The six phenyl rings and the 1,2-phenylene ring were refined as rigid hexagons. Each H atom was refined with an isotropic displacement parameter equal to $1.5U_{eq}$ of the parent C atom.

Data collection: *CAD*-4 *VAX*/*PC* (Enraf–Nonius, 1988). Cell refinement: *CAD*-4 *VAX*/*PC*. Data reduction: *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai & Pritzkow, 1994). Software used to prepare material for publication: *SHELXL*93.

I thank the University of Malaya (PJP 65/95) for supporting this work.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KH1076). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). C**52**, 1367-1369

Butyldichlorophenyltin–1,2-Bis(diphenylphosphoryl)ethane (1/1)

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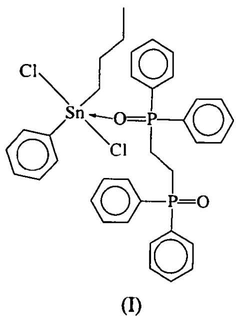
Abstract

The phosphine oxide ligand bridges the molecules of the Lewis acid in butyldichlorophenyltin–1,2-bis(diphenylphosphoryl)ethane (1/1), $[SnCl_2(C_4H_9)(C_6H_5)\{P_2O_2(C_2H_4)(C_6H_5)_4\}]$, to form chains, *i.e. catena*-poly[butyldichlorophenyltin-μ-1,2-bis(diphenylphosphoryl)ethane-*O*:*O*′]. The Sn atom shows distorted all-*trans* octahedral coordination.

Comment

The bis(diphenylphosphoryl)methane ligand chelates to the Sn atom of dibutyltin dichloride in an unsymmetrical bidentate manner [Sn—O = 2.29 (1), 2.58 (1) Å; Pelizzi, Tarasconi, Vitali & Pelizzi, 1987]. A second methylene linkage separating the diphenylphosphoryl groups should favour the formation of a seven-membered stannole ring, but the ligand instead links the dibutyldichlorotin molecules into a linear chain [Sn—O = 2.386 (7), 2.640 (7) Å; Harrison, Sharpe, Pelizzi, Pelizzi & Tarasconi, 1983]. The long dative bond results in steric repulsion between one of the two butyl chains of the Lewis acid and a phenyl ring of the adjacent Lewis base. The butyldichlorophenyltin complex of 1,2-bis(diphenylphosphoryl)ethane, (I), has a much stronger dative Sn—O bond [2.243 (2) Å] as the phenyl ring can rotate along the Sn—C axis to avoid such an interaction. The Sn—O—P angle [169.5 (1)°] is comparable with angles found in the dibutyldichlorotin



(I)

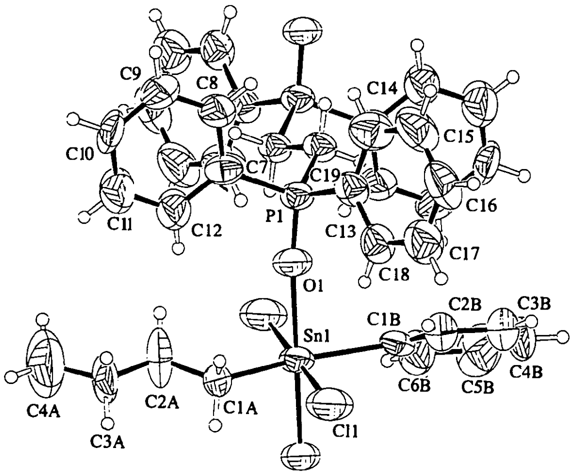


Fig. 1. *ZORTEP* (Zsolnai & Pritzkow, 1994) plot of the title complex at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

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adduct [163.9 (4), 167.8 (4)°; Harrison, Sharpe, Pelizzi, Pelizzi & Tarasconi, 1983] and other phosphine oxide complexes of organotin halides (Rheingold, Ng & Zuckerman, 1984).

Experimental

Crystals of the compound (Kumar Das, Ng, Smith & Hill, 1982) were grown from its solution in ethanol.

