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Chlorotris[tetracarbonyl(triphenylphosphine)manganio]tin(IV)

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Abstract. $[SnCl\{Mn(C_{18}H_{15}P)(CO)_4\}_3]$, $C_{66}H_{45}ClMn_3O_{12}PSn$, $M_r = 1442$, triclinic, $P\bar{1}$, $a = 13{\cdot}413\,(3)$, $b = 22{\cdot}768\,(4)$, $c = 10{\cdot}916\,(3)$ Å, $\alpha = 85{\cdot}91\,(10)$, $\beta = 89{\cdot}76\,(10)$, $\gamma = 107{\cdot}74\,(10)°$, $U = 3166$ Å^3, $Z = 2$, $D_c = 1{\cdot}513$ Mg m^{-3}, $F(000) = 1448$, Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71069$ Å, $\mu(\text{Mo}\,K\alpha) = 10{\cdot}7$ mm^{-1}. Final $R = 0{\cdot}056$ for 7633 unique diffractometer data ($I > 3\sigma$). The three $Mn[(C_6H_5)_3P](CO)_4$ groups and the Cl atom form a distorted tetrahedron round the Sn atom with Sn—Mn distances 2·718 (1), 2·715 (1), 2·705 (1) Å and Sn—Cl 2·461 (2) Å.

Introduction. Red crystals were prepared by reaction of $SnCl_2$ with $[Mn_2\{(C_6H_5)_3P\}_2(CO)_8]$ in 1,2,3,4-tetrahydronaphthalene at 403 K in a Schlenk tube at atmospheric pressure (Haupt & Schwab, 1978).

Data were collected with a crystal 0·23 × 0·30 × 0·52 mm. Cell parameters were determined by least squares from the diffractometer angles of 19 reflexions measured with a Hilger & Watts Y290 automatic four-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation and a scintillation counter. The intensities of 8325 reflexions ($I > 3\sigma$) with $2 \le \theta \le 24{\cdot}6°$ were measured by the $\omega/2\theta$ scan technique, with a scan width $\Delta 2\theta = 1{\cdot}34° + 0{\cdot}34°\tan\theta$ from background to background and a scan speed of 0·02° s^{-1} in 2θ. Backgrounds were measured at each end of the scan range for 7 s. Five standards were measured every fifty reflexions, and showed only random deviations from their mean intensities. Lp but no absorption corrections were applied, and after averaging the equivalent reflexions the data set contained 7633 independent reflexions.

The structure was solved by Patterson and Fourier methods and refined by blocked full-matrix least squares with anisotropic temperature factors for Sn, Mn, Cl and P, isotropic for C and O, with *SHELX* (Sheldrick, 1976). The phenyl rings were treated as rigid bodies (C—C = 1·395 Å, C—C—C = 120°). Complex neutral-atom scattering factors were from *International Tables for X-ray Crystallography* (1974) and Cromer & Liberman (1970). Refinement converged to $R = 0{\cdot}056$ with unit weights.*

The data were processed on an IBM 370/158 computer. Figures were drawn with *PLUTO* (W. D. S. Motherwell & W. Clegg) and *POP*1 (B. W. van de Waal).

Discussion. The molecular structure is shown in Fig. 1; a stereoview of the molecule is given in Fig. 2. The

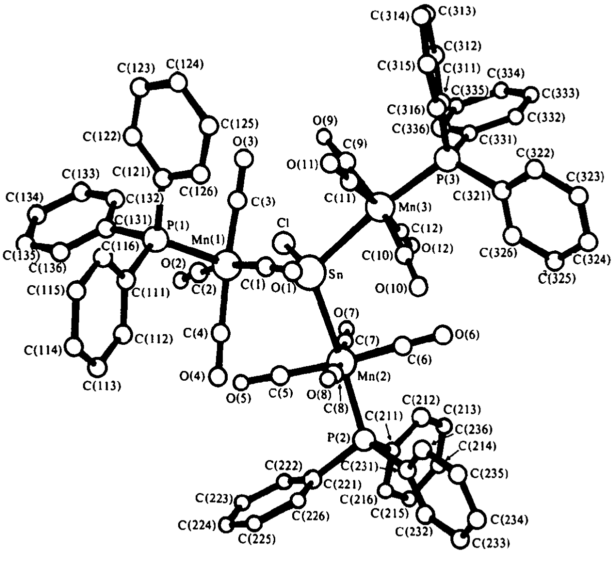

Fig. 1. General view of the molecule.

