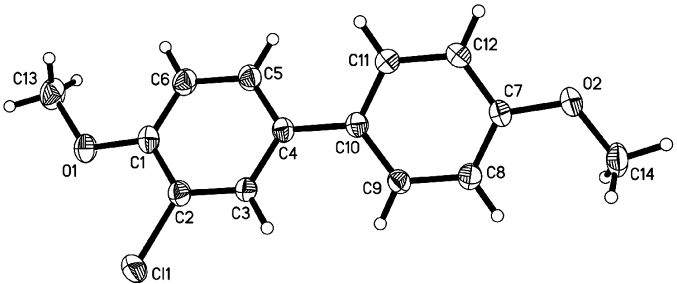


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

little delocalization of π-electron density over the two aromatic rings, consistent with the aforementioned twist. The two methoxy groups are rotated slightly out of the plane of their respective benzene rings, as seen in the C13—O1—C1—C2 and C14—O2—C7—C8 torsion angles of 173.3 (4) and 7.1 (6)°, respectively. These conformational features, we believe, are to a large extent controlled by the extensive non-covalent intermolecular interactions operating in the structure.

Inversion-related molecules form dimeric pairs through C—H···O interactions. Within the dimeric unit, the two aromatic rings have a parallel-offset arrangement.

The crystal structure is further stabilized by weak C—H···O interactions between the dimers, forming a network structure as represented in Fig. 2.

Experimental

The title compound was synthesized by the coupling reaction of an activated halohydrocarbon and phenylmagnesium bromide in refluxing anhydrous diethyl ether according to the literature method of Sekiya & Ishikawa (1976). Crystals suitable for X-ray structural analysis were grown by slow evaporation of an ethanol solution of the compound held at room temperature.

Crystal data

$C_{14}H_{13}ClO_2$
$M_r = 248.69$
Orthorhombic, *Pbca*
$a = 9.807$ (7) Å
$b = 9.193$ (6) Å
$c = 26.607$ (17) Å
$V = 2399$ (3) Å^3
$Z = 8$
$D_x = 1.377$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 2269 reflections
$\theta = 2.6$–$26.7°$
$\mu = 0.30$ mm^{-1}
$T = 292$ (2) K
Block, colourless
0.24 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.918$, $T_{\max} = 0.976$
11187 measured reflections

2105 independent reflections
1269 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.075$
$\theta_{\max} = 25.0°$
$h = -10 \rightarrow 11$
$k = -9 \rightarrow 10$
$l = -31 \rightarrow 31$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.131$
$S = 1.07$
2105 reflections
157 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0164P)^2 + 5.1258P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.23$ e Å^{-3}
$\Delta\rho_{\min} = -0.27$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0011 (3)

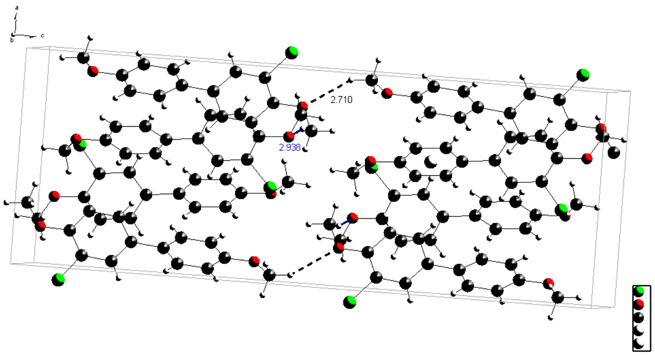


Figure 2
The packing in (I), viewed along the *a* axis.

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.96 Å, and with $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$ for methyl H atoms and $1.2U_{\rm eq}({\rm C})$ for other H atoms. Each methyl group was allowed to rotate freely about its C—C bond.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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References

Bartels, B. & Helmchen, G. (1999). *J. Chem. Soc. Chem. Commun.* pp. 741–742.
Bruker (1997). *SHELXTL.* Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1998). *SAINT* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Imai, Y., Zhang, W., Kida, T., Nakatsuji, Y. & Ikeda, I. (2000). *J. Org. Chem.* **65**, 3326–3333.
Longmire, J. M., Zhu, G. & Zhang, X. (1997). *Tetrahedron Lett.* **38**, 375–378.
Mikami, K., Aikawa, K., Yusa, Y. & Hatano, M. (2002). *Org. Lett.* **4**, 91–94.
Mikami, K. & Yamanaka, M. (2003). *Chem. Rev.* **103**, 3369–3400.
Pàmies, O., Net, G., Ruiz, A. & Claver, C. (2000). *Tetrahedron Asymmetry*, **11**, 1097–1108.
Sekiya, A. & Ishikawa, N. (1976). *J. Org. Chem.* **118**, 349–354.
Sheldrick, G. M. (1996). *SADABS.* University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Superchi, S., Casarini, D., Laurita, A., Bavoso, A. & Rosini, C. (2001). *Angew. Chem. Int. Ed.* **40**, 451–454.
Tene, M., Wabo, H. K., Kamnaing, P., Tsopmo, A., Tane, P., Ayafor, J. F. & Sterner, O. (2000). *Photochemistry*, **54**, 975–978.
Uchida, I., Shigematsu, N., Ezaki, M., Hashimoto, M., Aoki, H. & Imanaka, I. (1985). *J. Antibiot.* **38**, 1462–1468.

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