Crystal data

[$SnCl_2(C_4H_9)(C_6H_5)$-
($C_{26}H_{24}O_2P_2$)]
$M_r = 754.19$
Triclinic
$P\bar{1}$
$a = 9.1876\,(6)$ Å
$b = 9.593\,(1)$ Å
$c = 11.0473\,(7)$ Å
$\alpha = 78.839\,(8)°$
$\beta = 65.036\,(5)°$
$\gamma = 86.203\,(9)°$
$V = 865.9\,(2)$ Å^3
$Z = 1$
$D_x = 1.446$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 12–13°$
$\mu = 1.015$ mm^{-1}
$T = 300\,(2)$ K
Block
0.43 × 0.18 × 0.18 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
$T_{\min} = 0.972$, $T_{\max} = 1.000$
4217 measured reflections
3968 independent reflections

3138 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int} = 0.0076$
$\theta_{\max} = 27.47°$
$h = 0 \rightarrow 11$
$k = -12 \rightarrow 12$
$l = -12 \rightarrow 14$
3 standard reflections
frequency: 60 min
intensity decay: 1.4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0407$
$wR(F^2) = 0.1091$
$S = 1.063$
3968 reflections
325 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0618P)^2 + 0.2538P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.601$ e Å^{-3}
$\Delta\rho_{\min} = -0.768$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Sn1	0	0	0	0.0648 (2)
Cl1	0.0717 (1)	0.1771 (1)	0.1132 (1)	0.0942 (3)
P1	−0.43154 (8)	0.09398 (9)	0.13676 (7)	0.0496 (2)
O1	−0.2605 (3)	0.0538 (3)	0.0977 (2)	0.0677 (6)
C1†	−0.021 (2)	−0.106 (2)	0.187 (1)	0.052 (3)
C2†	−0.091 (3)	−0.256 (2)	0.223 (2)	0.098 (6)
C3†	−0.093 (4)	−0.326 (2)	0.362 (2)	0.11 (1)
C4†	−0.148 (5)	−0.480 (2)	0.400 (3)	0.18 (2)
C1*A*†	−0.064 (3)	−0.162 (3)	0.169 (2)	0.065 (7)
C2*A*†	0.053 (2)	−0.194 (2)	0.236 (2)	0.117 (7)
C3*A*†	−0.005 (3)	−0.312 (3)	0.362 (2)	0.13 (1)
C4*A*†	−0.172 (4)	−0.370 (4)	0.395 (5)	0.22 (3)
C1*B*	0.0273 (9)	0.1855 (5)	−0.1659 (5)	0.059 (3)
C2*B*	−0.0013 (8)	0.3261 (6)	−0.1491 (6)	0.083 (2)
C3*B*	0.0368 (9)	0.4342 (5)	−0.2620 (8)	0.107 (4)
C4*B*	0.104 (1)	0.4017 (8)	−0.3917 (6)	0.102 (6)
C5*B*	0.132 (1)	0.2611 (9)	−0.4085 (5)	0.124 (6)
C6*B*	0.094 (1)	0.1530 (6)	−0.2956 (6)	0.084 (2)
C7†	−0.551 (1)	−0.0187 (9)	0.2908 (6)	0.056 (6)
C8†	−0.713 (1)	0.0080 (8)	0.3606 (8)	0.082 (4)
C9†	−0.8039 (8)	−0.0790 (9)	0.4834 (8)	0.093 (4)
C10†	−0.733 (1)	−0.1926 (8)	0.5362 (7)	0.075 (6)
C11†	−0.571 (1)	−0.2193 (8)	0.4664 (9)	0.097 (4)
C12†	−0.4799 (9)	−0.132 (1)	0.3437 (9)	0.082 (4)
C13†	−0.447 (2)	0.269 (1)	0.154 (2)	0.056 (3)
C14†	−0.589 (1)	0.344 (1)	0.180 (2)	0.083 (4)
C15†	−0.592 (1)	0.487 (1)	0.190 (2)	0.097 (4)
C16†	−0.453 (1)	0.5539 (8)	0.174 (1)	0.090 (4)
C17†	−0.3121 (9)	0.478 (1)	0.148 (1)	0.095 (4)
C18†	−0.309 (1)	0.336 (1)	0.138 (1)	0.067 (3)
C7*A*†	−0.564 (1)	−0.0179 (8)	0.2935 (6)	0.045 (5)
C8*A*†	−0.726 (1)	−0.0406 (9)	0.3300 (9)	0.072 (3)
C9*A*†	−0.8179 (9)	−0.128 (1)	0.452 (1)	0.094 (4)
C10*A*†	−0.748 (1)	−0.192 (1)	0.5382 (7)	0.109 (9)
C11*A*†	−0.586 (1)	−0.1694 (9)	0.5017 (7)	0.082 (3)
C12*A*†	−0.4935 (8)	−0.0823 (9)	0.3794 (7)	0.063 (2)
C13*A*†	−0.475 (2)	0.281 (1)	0.162 (2)	0.062 (3)
C14*A*†	−0.624 (2)	0.338 (1)	0.176 (2)	0.080 (3)
C15*A*†	−0.660 (1)	0.474 (1)	0.203 (2)	0.102 (4)
C16*A*†	−0.549 (1)	0.5543 (9)	0.217 (1)	0.100 (4)
C17*A*†	−0.401 (1)	0.498 (1)	0.203 (1)	0.093 (4)
C18*A*†	−0.364 (1)	0.361 (1)	0.176 (1)	0.075 (3)
C19	−0.5019 (3)	0.0749 (3)	0.0111 (3)	0.0505 (6)

