Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *Xtal*3.0 (Hall & Stewart, 1990). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976) and *PLUTON*92 (Spek, 1992). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1141). 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**, 1365–1367

2-Triphenylstannyl-1,2-benzisothiazol-3(2*H*)-one 1,1-Dioxide–Triphenylphosphine Oxide (1/1)

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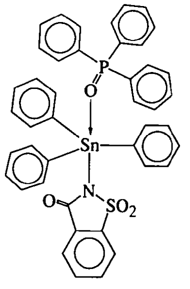
Abstract

The Sn atom in the 1/1 2-triphenylstannyl-1,2-benzisothiazol-3(2*H*)-one 1,1-dioxide–triphenylphosphine oxide complex, triphenyl(1,1,3-trioxo-2,3-dihydro-1,2-benzothiazol-2-yl)(triphenylphosphine oxide-*O*)tin, [Sn(C_6H_5)$_3$($C_7H_4NO_3S$){PO($C_{18}H_{15}$)}], shows *trans*-C_3SnNO trigonal bipyramidal coordination.

Comment

2-Triphenylstannyl-1,2-benzisothiazol-3(2*H*)-one 1,1-dioxide forms 1/1 complexes with carbonyl (Ng, Kuthubutheen, Zainudin, Chen, Kumar Das, Schulze, Molloy, Yip & Mak, 1991), amine oxide (Ng, 1994) and sulfoxide (Ng, Chen & Kumar Das, 1992) donor ligands. The present phosphine oxide complex, (I), adopts a similar structure with the Sn atom displaying *trans*-C_3SnNO trigonal bipyramidal coordination. The Sn—O bond in this complex is somewhat shorter than those found in the complexes mentioned above, reflecting the greater Lewis basicity of triphenylphosphine oxide. This distance [2.341 (3) Å] compares well with that [2.391 (4) Å] found in chlorotriphenyltin triphenyl-



(I)

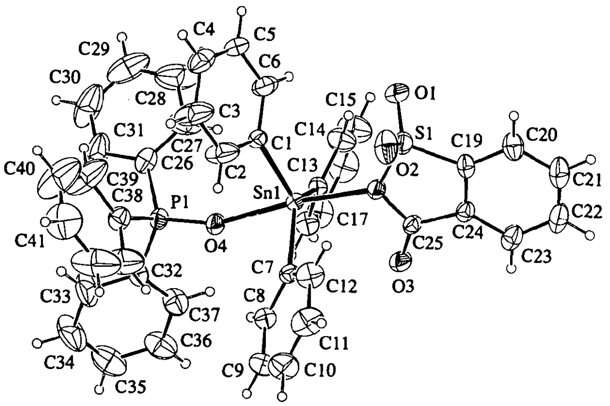


Fig. 1. *ZORTEP* (Zsolnai & Pritzkow, 1994) plot of the title complex at the 30% probability level. H atoms are drawn as spheres of arbitrary radii.

phosphine oxide (Ng & Kumar Das, 1992), which suggests that the stannylimide entity possesses Lewis acidity comparable with that of chlorotriphenyltin, a compound that forms a plethora of 1/1 adducts with O-atom donors.

Experimental

The compound was synthesized from triphenyltin hydroxide, saccharin and triphenylphosphine oxide (Ng, Kuthubutheen, Zainudin, Chen, Kumar Das, Schulze, Molloy, Yip & Mak, 1991), and crystals were grown from its solution in ethanol.

