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4-Bromo-2-chloro-1-methoxybenzene

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Key indicators

Single-crystal X-ray study
T = 90 K
Mean σ(C–C) = 0.002 Å
R factor = 0.019
wR factor = 0.041
Data-to-parameter ratio = 18.8

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

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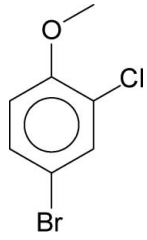
The title compound, C_7H_6BrClO, is a starting material for the synthesis of hydroxylated metabolites of polychlorinated biphenyls (PCBs). The title compound does not display any unusual bond distances and angles. The methoxy group is rotated slightly out of the plane of the benzene ring.

Comment

PCBs are an important group of persistent organic pollutants (Robertson & Hansen, 2001). Many PCB congeners are metabolized by cytochrome P-450 to hydroxylated metabolites. Some of these hydroxylated PCBs are persistent and have been found in wildlife and in humans, an observation that raises human health concerns (Bergman *et al.*, 1994; Letcher *et al.*, 2000). We have shown that some 4-hydroxy PCBs can be subject to phase II metabolism and are further metabolized by sulfonation (Liu *et al.*, 2006) or glucuronidation (Tampal *et al.*, 2002). During our attempts to synthesize several PCB metabolites for future structure–activity relationship (SAR) studies, we obtained the title compound, (I), as a precursor of the Suzuki coupling reaction.



(I)

For such SAR studies it is helpful to know the structure of the hydroxylated PCBs. Unfortunately, no crystal structures of relevant 4-hydroxy PCBs with a single *ortho*-chlorine substituent in a position *ortho* to the OH group have been reported to date. Moreover, only a few related structures, *e.g.* 2-chlorophenol (Oswald *et al.*, 2005) and 3-chloro-4,4′-dimethoxybiphenyl (Tan *et al.*, 2005), have been reported. Knowledge of the three-dimensional structure of the title compound, (I), may therefore be useful in understanding the phase II metabolism of hydroxylated PCB metabolites with a 3-chlorobiphenyl-4-ol substructure.

The title compound does not display any unusual bond distances and angles. The methoxy group is rotated slightly out of the plane of the benzene ring, with a C7—O1—C1—C6 torsion angle of 4.1 (2)°. A related structure with a chloro substituent *ortho* to the methoxy group also adopts a similar conformation in the solid state, with torsion angles of 7.1 (6)

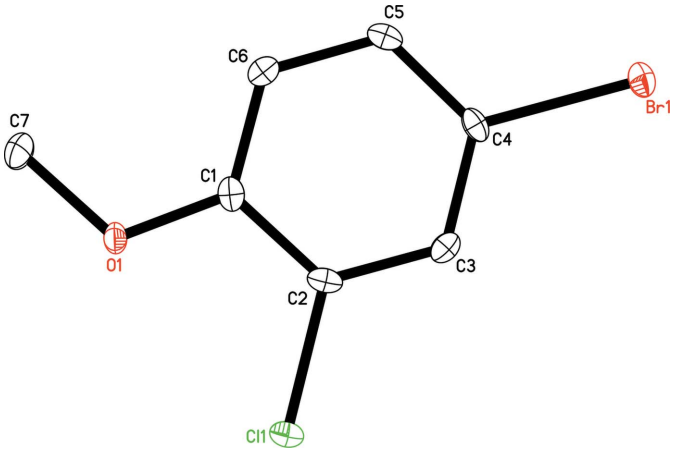


Figure 1
The molecular structure of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted.

and −6.2 (6)° (Tan *et al.*, 2005). With the exception of a close Br···O distance of 3.245 (2) Å between the molecule at (x, y, z) and its symmetry equivalent at ($x, \frac{3}{2} - y, z - \frac{1}{2}$), there are no noteworthy intermolecular interactions.

Experimental

The title compound was synthesized by methylation of 4-bromo-2-chlorophenol using well-known procedures (Lehmler & Robertson, 2001). Crystals suitable for X-ray diffraction analysis were grown by slow evaporation of a saturated solution of the title compound in $CHCl_3$.

Crystal data

C_7H_6BrClO
M_r = 221.47
Orthorhombic, *Pbca*
a = 10.7164 (1) Å
b = 8.1340 (2) Å
c = 17.4336 (4) Å
V = 1519.64 (5) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 5.68 mm^{-1}
T = 90.0 (2) K
0.30 × 0.28 × 0.24 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
T_{min} = 0.191, T_{max} = 0.256
22065 measured reflections
1747 independent reflections
1416 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.019
$wR(F^2)$ = 0.041
S = 0.96
1747 reflections
93 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

H atoms were found in difference Fourier maps and subsequently placed in idealized positions with constrained C—H distances of 0.98 (methyl) or 0.95 Å (aromatic) and U_{iso}(H) = 1.5U_{eq}(methyl C) or 1.2U_{eq}(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: *SHELXL97* and local procedures.

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