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* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35816 (49 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Fractional coordinates* ($\times 10^4$) *and* *U* ($Å^2 \times 10^4$)

For each rigid phenyl ring only the coordinates of three C atoms are given.

	x	*y*	*z*	*U*
Sn	875 (0)	7586 (0)	2825 (0)	255 (2)
Cl	2074 (1)	7404 (1)	4404 (2)	396 (9)
Mn(1)	1292 (1)	6941 (0)	990 (1)	281 (5)
P(1)	2007 (1)	6561 (1)	−580 (2)	316 (8)
C(1)	714 (6)	7414 (4)	5 (7)	471 (19)
O(1)	276 (5)	7700 (3)	−585 (6)	702 (17)
C(2)	1645 (5)	6464 (3)	2210 (6)	370 (16)
O(2)	1835 (4)	6143 (3)	2980 (5)	563 (14)
C(3)	2556 (5)	7535 (3)	1019 (6)	364 (16)
O(3)	3368 (4)	7905 (2)	1037 (5)	505 (13)
C(4)	9999 (6)	6381 (4)	1094 (7)	470 (19)
O(4)	9157 (5)	6029 (3)	1176 (6)	706 (17)
C(111)	1156 (4)	6100 (2)	−1697 (4)	369 (16)
C(113)	9593 (4)	5279 (2)	−2117 (4)	639 (24)
C(115)	819 (4)	5798 (2)	−3769 (4)	574 (22)
C(121)	2922 (4)	7175 (2)	−1578 (5)	404 (17)
C(123)	4624 (4)	7681 (2)	−2536 (5)	999 (37)
C(125)	3205 (4)	8112 (2)	−2898 (5)	676 (25)
C(131)	2789 (4)	6063 (2)	−30 (5)	442 (18)
C(133)	4138 (4)	5917 (2)	1306 (5)	753 (28)
C(135)	3165 (4)	5088 (2)	109 (5)	887 (33)
Mn(2)	8989 (1)	7141 (0)	4027 (1)	306 (5)
P(2)	7350 (1)	6711 (1)	4876 (2)	344 (9)
C(5)	9222 (6)	6399 (3)	3955 (7)	429 (17)
O(5)	9378 (5)	5924 (3)	3903 (5)	607 (15)
C(6)	8888 (6)	7907 (3)	4153 (7)	413 (17)
O(6)	8799 (5)	8393 (3)	4280 (6)	645 (16)
C(7)	9688 (6)	7220 (3)	5470 (7)	437 (18)
O(7)	93 (5)	7259 (3)	6405 (6)	680 (17)
C(8)	8560 (6)	7120 (4)	2481 (8)	494 (19)
O(8)	8318 (5)	7113 (3)	1446 (6)	771 (19)
C(211)	7139 (5)	6775 (3)	6526 (4)	464 (18)
C(213)	7483 (5)	7406 (3)	8257 (4)	952 (35)
C(215)	6193 (5)	6395 (3)	8454 (4)	869 (32)
C(221)	6887 (5)	5881 (2)	4678 (5)	454 (18)
C(223)	7001 (5)	4855 (2)	5201 (5)	867 (32)
C(225)	6235 (5)	5077 (2)	3292 (5)	975 (37)
C(231)	6290 (4)	6990 (2)	4232 (5)	403 (17)
C(233)	4501 (4)	7006 (2)	4397 (5)	916 (34)
C(235)	5642 (4)	7613 (2)	2744 (5)	640 (24)
Mn(3)	1590 (1)	8834 (0)	2484 (1)	280 (5)
P(3)	2030 (1)	9887 (1)	2462 (2)	297 (8)
C(9)	2878 (5)	8793 (3)	2858 (6)	375 (16)
O(9)	3716 (4)	8772 (3)	3079 (5)	582 (15)
C(10)	280 (6)	8751 (3)	1953 (7)	411 (17)
O(10)	9456 (5)	8683 (3)	1551 (5)	625 (15)
C(11)	1986 (5)	8822 (3)	903 (7)	393 (17)
O(11)	2252 (4)	8815 (3)	−111 (5)	589 (15)
C(12)	1257 (5)	8747 (3)	4117 (6)	385 (16)
O(12)	1086 (4)	8690 (3)	5166 (5)	576 (15)
C(311)	2907 (4)	288 (2)	1176 (4)	405 (17)
C(313)	4642 (4)	847 (2)	363 (4)	885 (33)
C(315)	3204 (4)	521 (2)	−1014 (4)	749 (28)
C(321)	917 (3)	197 (2)	2386 (4)	361 (16)
C(323)	32 (3)	919 (2)	1602 (4)	696 (26)
C(325)	9226 (3)	147 (2)	3242 (4)	597 (22)
C(331)	2714 (4)	272 (2)	3787 (4)	354 (16)
C(333)	3286 (4)	1191 (2)	4902 (4)	549 (21)
C(335)	3852 (4)	304 (2)	5508 (4)	613 (23)