Crystal data

$[Sn(C_6H_5)_3(C_7H_4NO_3S)$-
$(C_{18}H_{15}OP)]$
$M_r = 810.43$
Monoclinic
$C2/c$
$a = 34.086\,(5)$ Å
$b = 10.0389\,(9)$ Å
$c = 23.778\,(4)$ Å
$\beta = 110.114\,(6)°$
$V = 7640\,(2)$ Å^3
$Z = 8$
$D_x = 1.409$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10$–$12°$
$\mu = 0.808$ mm^{-1}
$T = 300\,(2)$ K
Cube
$0.36 \times 0.36 \times 0.36$ mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: refined from ΔF (*DIFABS*; Walker & Stuart, 1983)
$T_{\min} = 0.841$, $T_{\max} = 1.000$
6696 measured reflections

6696 independent reflections
4556 observed reflections $[I > 2\sigma(I)]$
$\theta_{\max} = 24.98°$
$h = -40 \rightarrow 37$
$k = 0 \rightarrow 11$
$l = 0 \rightarrow 28$
3 standard reflections
frequency: 60 min
intensity decay: 0.4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0485$
$wR(F^2) = 0.1178$
$S = 1.018$
6696 reflections
376 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0570P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.457$ e Å^{-3}
$\Delta\rho_{\min} = -0.437$ 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	0.13363 (1)	0.24041 (3)	0.02357 (1)	0.0402 (1)
S1	0.12022 (4)	0.4416 (2)	0.13850 (6)	0.0553 (4)
P1	0.10697 (4)	0.0613 (1)	−0.12375 (6)	0.0475 (3)
O1	0.1017 (1)	0.5501 (4)	0.0998 (2)	0.083 (1)
O2	0.0933 (1)	0.3582 (5)	0.1573 (2)	0.087 (1)
O3	0.2176 (1)	0.3009 (4)	0.1293 (2)	0.0568 (9)
O4	0.1282 (1)	0.1210 (3)	−0.0634 (1)	0.0516 (9)
N1	0.1488 (1)	0.3524 (4)	0.1097 (2)	0.045 (1)
C1	0.0673 (1)	0.2628 (4)	−0.0085 (2)	0.045 (1)
C2	0.0439 (1)	0.1477 (3)	−0.0120 (2)	0.094 (2)
C3	0.0005 (1)	0.1530 (4)	−0.0359 (2)	0.122 (3)
C4	−0.0195 (1)	0.2733 (5)	−0.0565 (2)	0.080 (2)
C5	0.0039 (1)	0.3884 (4)	−0.0530 (2)	0.076 (2)
C6	0.0473 (1)	0.3831 (3)	−0.0291 (2)	0.073 (2)
C7	0.1521 (1)	0.0564 (3)	0.0699 (1)	0.045 (1)
C8	0.1745 (1)	−0.0397 (4)	0.0517 (2)	0.060 (2)
C9	0.1857 (1)	−0.1580 (3)	0.0837 (2)	0.088 (2)
C10	0.1745 (2)	−0.1801 (3)	0.1338 (2)	0.094 (2)
C11	0.1520 (1)	−0.0840 (5)	0.1520 (2)	0.093 (2)
C12	0.1408 (1)	0.0342 (4)	0.1201 (2)	0.071 (2)
C13	0.1685 (1)	0.3784 (3)	−0.0089 (2)	0.047 (1)
C14	0.1581 (1)	0.5128 (4)	−0.0125 (2)	0.076 (2)
C15	0.1792 (2)	0.6028 (3)	−0.0362 (2)	0.103 (3)
C16	0.2107 (1)	0.5584 (4)	−0.0563 (2)	0.096 (2)
C17	0.2211 (1)	0.4240 (5)	−0.0527 (2)	0.080 (2)
C18	0.2000 (1)	0.3340 (3)	−0.0290 (2)	0.062 (2)
C19	0.1617 (1)	0.4968 (3)	0.1999 (1)	0.049 (1)
C20	0.1630 (1)	0.5829 (4)	0.2463 (2)	0.069 (2)
C21	0.2009 (1)	0.6127 (4)	0.2905 (1)	0.080 (2)
C22	0.2376 (1)	0.5565 (4)	0.2883 (1)	0.078 (2)
C23	0.2364 (1)	0.4704 (4)	0.2420 (2)	0.063 (2)
C24	0.1984 (1)	0.4405 (3)	0.1978 (1)	0.045 (1)
C25	0.1910 (2)	0.3575 (5)	0.1436 (2)	0.044 (1)
C26	0.0856 (1)	0.1871 (4)	−0.1790 (2)	0.059 (2)
C27	0.0705 (2)	0.1568 (5)	−0.2398 (2)	0.121 (3)
C28	0.0537 (2)	0.2567 (7)	−0.2816 (2)	0.147 (5)
C29	0.0520 (2)	0.3869 (6)	−0.2627 (3)	0.126 (4)
C30	0.0671 (2)	0.4173 (4)	−0.2020 (3)	0.144 (4)
C31	0.0839 (2)	0.3174 (5)	−0.1601 (2)	0.109 (3)
C32	0.1438 (1)	−0.0335 (4)	−0.1459 (2)	0.055 (1)
C33	0.1311 (1)	−0.1352 (5)	−0.1880 (2)	0.107 (3)
C34	0.1607 (2)	−0.2063 (5)	−0.2040 (2)	0.129 (4)
C35	0.2028 (2)	−0.1758 (5)	−0.1779 (2)	0.108 (3)
C36	0.2155 (1)	−0.0742 (5)	−0.1358 (2)	0.093 (2)
C37	0.1859 (1)	−0.0030 (4)	−0.1198 (2)	0.074 (2)
C38	0.0661 (1)	−0.0523 (4)	−0.1247 (2)	0.062 (2)
C39	0.0772 (1)	−0.1585 (5)	−0.0848 (3)	0.128 (4)
C40	0.0468 (2)	−0.2469 (5)	−0.0810 (3)	0.167 (5)
C41	0.0054 (2)	−0.2292 (6)	−0.1172 (3)	0.137 (4)
C42	−0.0057 (1)	−0.1230 (7)	−0.1571 (3)	0.177 (5)
C43	0.0247 (2)	−0.0346 (6)	−0.1608 (2)	0.157 (5)

