Related literature. The title compound was obtained in an attempt to form Cp′(PPh$_3$)(CO)Mn-*cyclo*-[=C—C(H)(Pr)C(CH$_3$)$_2$O] from acetone and $[(PPh_3)_2N]^+[Cp'(PPh_3)(CO)Mn—C{\equiv}CPr]^-$ in THF. There is only one Cp′Mn(CO)(phosphine)$_2$ structure found in the *Cambridge Structural Database* (1992). Barbeau & Dubey (1974) have reported the structure of [(η^5-Cp—Cp)-Mn(CO)(PPh$_3$)$_2$]·C$_6$H$_6$, but with considerably higher residuals and e.s.d.'s than the current structure.

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Acta Cryst. (1992). C**48**, 1834–1835

Structure of [2-(Chlorodimethylstannyl)ethyl]diphenylphosphine Selenide

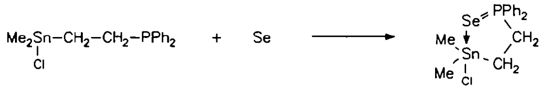
By Hans Preut, Bernd Godry and Terence N. Mitchell

Fachbereich Chemie der Universität Dortmund, Postfach 500500, *D*-4600 *Dortmund* 50, *Germany*

(*Received* 5 *December* 1991; *accepted* 6 *February* 1992)

Abstract. Chloro(ethyldiphenylphosphine selenido-*C,Se*)dimethyltin, $C_{16}H_{20}ClPSeSn$, $M_r = 476.41$, orthorhombic, $P2_12_12_1$, $a = 10.721$ (1), $b = 10.716$ (1), $c = 16.365$ (2) Å, $V = 1880.1$ (3) Å^3, $Z = 4$, $D_x = 1.683$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 3.50$ mm^{-1}, $F(000) = 928$, $T = 291$ (1) K, final $R = 0.048$ for 2879 unique observed [$F \geq 4.0\sigma(F)$] diffractometer data. The atoms bound to Sn form a distorted trigonal bipyramid with Se and Cl in the apical positions [Sn—Se 3.022 (1), Sn—Cl 2.500 (3) Å, Se—Sn—Cl 173.21 (8)°] and the C atoms in the equatorial positions [Sn—C 2.13 (1), 2.12 (1), 2.145 (9) Å, C—Sn—C 120.5 (4), 120.6 (5), 117.6 (5), C—Sn—Cl 90.4 (2), 93.7 (4), 97.4 (4), C—Sn—Se 85.0 (2), 84.3 (4), 89.3 (4)°]. The atoms bound to P form a slightly distorted tetrahedron with bond angles in the range 105.9 (4)–113.1 (2)°. The five-membered ring has a half-chair conformation; the atoms P, Se, Sn and C(3) are nearly coplanar. The molecules are separated by normal van der Waals contacts.

Experimental. The title compound was prepared by heating [2-(chlorodimethylstannyl)ethyl]diphenylphosphine (Weichmann, 1984) with an excess of Se powder in chloroform for 2 h. Colourless crystals were obtained by recrystallization of the crude product from hexane/chloroform; m.p. 391–392 K; yield 42%. A crystal of size ~0.26 × 0.40 × 0.42 mm was used. The crystal was mounted on the tip of a glass fibre with shellac. Intensity data were collected using $\omega/2\theta$ scans with variable scan speed of 1.5–15.0° min^{-1} in θ and scan width 1.2° + dispersion. A Nicolet *R*3*m*/*V* diffractometer with graphite-monochromated Mo $K\alpha$ radiation was used for preliminary examinations and data collection. The lattice parameters were determined from a symmetry-constrained least-squares fit of the angular settings for 50 reflections with $2\theta_{max} = 29.7°$. ω scans of low-order reflections along the three crystal axes showed acceptable mosaicity. Six standard reflections (400, 060, 006, $\bar{4}00$, $0\bar{6}0$, $00\bar{6}$) were recorded every 300 reflections, and showed only random deviations during 123.95 h of X-ray exposure. 10679 reflections with $2.0 \leq 2\theta \leq 50.0°$, $-13 \leq h \leq 13$, $-13 \leq k \leq 13$, $-20 \leq l \leq 20$ were measured. The data were cor-