Table 2. *Bond lengths* (Å) *and angles* (°)

Sn—Cl	2·461 (2)	C(4)—O(4)	1·167 (9)
Sn—Mn(1)	2·718 (1)	C(5)—O(5)	1·167 (10)
Sn—Mn(2)	2·715 (1)	C(6)—O(6)	1·168 (10)
Sn—Mn(3)	2·705 (1)	C(7)—O(7)	1·156 (10)
Mn(1)—C(1)	1·806 (7)	C(8)—O(8)	1·176 (11)
Mn(1)—C(2)	1·814 (7)	C(9)—O(9)	1·165 (7)
Mn(1)—C(3)	1·820 (6)	C(10)—O(10)	1·161 (7)
Mn(1)—C(4)	1·811 (7)	C(11)—O(11)	1·164 (9)
Mn(1)—P(1)	2·304 (2)	C(12)—O(12)	1·157 (9)
Mn(2)—C(5)	1·816 (8)	Cl—Sn—Mn(1)	101·1 (1)
Mn(2)—C(6)	1·804 (8)	Cl—Sn—Mn(2)	101·9 (1)
Mn(2)—C(7)	1·828 (7)	Cl—Sn—Mn(3)	100·2 (1)
Mn(2)—C(8)	1·784 (8)	Mn(1)—Sn—Mn(2)	118·14 (5)
Mn(2)—P(2)	2·279 (2)	Mn(1)—Sn—Mn(3)	116·85 (5)
Mn(3)—C(9)	1·804 (5)	Mn(2)—Sn—Mn(3)	114·17 (5)
Mn(3)—C(10)	1·811 (7)	Sn—Mn(1)—C(1)	85·0 (2)
Mn(3)—C(11)	1·807 (7)	Sn—Mn(1)—C(2)	85·4 (2)
Mn(3)—C(12)	1·819 (7)	Sn—Mn(1)—C(3)	80·9 (2)
Mn(3)—P(3)	2·286 (2)	Sn—Mn(1)—C(4)	93·6 (3)
P(1)—C(111)	1·833 (5)	Sn—Mn(1)—P(1)	167·1 (1)
P(1)—C(121)	1·836 (5)	C(1)—Mn(1)—C(2)	167·6 (3)
P(1)—C(131)	1·838 (5)	C(1)—Mn(1)—C(3)	94·2 (3)
P(2)—C(211)	1·844 (5)	C(1)—Mn(1)—C(4)	85·9 (3)
P(2)—C(221)	1·830 (5)	C(1)—Mn(1)—P(1)	95·7 (2)
P(2)—C(231)	1·848 (4)	C(2)—Mn(1)—C(3)	92·1 (3)
P(3)—C(311)	1·826 (5)	C(2)—Mn(1)—C(4)	86·8 (3)
P(3)—C(321)	1·835 (4)	C(2)—Mn(1)—P(1)	95·4 (2)
P(2)—C(331)	1·845 (5)	C(3)—Mn(1)—C(4)	174·5 (3)
C(1)—O(1)	1·166 (9)	C(3)—Mn(1)—P(1)	86·2 (2)
