Table 1. *Atomic occupation factors (a), positional parameters and equivalent isotropic temperature parameters* (Å^2)

$B_{eq} = (4/3)\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	a	*x*	*y*	*z*	B_{eq}
K	0.327 (3)	0.36685 (10)	0.36685	0.36685	3.98 (3)
Si	0.653 (10)	0.37690 (7)	0.34345 (5)	0.08183 (6)	1.11 (1)
B	0.347	0.37690	0.34345	0.08183	1.11
O(1)	1.0	0.2908 (1)	0.4251 (1)	0.1200 (1)	1.50 (3)
O(2)	1.0	0.4871 (1)	0.3927 (1)	0.1092 (2)	2.11 (3)

Table 2. *Selected bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

$(Si,B)O_4$ tetrahedra

(Si,B)—O(1)	1.573 (2)	O(1)—(Si,B)—O(1^i)	111.24 (9)
—O(1^i)	1.567 (2)	—O(2)	106.7 (1)
—O(2)	1.562 (2)	—O(2^{ii})	108.0 (1)
—O(2^{ii})	1.554 (2)	O(1^i)—(Si,B)—O(2)	109.1 (1)
		—O(2^{ii})	109.6 (1)
O(1)—O(1^i)	2.592 (2)	O(2)—(Si,B)—O(2^{ii})	112.3 (1)
—O(2)	2.515 (2)		
—O(2^{ii})	2.530 (2)	(Si,B)—O(1)—(Si,B^{iii})	126.9 (1)
O(2)—O(1^i)	2.549 (3)	(Si,B)—O(2)—(Si,B^{iv})	144.2 (1)
—O(2)	2.588 (3)		
O(1^i)—O(2^{ii})	2.550 (2)		

Shortest contacts (<3.5 Å) of K atoms

K—O(1^v)	2.800 (2) × 3	K—O(1)	3.341 (2) × 3
—O(2^{ii})	3.291 (2) × 3	—O(2^v)	3.405 (2) × 3

Symmetry code: (i) $\frac{1}{4}+z, \frac{3}{4}-y, \frac{1}{4}-x$; (ii) $\frac{3}{4}-y, -\frac{1}{4}+x, \frac{1}{4}-z$; (iii) $\frac{1}{4}-z, \frac{3}{4}-y, -\frac{1}{4}+x$; (iv) $\frac{1}{4}+y, \frac{3}{4}-x, \frac{1}{4}-z$; (v) $x, 1-y, \frac{1}{2}-z$.

Related literature. Structure analysis results for $K(BSi_2O_6)$ were published by Kamei, Tsuda, Fukunaga, Yoshida & Ihara (1979) and Ihara & Kamei (1980). They refined the structure to $R = 0.0945$ using isotropic temperature factor coefficients (no occupancy factors were refined).

The authors are indebted to Dr J. Voldán for initiating this work and to Dr V. Figusch for supplying the crystals.

References

FIGUSCH, V. (1981). *Silikáty (Prague)*, **25**, 73–74.

IHARA, M. & KAMEI, F. (1980). *Yogyo Kyokai Shi*, **88**, 32–35.

JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

KAMEI, F., TSUDA, T., FUKUNAGA, J., YOSHIDA, N. & IHARA, M. (1979). *Asahi Garasu Kogyo Gijutsu Shoreikai Kenkyu Hokoku*, **34**, 313–327.

PEACOR, D. R. (1968). *Z. Kristallogr.* **127**, 213–224.

PETŘÍČEK, V. & MALÝ, K. (1988). The *SDS* system. Program package for X-ray structure determination. Institute of Physics, Czechoslovak Academy of Sciences.

Syntex (1976). The Syntex *XTL/E-XTL* structure determination system. Syntex Analytical Instruments, Inc., 10041 Bubb Road, Cupertino, CA 95014, USA.

VOLDÁN, J. (1979). *Silikáty (Prague)*, **23**, 133–143.

