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2,2′,5′,6-Tetrachloro-4-[(1*S*)-1-methylpropoxy]biphenyl

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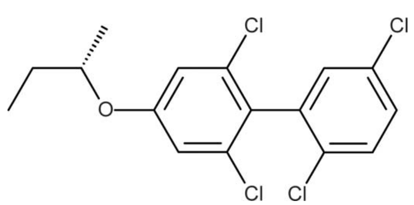
Key indicators: single-crystal X-ray study; T = 90 K; mean σ(C–C) = 0.003 Å; R factor = 0.034; wR factor = 0.081; data-to-parameter ratio = 19.8.

In the title molecule, $C_{16}H_{14}Cl_4O$, the dihedral angle between the least-square planes of the benzene rings is 84.40 (7)°. No unusual intermolecular interactions are present.

Related literature

For related literature about polychlorinated biphenyls, see: Lehmler *et al.* (2010); Warner *et al.* (2009). For crystal structures of PCB derivatives with two or less *ortho* chlorine substituents, see: Mannila & Rissanen (1994); Miao *et al.* (1996); Rissanen *et al.* (1988*a*); Shaikh *et al.* (2008); Singh *et al.* (1986); van der Sluis *et al.* (1990); Vyas *et al.* (2006). For crystal structures of PCB derivatives with three *ortho* chlorine substituents, see: Lehmler *et al.* (2005); Rissanen *et al.* (1988*b*). For crystal structures of PCB derivatives with four *ortho* chlorine substituents, see: Pedersen (1975); Singh & McKinney (1979). For literature about the Mitsunobu reaction, see: Fujita *et al.* (2001).



Experimental

Crystal data

$C_{16}H_{14}Cl_4O$
M_r = 364.07
Orthorhombic, $P2_12_12_1$
a = 10.3301 (2) Å
b = 10.5415 (2) Å
c = 15.2160 (3) Å
V = 1656.94 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.71 mm^{-1}
T = 90 K
0.25 × 0.25 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.843, T_{max} = 0.946
22321 measured reflections
3797 independent reflections
3351 reflections with $I > 2\sigma(I)$
R_{int} = 0.053

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.081
S = 1.09
3797 reflections
192 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Absolute structure: Flack (1983), 1625 Friedel pairs
Flack parameter: 0.00 (6)

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, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97* and local procedures.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG5329).

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2,2′,5′,6-Tetrachloro-4-[(1*S*)-1-methylpropoxy]biphenyl

Hans-Joachim Lehmler, Huimin Wu and Sean Parkin

S1. Comment

The title compound was synthesized as an intermediate in ongoing efforts to synthesize atropisomerically pure hydroxylated polychlorinated biphenyls (PCBs) for metabolism and toxicological studies (Lehmler *et al.*, 2010; Warner *et al.*, 2009). The dihedral angle between the two phenyl rings of the title compound, an important determinant of the toxicity of PCBs, was 84.40 (7)°. Comparable solid state dihedral (82–83°) have been reported for structurally related PCB derivatives with three *ortho* chlorine substituents (Lehmler *et al.*, 2005; Rissanen *et al.*, 1988*b*). Slightly larger (84–87°) dihedral angles have been observed for PCB derivatives with four *ortho* chlorine substituents (Pedersen, 1975; Singh & McKinney, 1979). Smaller solid state dihedral angles have been reported for PCB derivatives with zero, one or two *ortho* chlorine substituents due to the smaller steric demand of multiple hydrogen substituents in *ortho* position (Mannila & Rissanen, 1994; Miao *et al.*, 1996; Rissanen *et al.*, 1988*a*; Shaikh *et al.*, 2008; Singh *et al.*, 1986; van der Sluis *et al.*, 1990; Vyas *et al.*, 2006).