C(2)—O(2)	1·154 (9)	C(4)—Mn(1)—P(1)	99·3 (3)
C(3)—O(3)	1·158 (8)		
Sn—Mn(2)—C(5)	83·7 (2)	C(10)—Mn(3)—P(3)	93·6 (3)
Sn—Mn(2)—C(6)	92·5 (2)	C(11)—Mn(3)—C(12)	171·6 (3)
Sn—Mn(2)—C(7)	88·1 (2)	C(11)—Mn(3)—P(3)	95·5 (2)
Sn—Mn(2)—C(8)	80·6 (2)	C(12)—Mn(3)—P(3)	91·5 (2)
Sn—Mn(2)—P(2)	174·39 (7)	Mn(1)—P(1)—C(111)	120·1 (1)
C(5)—Mn(2)—C(6)	174·3 (3)	Mn(1)—P(1)—C(121)	112·8 (2)
C(5)—Mn(2)—C(7)	87·6 (3)	Mn(1)—P(1)—C(131)	113·1 (2)
C(5)—Mn(2)—C(8)	91·0 (4)	C(111)—P(1)—C(121)	102·0 (2)
C(5)—Mn(2)—P(2)	93·5 (2)	C(111)—P(1)—C(131)	102·9 (2)
C(6)—Mn(2)—C(7)	88·1 (3)	C(121)—P(1)—C(131)	104·2 (2)
C(6)—Mn(2)—C(8)	92·6 (4)	Mn(2)—P(2)—C(211)	118·9 (2)
C(6)—Mn(2)—P(2)	90·7 (2)	Mn(2)—P(2)—C(221)	110·4 (2)
C(7)—Mn(2)—C(8)	168·6 (3)	Mn(2)—P(2)—C(231)	117·5 (2)
C(7)—Mn(2)—P(2)	96·7 (2)	C(211)—P(2)—C(221)	105·0 (3)
C(8)—Mn(2)—P(2)	94·6 (2)	C(211)—P(2)—C(231)	100·0 (2)
Sn—Mn(3)—C(9)	88·3 (2)	C(221)—P(2)—C(231)	103·3 (3)
Sn—Mn(3)—C(10)	83·7 (3)	Mn(3)—P(3)—C(311)	113·3 (2)
Sn—Mn(3)—C(11)	93·1 (2)	Mn(3)—P(3)—C(321)	114·8 (2)
Sn—Mn(3)—C(12)	80·3 (2)	Mn(3)—P(3)—C(331)	118·8 (2)
Sn—Mn(3)—P(3)	170·9 (1)	C(311)—P(3)—C(321)	105·5 (2)
C(9)—Mn(3)—C(10)	169·5 (4)	C(311)—P(3)—C(331)	102·2 (2)
C(9)—Mn(3)—C(11)	85·8 (3)	C(321)—P(3)—C(331)	100·4 (2)
C(9)—Mn(3)—C(12)	88·9 (3)		
C(9)—Mn(3)—P(3)	95·4 (2)		
C(10)—Mn(3)—C(11)	87·8 (3)		
C(10)—Mn(3)—C(12)	96·4 (3)		