Acta Cryst. (1992). C**48**, 1832–1834

Carbonyl(1-methyl-η^5-cyclopentadienyl)bis(triphenylphosphine)manganese Dichloromethane Solvate

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(*Received* 18 *November* 1991; *accepted* 17 *February* 1992)

Abstract. $[Mn(C_{18}H_{15}P)_2(C_6H_7)(CO)]\cdot CH_2Cl_2$, $M_r = 771.53$, triclinic, $P\bar{1}$, $a = 9.875$ (3), $b = 14.794$ (5), $c = 14.917$ (5) Å, $\alpha = 71.99$ (2), $\beta = 70.41$ (2), $\gamma = 74.67$ (2)°, $V = 1920.8$ (9) Å^3, $Z = 2$, $D_x = 1.334$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 5.80$ cm^{-1}, $F(000) = 800$, $T = 296$ K, $R_{(F)} = 0.0514$ for 4733 observed reflections and 379 parameters. The structure around the central Mn atom has the expected three-legged piano-stool arrangement. The Cp′-ring methyl group is positioned between the two triphenylphosphine groups and nearly eclipses the carbonyl group (Cp′ = η^5-$C_5H_4CH_3$).

Experimental. An orange–red crystal from dichloromethane (0.41 × 0.41 × 0.52 mm) was used for data collection on a Nicolet *R3m* diffractometer with graphite-monochromated radiation and ω scans. Lattice parameters were determined from least-squares fit of 25 reflections ($20 \le 2\theta \le 25°$). No absorption correction was applied ($\mu = 5.80$ cm^{-1}).

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0108-2701/92/101832-03$06.00

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^3$)

Equivalent isotropic U is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Mn	5901.8 (6)	2232.9 (4)	3043.1 (4)	31.3 (2)
P(1)	3620 (1)	3038 (1)	3155 (1)	32 (1)
P(2)	6359 (1)	1463 (1)	1864 (1)	37 (1)
O(1)	7219 (3)	3847 (2)	1638 (2)	62 (1)
C(1)	6647 (4)	3205 (3)	2174 (3)	41 (2)
C(2)	5573 (5)	2166 (3)	4552 (3)	46 (2)
C(3)	5393 (5)	1278 (3)	4475 (3)	46 (2)
C(4)	6733 (5)	865 (3)	3903 (3)	46 (2)
C(5)	7723 (4)	1488 (3)	3626 (3)	47 (2)
C(6)	7020 (4)	2308 (3)	4036 (3)	45 (2)
C(7)	7748 (5)	3084 (4)	3982 (4)	66 (2)
C(11)	1865 (3)	1624 (2)	3913 (2)	50 (2)
C(12)	962	1031	4658	68 (2)
C(13)	441	1185	5601	73 (2)
C(14)	822	1932	5799	67 (2)
C(15)	1725	2524	5053	52 (2)
C(16)	2247	2371	4110	39 (2)
C(21)	1711 (2)	4692 (2)	3675 (2)	53 (2)
C(22)	1350	5592	3894	67 (2)
C(23)	2426	5991	3971	71 (3)
C(24)	3863	5491	3827	66 (3)
C(25)	4224	4591	3607	50 (2)
C(26)	3148	4192	3531	38 (2)
C(31)	3563 (2)	4242 (2)	1346 (2)	43 (2)
C(32)	3007	4739	547	50 (2)
C(33)	1725	4550	500	57 (2)
C(34)	998	3864	1251	61 (2)
C(35)	1553	3366	2051	52 (2)
C(36)	2835	3555	2098	36 (1)
C(41)	4676 (2)	2227 (2)	542 (2)	53 (2)
C(42)	4375	2748	−345	71 (3)
C(43)	5453	3178	−1117	81 (3)
C(44)	6831	3086	−1002	85 (3)
C(45)	7132	2565	−115	67 (2)
C(46)	6054	2136	657	44 (2)
C(51)	4662 (3)	25 (2)	2997 (2)	50 (2)
C(52)	4040	−773	3150	71 (3)
C(53)	4268	−1181	2372	81 (3)
C(54)	5119	−791	1441	83 (3)
C(55)	5742	7	1288	65 (2)
C(56)	5513	415	2066	44 (2)
C(61)	9346 (4)	1526 (2)	1269 (3)	74 (3)
C(62)	10834	1137	1084	112 (4)
C(63)	11305	146	1168	132 (5)
C(64)	10287	−456	1436	115 (4)
C(65)	8799	−67	1621	78 (3)
C(66)	8328	924	1537	53 (2)
Cl(1)	7766 (3)	5824 (3)	3413 (2)	192 (2)
Cl(2)	8834 (4)	7049 (3)	1513 (3)	267 (3)
C(100)	8578 (15)	5910 (7)	2166 (8)	240 (10)

