The central C_8N_8 ring is approximately planar, but is not coplanar with the benzene rings.* Displacement of Nb from the central C_8N_8 plane may be compared with the values of the out-of-plane displacement for oxoacetato($\alpha,\beta,\gamma,\delta$-tetraphenylporphinato)niobium (Lecomte, Protas, Guilard, Fliniaux & Fournari, 1976) (1·0 Å) and tri-μ-oxo-bis[($\alpha,\beta,\gamma,\delta$-tetraphenylporphinato)niobium] (Johnson & Scheidt, 1978) (1·02, 1·00 Å).

Bond distances and angles are listed in Table 2. The Nb—Cl distances are longer than those for the non-bridging Nb—Cl bonds in $NbCl_5$ (Zalkin & Sands, 1958) (2·250, 2·302 Å), $NbOCl_3$ (Sands, Zalkin & Elson, 1959) (2·24 Å), $NbCl_5POCl_3$ (Brändén & Lindqvist, 1963) (2·25 ~ 2·35 Å) and $NbCl_4F$ (Preiss, 1968) (2·26 ~ 2·31 Å). This indicates an ionic component in the Nb—Cl bonds of $[NbCl_2(pc)]$.

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* See previous footnote.

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

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Abstract. $[SnBr\{Mn(C_{18}H_{15}P)(CO)_4\}_3]$, $C_{66}H_{45}BrMn_3O_{12}P_3Sn$, $M_r = 1486\cdot4$, triclinic, $P\bar{1}$, $a = 13\cdot515$ (3), $b = 22\cdot709$ (4), $c = 11\cdot004$ (3) Å, $\alpha = 86\cdot15$ (10), $\beta = 90\cdot05$ (10), $\gamma = 108\cdot33$ (10)°, $U = 3198$ Å^3, $Z = 2$, $D_c = 1\cdot544$ Mg m^{-3}, $F(000) = 1484$, Mo $K\alpha$ radiation, $\lambda = 0\cdot71069$ Å, μ(Mo $K\alpha$) = 1·6 mm^{-1}. Final $R = 0\cdot057$ for 8333 unique diffractometer data ($I > 3\sigma$) and 105 refined parameters. The three $Mn[(C_6H_5)_3P](CO)_4$ groups and the Br atom form a distorted tetrahedron round the Sn atom with Sn—Mn distances 2·727 (1), 2·718 (1), 2·708 (1) Å and Sn—Br 2·615 (1) Å.

Introduction. In the course of our investigations on the influence of packing forces in molecular crystals on the geometry of the molecules we have now prepared the compound $[SnBr\{Mn[(C_6H_5)_3P](CO)_4\}_3]$ and determined the crystal structure. The red crystals were prepared by reaction of $SnBr_2$ 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 0·38 × 0·46 × 0·69 mm. Cell parameters were determined by a least-squares procedure from the diffractometer angles of 15 reflexions measured with a Hilger & Watts Y 290 automatic four-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation and a scintillation counter. The intensities of 9500 reflexions ($I > 3\sigma$) with $2 \le \theta \le 24\cdot6°$ were measured by the $\omega/2\theta$ scan technique, with a scan width $\Delta 2\theta$ (°) = 1·34 + 0·34 tan θ from background to background and a scan speed of 0·02°

s⁻¹ in 2θ. Backgrounds were measured at either end of the scan range for 7s. Five standards were measured every fifty reflexions, and showed only random deviations from their mean intensities. Lp but not absorption corrections were applied, and after averaging the equivalent reflexions the data set contained 8333 independent reflexions.

The structure was solved by Patterson and Fourier methods and refined by full-matrix least squares with anisotropic temperature factors for Sn, Mn, Br 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°). The scattering factors were taken from Cromer & Mann (1968) and Cromer & Liberman (1970). Refinement converged with unit weights to $R = 0.057$.* The figures were drawn with *PLUTO*, written by Drs W. D. S. Motherwell and W. Clegg, and *POP*1 written by B. W. van de Waal.

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* Lists of structure factors, thermal parameters and a complete list of atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36539 (52 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
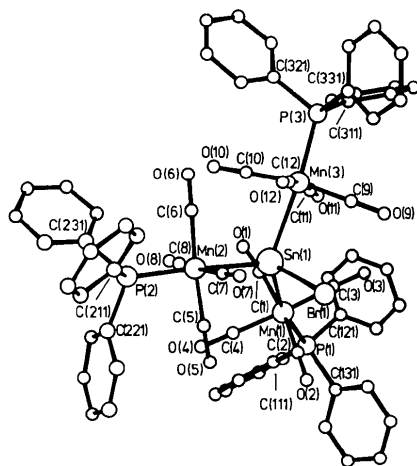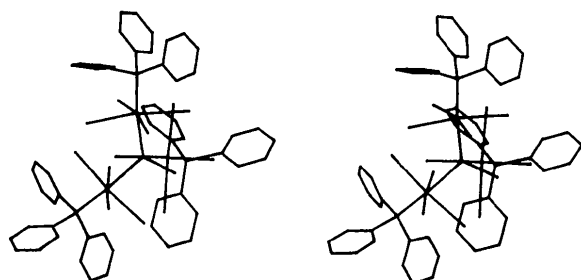

Fig. 1. General view of the molecule.