S2. Experimental

The title compound was synthesized by the Mitsunobu reaction of 2,2′,5′,6-tetrachloro-biphenyl-4-ol with (*R*)-isobutanol in THF (Fujita *et al.*, 2001). Crystals suitable for crystal structure analysis were obtained by slowly evaporating a methanolic solution of the title compound.

S3. Refinement

H atoms were found in difference Fourier maps and subsequently placed in idealized positions with constrained distances of 0.98 Å (RCH_3), 0.99 Å (R_2CH_2), 1.00 Å (R_3CH), 0.95 Å ($C_{sp2}H$), and with U_{iso}(H) values set to either 1.2U_{eq} or 1.5U_{eq} (RCH_3) of the attached atom. The absolute configuration was determined from 1625 Friedel pairs [Flack '*x*' = 0.00 (6)].

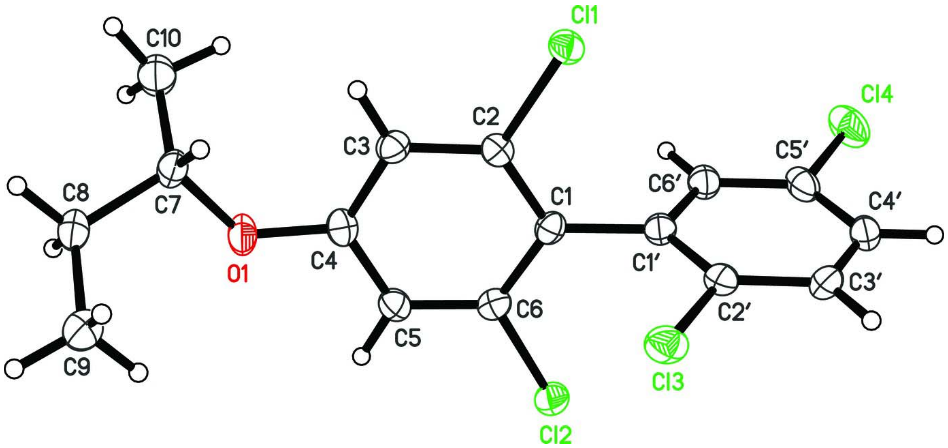

Figure 1

View of the title compound showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.

2,2′,5′,6-Tetrachloro-4-[(1*S*)-1-methylpropoxy]biphenyl

Crystal data

$C_{16}H_{14}Cl_4O$
$M_r = 364.07$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 10.3301$ (2) Å
$b = 10.5415$ (2) Å
$c = 15.2160$ (3) Å
$V = 1656.94$ (6) Å^3
$Z = 4$
$F(000) = 744$
$D_x = 1.459$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2184 reflections
$\theta = 1.0$–27.5°
$\mu = 0.71$ mm^{-1}
$T = 90$ K
Plate, colourless
0.25 × 0.25 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 9.1 pixels mm^{-1}
ω scans at fixed $\chi = 55°$
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
$T_{min} = 0.843$, $T_{max} = 0.946$
22321 measured reflections
3797 independent reflections
3351 reflections with $I > 2\sigma(I)$
$R_{int} = 0.053$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.4°$
$h = -13 \rightarrow 13$
$k = -13 \rightarrow 13$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.081$
$S = 1.09$
3797 reflections
192 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0401P)^2 + 0.5301P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