$2\theta_{max} = 50°$ ($h = \pm 12$, $k = \pm 18$, $l = +18$). Three standard reflections ($\bar{5}2\bar{4}$, $0\bar{9}0$, $01\bar{7}$) showed 2% decay for 7057 reflections collected. 6772 reflections were unique ($R_{int} = 1.70\%$), of which 4733 were observed with $F_o > 5\sigma(F_o)$ (2106 unobserved reflections). Direct methods (*SOLV*) were used to solve the structure. Least-squares refinement was on 379 parameters. All non-H atoms were refined anisotropically; all H-atom positions were calculated (C—H = 0.960 Å, $U = 1.2U$ for attached C); phenyl rings were constrained as rigid planar hexagons (C—C = 1.395 Å). $R_{(F)} = 5.14$, $wR_{(F)} = 5.57\%$, $S = 1.668$ [$w^{-1} = \sigma^2(F_o) + gF_o^2$, $g = 0.0008$]; $(\Delta/\sigma)_{max} = 0.055$; $\Delta\rho_{max} = 0.645$, $\Delta\rho_{min} = -0.640$ e Å^{-3}. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99, 149). *SHELXTL* (Sheldrick, 1985) was used for computations.

Table 2. *Selected bond lengths* (Å) *and angles* (°)

Mn—P(1)	2.230 (1)	P(2)—C(56)	1.848 (4)
Mn—P(2)	2.243 (1)	P(2)—C(66)	1.855 (3)
Mn—C(1)	1.749 (4)	O(1)—C(1)	1.171 (5)
Mn—C(2)	2.138 (4)	C(2)—C(3)	1.415 (7)
Mn—C(3)	2.145 (3)	C(2)—C(6)	1.415 (6)
Mn—C(4)	2.153 (4)	C(3)—C(4)	1.411 (5)
Mn—C(5)	2.148 (4)	C(4)—C(5)	1.395 (7)
Mn—C(6)	2.166 (5)	C(5)—C(6)	1.434 (6)
P(1)—C(16)	1.841 (3)	C(6)—C(7)	1.475 (8)
P(1)—C(26)	1.856 (3)	Cl(1)—C(100)	1.740 (11)
P(1)—C(36)	1.852 (3)	Cl(2)—C(100)	1.702 (11)
P(2)—C(46)	1.848 (3)	Mn—CNT*	1.782 (3)
P(1)—Mn—CNT	120.7 (1)	Mn—P(1)—C(16)	112.9 (1)
P(1)—Mn—P(2)	103.9 (1)	Mn—P(1)—C(26)	116.7 (1)
P(1)—Mn—C(1)	92.5 (1)	Mn—P(1)—C(36)	123.9 (1)
P(2)—Mn—CNT	119.7 (1)	Mn—P(2)—C(46)	120.5 (1)
P(2)—Mn—C(1)	90.5 (2)	Mn—P(2)—C(56)	122.1 (1)
C(1)—Mn—CNT	122.9 (1)	Mn—P(2)—C(66)	108.9 (2)

*CNT = Centroid of atoms C(2) to C(6).