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

Sn1—C1	2.137 (3)	Sn1—N1	2.236 (4)
Sn1—C7	2.132 (3)	Sn1—O4	2.341 (3)
Sn1—C13	2.134 (3)		
C1—Sn1—C7	111.3 (1)	C7—Sn1—N1	91.6 (1)
C1—Sn1—C13	117.1 (1)	C7—Sn1—O4	86.1 (1)
C1—Sn1—N1	99.0 (1)	C13—Sn1—N1	91.3 (1)
C1—Sn1—O4	89.2 (1)	C13—Sn1—O4	84.2 (1)
C7—Sn1—C13	130.4 (1)	N1—Sn1—O4	171.8 (1)

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.

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

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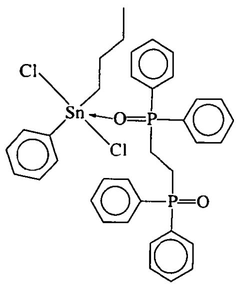
Abstract

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)

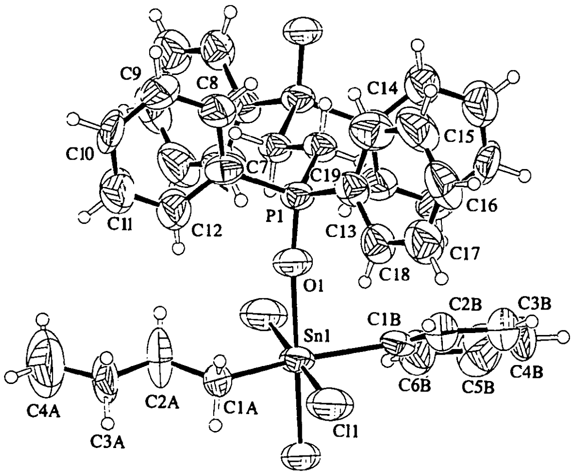


Fig. 1. *ZORTEP* (Zsolnai & Pritzkow, 1994) plot of the title complex at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

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