Absolute structure: Flack (1983), 1625 Friedel pairs

Absolute structure parameter: 0.00 (6)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against all reflections. The weighted R-value wR and goodness of fit S are based on F^2. Conventional R-values R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-values based on F^2 are statistically about twice as large as those based on F, and R-values based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	0.09110 (15)	0.46820 (16)	0.13102 (10)	0.0267 (4)
Cl1	0.32228 (5)	0.50012 (5)	0.42961 (3)	0.02188 (13)
Cl2	0.53731 (6)	0.28777 (6)	0.14184 (4)	0.02615 (14)
Cl3	0.44229 (6)	0.14044 (5)	0.36487 (4)	0.02755 (14)
Cl4	0.86539 (7)	0.55656 (7)	0.40389 (4)	0.03806 (18)
C1′	0.5425 (2)	0.3734 (2)	0.33248 (14)	0.0207 (5)
C1	0.4219 (2)	0.3999 (2)	0.28166 (14)	0.0188 (5)
C2	0.3142 (2)	0.4581 (2)	0.31899 (14)	0.0185 (4)
C3	0.2007 (2)	0.4834 (2)	0.27365 (14)	0.0206 (5)
H3	0.1296	0.5237	0.3018	0.025*
C4	0.1940 (2)	0.4478 (2)	0.18498 (15)	0.0215 (5)
C5	0.2990 (2)	0.3874 (2)	0.14558 (15)	0.0217 (5)
H5	0.2945	0.3625	0.0857	0.026*
C6	0.4089 (2)	0.3641 (2)	0.19355 (14)	0.0194 (5)
C7	−0.0337 (2)	0.5044 (2)	0.16686 (15)	0.0243 (5)
H7	−0.0453	0.4652	0.2262	0.029*
C8	−0.1328 (2)	0.4502 (2)	0.10372 (16)	0.0274 (5)
H8A	−0.1179	0.4871	0.0447	0.033*
H8B	−0.2203	0.4764	0.1232	0.033*
C9	−0.1291 (3)	0.3067 (3)	0.0965 (2)	0.0373 (7)
H9A	−0.0428	0.2797	0.0773	0.056*
H9B	−0.1938	0.2785	0.0536	0.056*
H9C	−0.1484	0.2691	0.1539	0.056*
C10	−0.0439 (3)	0.6479 (2)	0.17430 (17)	0.0325 (6)
H10A	0.0290	0.6802	0.2089	0.049*
H10B	−0.1254	0.6703	0.2034	0.049*
H10C	−0.0419	0.6855	0.1154	0.049*
C2′	0.5617 (2)	0.2565 (2)	0.37208 (15)	0.0235 (5)
C3′	0.6732 (3)	0.2293 (2)	0.41872 (16)	0.0303 (6)
H3′	0.6846	0.1480	0.4446	0.036*
C4′	0.7682 (3)	0.3210 (3)	0.42750 (17)	0.0315 (6)
H4′	0.8454	0.3033	0.4592	0.038*
C5′	0.7492 (2)	0.4387 (3)	0.38960 (15)	0.0265 (5)

