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[Chlorobis(*p*-chlorophenyl)(*p*-tolyl)tin]-μ-1,2-bis(diphenylphosphoryl)ethane-κ^2O:O'-[bromobis(*p*-chlorophenyl)(*p*-tolyl)tin]

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In the title complex, $[Sn_2BrCl(C_7H_7)_2(C_6H_4Cl)_4(C_{26}H_{24}O_2P_2)]$, the 1,2-bis(diphenylphosphoryl)ethane ligand coordinates to a bromobis(*p*-chlorophenyl)(*p*-tolyl)tin molecule and also to a chlorobis(*p*-chlorophenyl)(*p*-tolyl)tin molecule across a center of inversion, involving disorder of Cl and Br. The Sn atoms are five-coordinate in a *trans*-C_3SnOX geometry.

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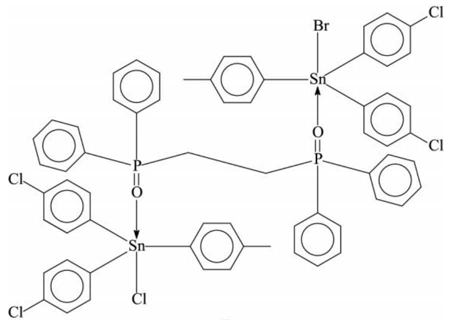
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.009 Å
Disorder in main residue
R factor = 0.054
wR factor = 0.114
Data-to-parameter ratio = 18.1

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

Comment

Triphenylphosphine oxide forms a large number of adducts with triorganotin Lewis acceptors (Lo *et al.*, 2001). The bidentate 1,2-bis(diphenylphosphoryl)ethane ligand does not chelate to triphenyltin chloride but instead binds through its double-bond O atoms to two of the Lewis acid molecules (Pelizzi & Pelizzi, 1980). The adduct is centrosymmetric, the center of symmetry being in the middle of the methylene–methylene linkage. A similar centrosymmetric arrangement is found in the mixed bis(4-chlorophenyl)(4-tolyl)bromotin complex (Wei *et al.*, 1990), as well as in the title complex, (I) (Fig. 1). Selected bond distances and angles are given in Table 1. The Sn atom is five-coordinate in a *trans*-C_3SnOX trigonal bipyramidal geometry. The 1/1 adduct of the ligand with butylphenyltin dichloride is also disordered over an inversion site (Ng & Kumar Das, 1996).



(I)

Experimental

Bis(*p*-chlorophenyl)di-*p*-tolyltin was prepared by the reaction of *p*-tolylmagnesium bromide and bis(*p*-chlorophenyl)tin dichloride in THF; the organotin reagent was itself prepared from another reaction between tetra(*p*-chlorophenyltin) (Ng, 1997) and stannic chloride. One of the *p*-tolyl rings was cleaved by using hydrochloric acid to form the mixed bis(*p*-chlorophenyl)(*p*-tolyl)tin chloride. The reagent (0.5 g, 1 mmol) and 1,2-bis(diphenylphosphoryl)ethane (0.4 g, 1 mmol) were each dissolved in ethanol and the two solutions mixed. The compound that separated was collected and recrystallized from chloroform. The molecule of bis(*p*-chlorophenyl)(*p*-tolyl)tin bromide in the crystal structure could have arisen from reaction between *p*-tolylmagnesium bromide and bis(*p*-chlorophenyl)tin dichloride, which could give this compound as a minor product. The compound was probably not removed in the subsequent steps.

Crystal data

$[Sn_2BrCl(C_7H_7)_2(C_6H_4Cl)_4$-
$(C_{26}H_{24}O_2P_2)]$
$M_r = 1411.55$
Monoclinic, $P2_1/n$
$a = 12.324$ (3) Å
$b = 12.842$ (3) Å
$c = 19.975$ (4) Å
$\beta = 107.54$ (3)°
$V = 3014.4$ (13) Å^3
$Z = 2$

$D_x = 1.555$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 10.0$–$13.4°$
$\mu = 1.81$ mm^{-1}
$T = 298$ (2) K
Block, colorless
0.15 × 0.15 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.744$, $T_{max} = 0.835$
5574 measured reflections
5308 independent reflections
3325 reflections with $I > 2\sigma(I)$