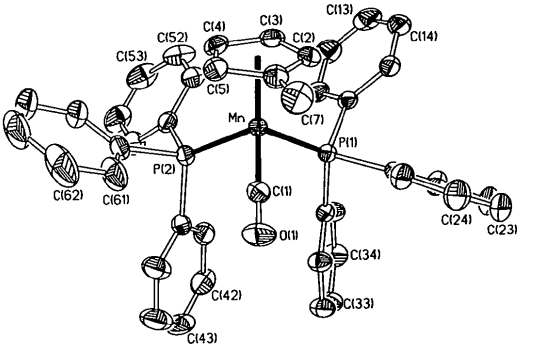

Fig. 1. The molecular structure of $[Mn(PPh_3)_2Cp'(CO)]$.

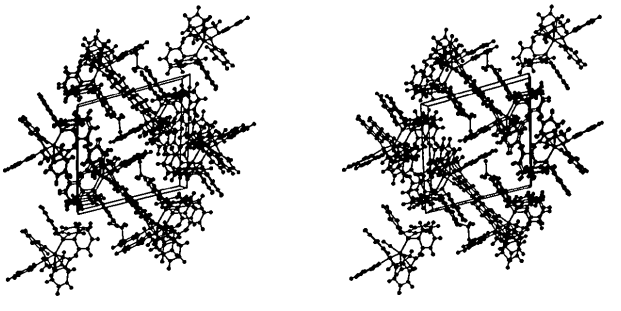

Fig. 2. Unit-cell packing diagram viewed down the *a* axis.

Atomic coordinates and isotropic thermal parameters are given in Table 1.* Selected bond lengths and angles are given in Table 2. The cocrystallized solvent molecule is labeled C(100), Cl(1), Cl(2), H(100*A*) and H(100*B*); the atom-labeling scheme of the Mn molecule is shown in Fig. 1. Fig. 2 shows the unit-cell packing.

* Lists of structure factors, anisotropic thermal parameters, intramolecular angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55194 (24 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH0611]

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$_3$)$_2$N]$^+$[Cp′(PPh$_3$)(CO)Mn—C≡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.

References

BARBEAU, C. & DUBEY, R. J. (1974). *Can. J. Chem.* **52**, 1140–1146.
Cambridge Structural Database (1992). Version 4.6. Cambridge Crystallographic Data Centre, Cambridge CB2 1EW, England.
SHELDRICK, G. M. (1985). *SHELXTL User's Manual.* Revsion 5.1. Nicolet XRD Corporation, Madison, Wisconsin, USA.

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

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

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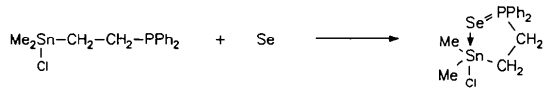
(*Received* 5 *December* 1991; *accepted* 6 *February* 1992)

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-

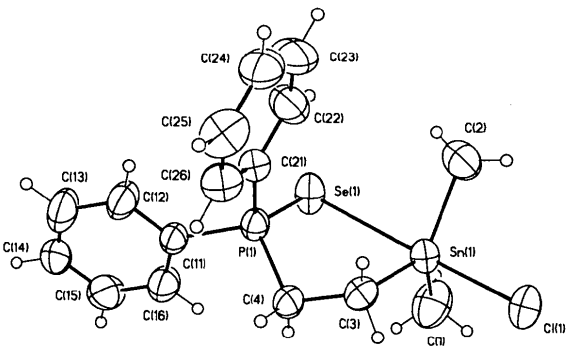


Fig. 1. General view (*SHELXTL-Plus*; Sheldrick, 1987) of the molecule, showing the atom-numbering scheme. Anisotropic ellipsoids represent 50% probability boundaries. H atoms are represented as spheres of arbitrary radii.

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