C6′	0.6381 (2)	0.4661 (2)	0.34183 (15)	0.0232 (5)
H6′	0.6272	0.5472	0.3157	0.028*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0178 (8)	0.0421 (11)	0.0203 (8)	0.0042 (7)	−0.0031 (7)	−0.0005 (8)
Cl1	0.0234 (3)	0.0254 (3)	0.0169 (2)	0.0005 (2)	0.0008 (2)	−0.0006 (2)
Cl2	0.0243 (3)	0.0305 (3)	0.0237 (3)	0.0061 (2)	0.0010 (2)	−0.0041 (2)
Cl3	0.0366 (3)	0.0196 (3)	0.0264 (3)	−0.0011 (2)	−0.0004 (3)	0.0009 (2)
Cl4	0.0238 (3)	0.0563 (4)	0.0341 (3)	−0.0085 (3)	−0.0017 (3)	−0.0120 (3)
C1′	0.0194 (12)	0.0243 (11)	0.0182 (10)	0.0029 (10)	0.0008 (9)	−0.0018 (9)
C1	0.0194 (12)	0.0159 (10)	0.0212 (11)	0.0010 (9)	−0.0003 (9)	0.0032 (8)
C2	0.0201 (12)	0.0185 (10)	0.0168 (10)	−0.0016 (9)	0.0004 (9)	0.0018 (9)
C3	0.0198 (12)	0.0215 (12)	0.0205 (10)	0.0017 (9)	0.0020 (9)	−0.0004 (9)
C4	0.0194 (12)	0.0240 (11)	0.0209 (11)	−0.0024 (10)	−0.0016 (9)	0.0032 (10)
C5	0.0230 (12)	0.0238 (11)	0.0182 (11)	−0.0001 (9)	−0.0013 (10)	−0.0013 (9)
C6	0.0174 (11)	0.0185 (11)	0.0223 (11)	−0.0011 (9)	0.0040 (9)	0.0004 (9)
C7	0.0178 (12)	0.0292 (11)	0.0258 (11)	0.0039 (10)	−0.0020 (9)	−0.0002 (10)
C8	0.0209 (12)	0.0294 (13)	0.0319 (12)	0.0032 (11)	−0.0061 (11)	−0.0004 (11)
C9	0.0304 (14)	0.0307 (14)	0.0507 (17)	0.0060 (12)	−0.0084 (14)	−0.0089 (13)
C10	0.0313 (14)	0.0272 (13)	0.0388 (14)	0.0007 (12)	−0.0068 (12)	−0.0012 (11)
C2′	0.0278 (13)	0.0237 (11)	0.0189 (11)	0.0045 (10)	−0.0021 (10)	−0.0035 (9)
C3′	0.0382 (15)	0.0269 (13)	0.0258 (12)	0.0154 (11)	−0.0072 (11)	−0.0039 (10)
C4′	0.0281 (14)	0.0423 (16)	0.0243 (12)	0.0143 (12)	−0.0086 (11)	−0.0104 (11)
C5′	0.0168 (12)	0.0398 (14)	0.0228 (12)	−0.0010 (11)	−0.0001 (10)	−0.0090 (11)
C6′	0.0213 (12)	0.0274 (12)	0.0210 (11)	0.0013 (10)	0.0000 (9)	−0.0008 (9)

Geometric parameters (Å, º)

O1—C4	1.360 (3)	C7—C10	1.520 (3)
O1—C7	1.451 (3)	C7—H7	1.0000
Cl1—C2	1.743 (2)	C8—C9	1.518 (4)
Cl2—C6	1.739 (2)	C8—H8A	0.9900
Cl3—C2′	1.740 (3)	C8—H8B	0.9900
Cl4—C5′	1.741 (3)	C9—H9A	0.9800
C1′—C2′	1.386 (3)	C9—H9B	0.9800
C1′—C6′	1.396 (3)	C9—H9C	0.9800
C1′—C1	1.493 (3)	C10—H10A	0.9800
C1—C2	1.391 (3)	C10—H10B	0.9800
C1—C6	1.399 (3)	C10—H10C	0.9800
C2—C3	1.386 (3)	C2′—C3′	1.383 (3)
C3—C4	1.402 (3)	C3′—C4′	1.384 (4)
C3—H3	0.9500	C3′—H3′	0.9500
C4—C5	1.393 (3)	C4′—C5′	1.382 (4)
C5—C6	1.372 (3)	C4′—H4′	0.9500
C5—H5	0.9500	C5′—C6′	1.389 (3)
C7—C8	1.515 (3)	C6′—H6′	0.9500