† Partial occupancy (see below).

Table 2. *Selected geometric parameters (Å, °)*

Sn1—C1	2.06 (1)	P1—O1	1.489 (2)
Sn1—C1*A*	2.08 (2)	P1—C7	1.770 (6)
Sn1—C1*B*	2.236 (4)	P1—C13	1.716 (8)
Sn1—O1	2.243 (2)	P1—C19	1.803 (3)
Sn1—O1^{i}	2.243 (2)	P1—C7*A*	1.817 (6)
Sn1—Cl1	2.559 (1)	P1—C13*A*	1.861 (8)
Sn1—Cl1^{i}	2.559 (1)	C19—C19^{ii}	1.500 (6)
C1—Sn1—C1*B*	157.6 (4)	O1^{i}—Sn1—Cl1	89.20 (7)
C1—Sn1—O1	88.4 (6)	O1^{i}—Sn1—Cl1^{i}	90.80 (7)
C1—Sn1—O1^{i}	91.6 (6)	Cl1—Sn1—Cl1^{i}	180.0
C1—Sn1—Cl1	72.6 (4)	O1—P1—C7	108.4 (3)
C1—Sn1—Cl1^{i}	107.4 (4)	O1—P1—C7*A*	111.5 (3)
C1*A*—Sn1—C1*B*	170.7 (8)	O1—P1—C13	107.8 (4)
C1*A*—Sn1—O1	83.3 (7)	O1—P1—C13*A*	114.7 (4)
C1*A*—Sn1—O1^{i}	96.7 (7)	O1—P1—C19	112.6 (1)
C1*A*—Sn1—Cl1	94.1 (8)	C7—P1—C13	111.6 (6)
C1*A*—Sn1—Cl1^{i}	85.9 (8)	C7—P1—C19	108.1 (3)
C1*B*—Sn1—O1	87.5 (2)	C7*A*—P1—C13*A*	106.5 (6)
C1*B*—Sn1—O1^{i}	92.5 (2)	C7*A*—P1—C19	105.9 (3)
C1*B*—Sn1—Cl1	85.5 (2)	C13—P1—C19	108.4 (6)
C1*B*—Sn1—Cl1^{i}	94.5 (2)	C19—P1—C13*A*	104.9 (5)
O1—Sn1—O1^{i}	180.0	P1—O1—Sn1	169.5 (1)
O1—Sn1—Cl1	90.80 (7)	C2—C1—Sn1	112.8 (9)
O1—Sn1—Cl1^{i}	89.20 (7)	C19^{ii}—C19—P1	113.6 (3)

Symmetry codes: (i) $-x, -y, -z$; (ii) $-1-x, -y, -z$.

The P and O atoms were found from the difference Fourier map calculated with Sn at (0,0,0). The Sn- and P-bonded phenyl rings were located from difference Fourier maps. Both P-bonded phenyl rings are disordered over two positions and they were refined as four rigid hexagons having 0.5 site occupancies. The butyl group was located in the latter stages of the refinement as two chains each of 0.25 site occupancies. The 1,2- and 1,3-related C atoms were constrained to be at 1.54±0.01 and 2.52±0.02 Å; both butyl chains were constrained to be flat. All non-H atoms were refined anisotropically. Each H atom was refined isotropically with $U_{\rm eq}$ equal to $1.5U_{\rm eq}$ of the parent C atom.