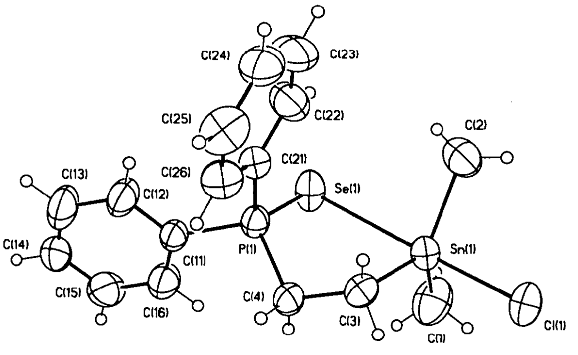


Fig. 1. General view (*SHELXTL-Plus*; Sheldrick, 1987) of the molecule, showing the atom-numbering scheme. Anisotropic ellipsoids represent 50% probability boundaries. H atoms are represented as spheres of arbitrary radii.

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Table 1. *Atomic coordinates and equivalent isotropic displacement parameters* (Å^2 × 10^4)

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

	x	y	z	U_{eq}
Sn(1)	0.90480 (6)	0.10277 (6)	0.23611 (4)	501
Se(1)	0.76660 (8)	0.1524 (1)	0.07848 (6)	574
Cl(1)	1.0345 (4)	0.0438 (3)	0.3571 (2)	897
P(1)	0.9429 (2)	0.1556 (2)	0.0182 (1)	417
C(1)	0.814 (1)	0.259 (1)	0.2896 (8)	921
C(2)	0.807 (1)	−0.069 (1)	0.2346 (8)	911
C(3)	1.0700 (8)	0.1271 (9)	0.1639 (5)	483
C(4)	1.0611 (7)	0.2114 (9)	0.0887 (5)	460
C(12)	0.9532 (7)	0.2091 (5)	−0.1485 (3)	615
C(13)	0.9445 (7)	0.2885 (5)	−0.2158 (3)	773
C(14)	0.9228 (7)	0.4156 (5)	−0.2043 (3)	753
C(15)	0.9098 (7)	0.4634 (5)	−0.1255 (3)	738
C(16)	0.9184 (7)	0.3841 (5)	−0.0582 (3)	633
C(11)	0.9401 (7)	0.2569 (5)	−0.0697 (3)	451
C(22)	0.9186 (5)	−0.1014 (6)	−0.0019 (4)	727
C(23)	0.9622 (5)	−0.2200 (6)	−0.0221 (4)	900
C(24)	1.0815 (5)	−0.2345 (6)	−0.0549 (4)	752
C(25)	1.1571 (5)	−0.1303 (6)	−0.0674 (4)	737
C(26)	1.1135 (5)	−0.0117 (6)	−0.0472 (4)	577
C(21)	0.9943 (5)	0.0028 (6)	−0.0144 (4)	454

Table 2. *Bond distances* (Å), *bond angles* (°), *torsion angles* (°), *least-squares planes*, *dihedral angle* (°) *and possible hydrogen bond* (Å, °)

Sn(1)—Se(1)	3.022 (1)	Se(1)—P(1)	2.132 (2)
Sn(1)—Cl(1)	2.500 (3)	P(1)—C(4)	1.815 (8)
Sn(1)—C(1)	2.13 (1)	P(1)—C(11)	1.803 (6)
Sn(1)—C(2)	2.12 (1)	P(1)—C(21)	1.808 (7)
Sn(1)—C(3)	2.145 (9)	C(3)—C(4)	1.53 (1)

C(2)—Sn(1)—C(3)	120.5 (4)	Se(1)—P(1)—C(11)	111.3 (2)
C(1)—Sn(1)—C(3)	120.6 (5)	Se(1)—P(1)—C(4)	109.3 (3)
C(1)—Sn(1)—C(2)	117.6 (5)	C(11)—P(1)—C(21)	108.4 (3)
Cl(1)—Sn(1)—C(3)	90.4 (2)	C(4)—P(1)—C(21)	105.9 (4)
Cl(1)—Sn(1)—C(2)	93.7 (4)	C(4)—P(1)—C(11)	108.6 (3)
Cl(1)—Sn(1)—C(1)	97.4 (4)	Sn(1)—C(3)—C(4)	117.6 (6)
Se(1)—Sn(1)—C(3)	85.0 (2)	P(1)—C(4)—C(3)	111.1 (6)
Se(1)—Sn(1)—C(2)	84.3 (4)	P(1)—C(11)—C(16)	118.9 (4)
Se(1)—Sn(1)—C(1)	89.3 (4)	P(1)—C(11)—C(12)	121.0 (4)
Se(1)—Sn(1)—Cl(1)	173.21 (8)	P(1)—C(21)—C(26)	119.6 (5)
Sn(1)—Se(1)—P(1)	87.88 (6)	P(1)—C(21)—C(22)	120.3 (5)
Se(1)—P(1)—C(21)	113.1 (2)		