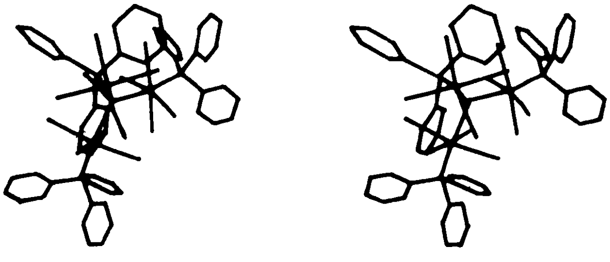

Fig. 2. Stereoscopic view of the molecule.

positional parameters and the equivalent values of the anisotropic temperature factors U_{ij} [$U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33} + 2U_{23}\cos\alpha + 2U_{13}\cos\beta + 2U_{12}\cos\gamma)$] or the isotropic temperature factors U are given in the column headed U in Table 1. Bond lengths and angles are given in Table 2.

Through ligand substitution of the stronger π-acceptor CO for $P(C_6H_5)_3$ at the Mn atom *trans* to the Sn^{IV} atom, the Sn—Mn bond is shortened compared with those in $[SnCl\{Mn(CO)_5\}_3]$ (Tsai, Flynn & Boer, 1967) [Sn—Mn: 2·720 (6), 2·746 (6), 2·753 (6), 2·703 (6), 2·745 (6), 2·758 (6) Å] and $[SnBr\{Mn(CO)_5\}_3]$ (Haupt, Preut & Wolfes, 1978) [Sn—Mn: 2·739 (2), 2·722 (2), 2·758 (2) Å]. In the crystalline state $[SnCl\{Mn[(C_6H_5)_3P](CO)_4\}_3]$ has significantly different Sn—Mn and Mn—P distances which are probably brought about by intermolecular interactions. The bond angles at the central Sn atom agree with those in $[SnCl\{Mn(CO)_5\}_3]$ and $[SnBr\{Mn(CO)_5\}_3]$.

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Di-μ-chloro-dichlorobis(2,6-lutidine)diplatinum(II)* Dichloromethane Solvate

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Abstract. $[Pt_2(C_7H_9N)_2Cl_4].CH_2Cl_2$, $C_{14}H_{18}Cl_4N_2Pt_2.CH_2Cl_2$, $M_r = 831{\cdot}24$, monoclinic, $C2/c$, $a = 20{\cdot}010$ (16), $b = 7{\cdot}574$ (5), $c = 14{\cdot}631$ (7) Å, $\beta = 96{\cdot}77$ (5)°, $V = 2202$ (3) Å^3, $Z = 4$, $D_x = 2{\cdot}507$, $D_m = 2{\cdot}49$ (2) Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ(Mo $K\alpha$) = 14·07 mm^{-1} and T = 295 K. The structure was refined by full-matrix least-squares analysis to a conventional R factor of 0·036 and R_w = 0·027 for 1670 reflexions. The molecule is dimeric with two chlorine bridges and a *trans* configuration for the two 2,6-lutidine ligands as required by the crystallographic inversion centre. The coordination around Pt is square planar. The terminal Pt—Cl bonds [2·272 (3) Å] are slightly shorter than normal, while the bridged bonds are slightly longer [2·320 (3) and 2·321 (3) Å]. The Pt—N bond is 2·020 (10) Å and the 2,6-lutidine ligand is perpendicular to the Pt coordination plane (88·5°). The dimeric molecule crystallizes with a molecule of solvent (dichloromethane). The C atom in CH_2Cl_2 is located on a C_2 axis.

Introduction. In attempting to synthesize complexes of the type *cis*-[Pt(am)(am′)Cl_2] (where am and am′ are different amines) to increase the screening range for anti-tumour properties of Pt compounds, we have isolated a new series of complexes [Pt(dmf)LCl_2], where dmf = dimethylformamide and L = a pyridine derivative (Kong & Rochon, 1979). In order to determine the configuration of the compounds, we studied the crystal structure of [Pt(dmf)(2,6-lutidine)Cl_2] (Rochon, Kong & Melanson, 1980).

When [Pt(dmf)LCl_2] is dissolved in certain solvents, such as chloroform, a dimer $[Pt_2L_2Cl_4]$ is partially formed. [Pt(dmf)(2,6-lut)Cl_2] (2,6-lut = 2,6-lutidine) was synthesized as described previously (Kong & Rochon, 1979) and dissolved in dichloromethane. Six weeks later, red crystals which proved to be the dimer $[Pt_2(2,6\text{-lut})_2Cl_4]$ were obtained at room temperature. Their crystal structure is reported here.

A set of precession photographs indicated that hkl, $h + k = 2n + 1$ and $h0l$, $l = 2n + 1$ are systematic absences. These extinctions are found for space groups Cc and $C2/c$. The cell parameters were obtained by least-squares refinement from the setting angles of 15 automatically centred reflections on a Syntex $P\bar{1}$ diffractometer using graphite-monochromatized Mo $K\alpha$ radiation.

The intensity data were collected from a crystal with eight faces and approximate dimensions 0·16 × 0·17 × 0·44 mm elongated along c. 2178 independent reflections were measured in the range $2\theta < 52°$ by the $2\theta/\theta$ scan technique using Mo $K\alpha$ radiation on a Syntex $P\bar{1}$

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* 2,6-Lutidine = 2,6-dimethylpyridine.

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