Data collection: *CAD*-4-*VAX/PC* (Enraf–Nonius, 1988). Cell refinement: *CAD*-4-*VAX/PC*. Data reduction: *NRCVAX* (Gabe, Le Page, Charland, Lee & White (1989). Program(s) used to solve structure: *SHELXL*93 (Sheldrick, 1993). Program(s) used to refine structure: *SHELXL*93. Molecular graphics: *ZORTEP* (Zsolnai & Pritzkow, 1994). Software used to prepare material for publication: *SHELXL*93.

We thank the University of Malaya (PJP 65/95) for supporting this work.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KH1077). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). **C52**, 1369–1371

Diisopropylammonium (Mercaptoacetato-*S*,*O*)triphenylstannate

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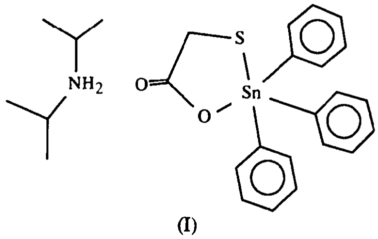
Abstract

In the title compound, $[NH_2(C_3H_7)_2][Sn(C_2H_2O_2S)(C_6H_5)_3]$, the mercaptoacetate dianion chelates to the Sn atom [Sn—S = 2.432 (1), Sn←O = 2.608 (3) Å, S—Sn—O = 74.2 (1)°], which displays *cis*-C_3SnOS trigonal bipyramidal coordination. Two stannate ions are linked together through two ammonium cations [N···O = 2.753 (5), 2.886 (5) Å] to give rise to a dimeric ion pair.

Comment

Triphenyltin mercaptides generally display tetrahedral coordination at the Sn atom as the Sn—S bond reduces the Lewis acidity of the triphenyltin cation, but in dicyclohexylammonium (2-mercaptobenzoato)triphenylstannate the carboxy entity engages in intramolecular coordination [Sn—O = 2.704 (3) Å] to tin. Hydrogen bonds [N···O = 2.803 (4), 2.880 (4) Å] from the cation link two ion pairs into a centrosymmetric dimer (Ng & Kumar Das, 1993). For the (mercaptoacetato)triphenylstannate analogue (Ng, Chin, Chen, Kumar Das & Mak, 1989), Mössbauer measurements also implied a *cis*-C_3SnOS trigonal bipyramidal geometry at Sn, but the insolubility of the compound in common organic solvents precluded its crystallographic verification.

The Sn atom in diisopropylammonium triphenyl(mercaptoacetato-*O*,*S*)stannate, (I), is five-coordinate in a *cis*-trigonal bipyramidal polyhedron whose apical positions are occupied by the C13 and O1 atoms [Sn—O = 2.608 (3) Å]. The Sn1—S1—C19—C20—O1 ring is not planar [Σ = 525 (1)°], and the S1—C19—C20 angle [115.1 (4)°] has been widened from the tetrahedral angle of 109.5°. Of the three Sn—C bonds, the axial bond [2.177 (5) Å] is statistically longer than the two equatorial bonds [2.144 (4), 2.146 (4) Å].



(I)

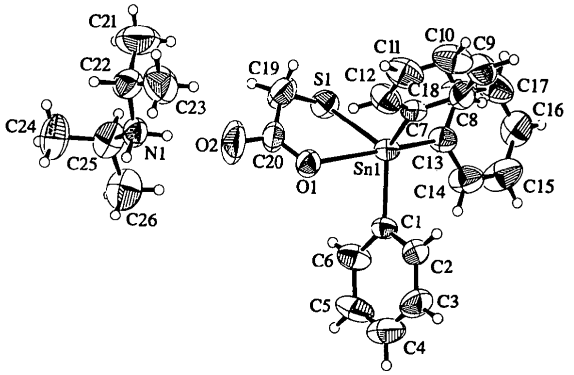


Fig. 1. Atomic labelling scheme for the title compound; displacement ellipsoids are plotted at the 50% probability level (*ZORTEP*; Zsolnai & Pritzkow, 1994).