Sn(1)—Se(1)—P(1)—C(4)	−30.3 (3)	Se(1)—Sn(1)—C(3)—C(4)	27.8 (6)
Se(1)—P(1)—C(4)—C(3)	59.6 (6)	C(3)—Sn(1)—Se(1)—P(1)	4.9 (2)
Sn(1)—C(3)—C(4)—P(1)	−58.3 (7)		

No.	Atoms in plane	Equation of the plane	χ^2
1	C(11),C(12),C(13),C(14),C(15), C(16)	$-0.9858x - 0.162y - 0.042z = -10.334$ Å	0.0
2	C(21),C(22),C(23),C(24),C(25), C(26)	$-0.374x + 0.103y - 0.922z = -3.77$ Å	0.0

	Dihedral angle
1,2	76.0 (2)

D—H···A	D···A	H···A	D—H···A
C(22)—H(22)···Se(1^{i})	3.433 (7)	2.879 (7)	117.8 (5)

Symmetry code: (i) x, y, z.

rected for Lorentz–polarization and for absorption effects (the latter *via* ψ-scan data, the maximum/minimum transmission factors being 1.00/0.77), and averaged ($R_{int} = 0.037$) to 3108 unique reflections, of which 2879 had $F \geq 4.0\sigma(F)$. The systematic absences ($h00$) $h = 2n + 1$, ($0k0$) $k = 2n + 1$, ($00l$) $l = 2n + 1$ conform to space group $P2_12_12_1$. The structure was solved *via* a Patterson function and $\Delta\rho$ maps. It was refined (on F) using full-matrix least squares with anisotropic displacement parameters for all non-H atoms and a common isotropic displacement parameter for the H atoms, which were placed in geometrically calculated positions (C—H 0.96 Å). The six-membered rings were refined as rigid bodies. Altogether, 158 parameters were refined. Weights, $w^{-1} = \sigma^2(F) + 0.0037F^2$, led to a featureless analysis of variance in terms of $\sin\theta$ and F_o. The refinement converged to $S = 1.39$, $R = 0.048$, $wR = 0.072$, $(\Delta/\sigma)_{max} \leq 0.000$ (no extinction correction). The structure was inspected for missed symmetries by using *MISSYM* (Le Page, 1987). The η refinement (Rogers, 1981) confirmed the proposed chirality. The largest peaks in the final $\Delta\rho$ map were ± 0.4 (3) e Å^{-3}. Atomic scattering factors for neutral atoms and real and imaginary dispersion terms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The programs used were *PARST* (Nardelli, 1983), *SHELXTL-Plus* (Sheldrick, 1987), *PLATON* (Spek, 1982) and *MISSYM* (Le Page, 1987). The molecule and the numbering scheme are shown in Fig. 1. Positional parameters and the equivalent values of the anisotropic displacement parameters for the non-H atoms are given in Table 1.* Bond lengths, bond angles, torsion angles, least-squares planes, a dihedral angle and a possible hydrogen bond are given in Table 2.

Related literature. The crystal structure of $(CH_3)_2Sn(Br)(CH_3)_2P(O)Ph^tBu$ is described by Weichmann, Mügge, Grand & Robert (1982), and literature on triorganotin halides with a pentacoordinated Sn centre resulting from the intramolecular coordination of a donor function is cited by these authors. The compound [2-(bromodimethylstannyl)ethyl]diphenylphosphine sulfide is isomorphous with the title compound (Preut, Godry & Mitchell, 1992).

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters, and a stereoscopic view of the unit cell have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55154 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL0530]

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