C4—O1—C7	120.62 (17)	C7—C8—H8B	108.8
C2′—C1′—C6′	118.5 (2)	C9—C8—H8B	108.8
C2′—C1′—C1	120.7 (2)	H8A—C8—H8B	107.7
C6′—C1′—C1	120.8 (2)	C8—C9—H9A	109.5
C2—C1—C6	115.7 (2)	C8—C9—H9B	109.5
C2—C1—C1′	122.57 (19)	H9A—C9—H9B	109.5
C6—C1—C1′	121.7 (2)	C8—C9—H9C	109.5
C3—C2—C1	123.9 (2)	H9A—C9—H9C	109.5
C3—C2—Cl1	118.18 (17)	H9B—C9—H9C	109.5
C1—C2—Cl1	117.91 (17)	C7—C10—H10A	109.5
C2—C3—C4	118.0 (2)	C7—C10—H10B	109.5
C2—C3—H3	121.0	H10A—C10—H10B	109.5
C4—C3—H3	121.0	C7—C10—H10C	109.5
O1—C4—C5	114.89 (19)	H10A—C10—H10C	109.5
O1—C4—C3	125.2 (2)	H10B—C10—H10C	109.5
C5—C4—C3	119.9 (2)	C3′—C2′—C1′	121.8 (2)
C6—C5—C4	119.8 (2)	C3′—C2′—Cl3	118.48 (18)
C6—C5—H5	120.1	C1′—C2′—Cl3	119.76 (18)
C4—C5—H5	120.1	C2′—C3′—C4′	119.7 (2)
C5—C6—C1	122.7 (2)	C2′—C3′—H3′	120.2
C5—C6—Cl2	118.25 (17)	C4′—C3′—H3′	120.2
C1—C6—Cl2	119.03 (18)	C5′—C4′—C3′	119.1 (2)
O1—C7—C8	105.22 (18)	C5′—C4′—H4′	120.4
O1—C7—C10	110.6 (2)	C3′—C4′—H4′	120.4
C8—C7—C10	112.1 (2)	C4′—C5′—C6′	121.5 (2)
O1—C7—H7	109.6	C4′—C5′—Cl4	119.38 (19)
C8—C7—H7	109.6	C6′—C5′—Cl4	119.1 (2)
C10—C7—H7	109.6	C5′—C6′—C1′	119.5 (2)
C7—C8—C9	113.9 (2)	C5′—C6′—H6′	120.3
C7—C8—H8A	108.8	C1′—C6′—H6′	120.3
C9—C8—H8A	108.8		

C2′—C1′—C1—C2	94.3 (3)	C2—C1—C6—Cl2	−178.73 (16)
C6′—C1′—C1—C2	−85.2 (3)	C1′—C1—C6—Cl2	−0.5 (3)
C2′—C1′—C1—C6	−83.9 (3)	C4—O1—C7—C8	−149.7 (2)
C6′—C1′—C1—C6	96.7 (3)	C4—O1—C7—C10	89.1 (3)
C6—C1—C2—C3	−1.5 (3)	O1—C7—C8—C9	61.7 (3)
C1′—C1—C2—C3	−179.7 (2)	C10—C7—C8—C9	−178.1 (2)
C6—C1—C2—Cl1	177.90 (17)	C6′—C1′—C2′—C3′	−1.2 (3)
C1′—C1—C2—Cl1	−0.4 (3)	C1—C1′—C2′—C3′	179.3 (2)
C1—C2—C3—C4	0.2 (3)	C6′—C1′—C2′—Cl3	177.88 (16)
Cl1—C2—C3—C4	−179.12 (18)	C1—C1′—C2′—Cl3	−1.6 (3)
C7—O1—C4—C5	166.6 (2)	C1′—C2′—C3′—C4′	0.9 (4)
C7—O1—C4—C3	−14.0 (3)	Cl3—C2′—C3′—C4′	−178.25 (19)
C2—C3—C4—O1	−178.5 (2)	C2′—C3′—C4′—C5′	0.3 (4)
C2—C3—C4—C5	0.8 (3)	C3′—C4′—C5′—C6′	−1.0 (4)
O1—C4—C5—C6	178.9 (2)	C3′—C4′—C5′—Cl4	177.56 (19)

C3—C4—C5—C6	−0.6 (3)	C4′—C5′—C6′—C1′	0.7 (3)
C4—C5—C6—C1	−0.8 (3)	Cl4—C5′—C6′—C1′	−177.93 (17)
C4—C5—C6—Cl2	179.69 (18)	C2′—C1′—C6′—C5′	0.4 (3)
C2—C1—C6—C5	1.7 (3)	C1—C1′—C6′—C5′	179.9 (2)
C1′—C1—C6—C5	−180.0 (2)		