$R_{int} = 0.033$
$\theta_{max} = 25.0°$
$h = -14 \rightarrow 0$
$k = 0 \rightarrow 15$
$l = -22 \rightarrow 23$
3 standard reflections
frequency: 60 min
intensity decay: 3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.114$
$S = 1.04$
5308 reflections
294 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0381P)^2 + 3.4106P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.48$ e Å^{-3}
$\Delta\rho_{min} = -0.60$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn1—C1	2.122 (3)	Sn1—O1	2.367 (4)
Sn1—C7	2.125 (3)	Sn1—Cl1′	2.449 (9)
Sn1—C13	2.114 (3)	Sn1—Br1	2.618 (4)
C1—Sn1—C7	120.4 (2)	C7—Sn1—Cl1′	91.4 (6)
C1—Sn1—C13	122.3 (2)	C7—Sn1—Br1	95.4 (2)
C1—Sn1—O1	87.0 (2)	C13—Sn1—O1	83.4 (2)
C1—Sn1—Cl1′	93.7 (7)	C13—Sn1—Cl1′	97.5 (6)
C1—Sn1—Br1	93.8 (3)	C13—Sn1—Br1	93.4 (2)
C7—Sn1—C13	115.8 (2)	O1—Sn1—Cl1′	178.4 (6)
C7—Sn1—O1	87.0 (2)	O1—Sn1—Br1	176.7 (2)

The structure is disordered in terms of the tin-bound Cl and Br atoms, as well as in the chloro/methyl substituent of the tin-bound aryl rings. The occupancy of the pair Br1/Cl1′ was initially allowed to refine freely; as this refined to almost 0.5, the occupancy was then fixed at exactly 0.5. Two of the tin-bound aryl rings should be 4-chlorophenyl rings and one the 4-tolyl ring, so that there are two Cl atoms to be shared among three aryl rings. The occupancies of atoms Cl1, Cl2 and Cl3 were refined to 0.85:0.69:0.46. The occupancy of atom Cl1 was then fixed as unity; as the refinement led to occupancies of atoms Cl2 and Cl3 of 0.63:0.37, the occupancies were set at exactly 0.67:0.33. For the Br1/Cl1′ pair of disordered atoms, the Sn—Br distance was restrained to 2.65 (1) Å and the Sn—Cl distance to 2.45 (1) Å. These values were taken from the literature, the Sn—Br distance from the structure of the bromotriphenyl adduct with triphenylphosphine oxide (Ng, 1995) and the Sn—Cl distance from the bromotriphenyltin adduct with the same donor ligand (Ng & Kumar Das, 1992). Additionally, the displacement parameters of the two atoms were made equal.

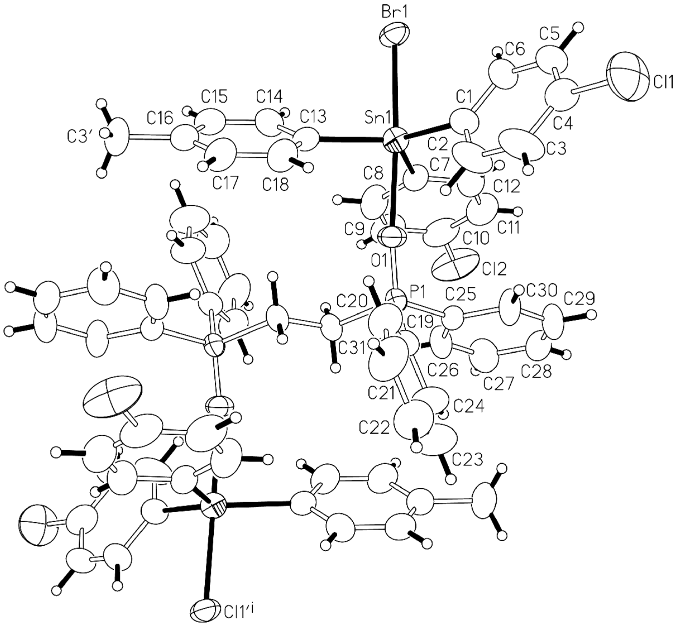


Figure 1
*ORTEP*II (Johnson, 1976) plot of the title complex (I), showing the atom-numbering scheme and displacement ellipsoids at the 50% probability level. [Symmetry code: (i) $1 - x, 1 - y, 1 - z$.]

The Cl—C_{aryl} [1.740 (5) Å] and CH_3—C_{aryl} [1.540 (5) Å] distances were also restrained, more tightly. For each ring, the atoms were restrained to lie in a plane within 0.01 Å. For each methyl group, the distances of the methyl C atom to the ring C atom two bonds apart were restrained to be equal.

All the aromatic rings were refined as rigid hexagons (C—C = 1.39 Å). The H atoms were placed at calculated positions [phenyl C—H = 0.93 Å and $U_{iso} = 1.2U_{eq}(C)$; methylene C—H = 0.97 Å and $U_{iso} = 1.2U_{eq}(C)$; methyl C—H = 0.96 Å and $U_{iso} = 1.5U_{eq}(C)$], and were included in the refinement in the riding-model approximation. The torsion angle of the methyl groups was refined.

Data collection: *CAD*-4/*PC* (Kretschmar, 1994); cell refinement: *CAD*-4 *VAX/PC Fortran System* (Enraf–Nonius, 1988); data reduction: *XCAD*4 (Harms, 1997) in *WinGX* (Farrugia, 1999); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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