Fig. 2. Stereoscopic view of the molecule.

Table 1. *Positional parameters* ($\times 10^4$) *and U* ($\text{Å}^2 \times 10^4$)

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

	x	y	z	U
Sn	906·8 (4)	7595·0 (2)	2823·9 (4)	274 (2)
Br	2195·4 (6)	7433·1 (4)	4490·9 (7)	419 (4)
Mn(1)	1309·4 (8)	6940·4 (5)	1002·3 (9)	298 (5)
P(1)	2010 (1)	6559 (1)	−558 (2)	342 (9)
C(1)	723 (6)	7407 (4)	22 (7)	447 (18)
O(1)	278 (5)	7679 (3)	−560 (6)	701 (17)
C(2)	1665 (6)	6462 (3)	2214 (7)	402 (17)
O(2)	1873 (5)	6141 (3)	2962 (5)	589 (15)
C(3)	2572 (5)	7548 (3)	1041 (6)	384 (16)
O(3)	3371 (4)	7918 (2)	1066 (5)	507 (13)
C(4)	15 (6)	6372 (4)	1113 (7)	469 (19)
O(4)	9186 (5)	6018 (3)	1192 (6)	707 (18)
C(111)	1161 (4)	6097 (2)	−1685 (4)	393 (16)
C(113)	9587 (4)	5278 (2)	−2116 (4)	677 (26)
C(115)	815 (4)	5793 (2)	−3745 (4)	611 (23)
C(121)	2940 (4)	7172 (2)	−1545 (5)	460 (18)
C(123)	4619 (4)	7638 (2)	−2554 (5)	1019 (39)
C(125)	3284 (4)	8135 (2)	−2789 (5)	723 (27)
C(131)	2780 (4)	6059 (2)	−10 (5)	474 (19)
C(133)	4153 (4)	5929 (2)	1284 (5)	822 (31)
C(135)	3134 (4)	5078 (2)	149 (5)	895 (34)
Mn(2)	9032·7 (8)	7138·5 (5)	4018 (1)	328 (5)
P(2)	7397 (1)	6701 (1)	4827 (2)	363 (9)
C(5)	9264 (6)	6392 (3)	3958 (7)	422 (17)
O(5)	9429 (5)	5926 (3)	3914 (6)	623 (16)
C(6)	8929 (6)	7908 (4)	4139 (7)	442 (18)
O(6)	8844 (5)	8390 (3)	4277 (6)	667 (17)
C(7)	9710 (6)	7223 (4)	5472 (7)	469 (19)
O(7)	110 (5)	7271 (3)	6404 (6)	731 (18)
C(8)	8611 (6)	7108 (4)	2468 (7)	481 (19)
O(8)	8353 (5)	7092 (3)	1435 (6)	768 (19)
C(211)	7163 (5)	6763 (3)	6474 (4)	497 (19)
C(213)	7498 (5)	7400 (3)	8195 (4)	958 (37)
C(215)	6209 (5)	6381 (3)	8373 (4)	953 (36)
C(221)	6922 (5)	5866 (2)	4606 (5)	477 (19)
C(223)	7007 (5)	4833 (2)	5134 (5)	866 (33)
C(225)	6266 (5)	5054 (2)	3226 (5)	930 (35)
C(231)	6356 (4)	6977 (2)	4156 (2)	428 (17)
C(233)	4571 (4)	6979 (2)	4259 (5)	854 (32)
C(235)	5749 (4)	7621 (2)	2693 (5)	627 (24)
Mn(3)	1602·0 (8)	8850·3 (4)	2462·7 (9)	293 (5)
P(3)	2025 (1)	9906 (1)	2439 (2)	312 (8)
C(9)	2908 (6)	8838 (3)	2810 (7)	399 (17)
O(9)	3741 (5)	8832 (3)	2996 (5)	591 (15)
C(10)	286 (6)	8745 (3)	1938 (7)	422 (17)
O(10)	9465 (5)	8676 (3)	1545 (6)	631 (16)
C(11)	1969 (6)	8829 (3)	882 (7)	416 (17)
O(11)	2228 (5)	8817 (3)	−115 (6)	613 (16)
C(12)	1283 (6)	8763 (3)	4101 (7)	397 (17)
O(12)	1131 (5)	8716 (3)	5137 (5)	586 (15)
C(311)	2888 (4)	308 (2)	1152 (4)	410 (17)
C(313)	4614 (4)	862 (2)	338 (4)	883 (33)
C(315)	3169 (4)	535 (2)	−1021 (4)	681 (26)
C(321)	915 (3)	208 (2)	2369 (5)	383 (16)
C(323)	32 (3)	927 (2)	1607 (5)	719 (27)
C(325)	9245 (3)	154 (2)	3236 (5)	643 (24)
C(331)	2716 (4)	300 (2)	3763 (4)	382 (16)
C(333)	3269 (4)	1221 (2)	4890 (4)	597 (23)
C(335)	3859 (4)	344 (2)	5468 (4)	638 (24)

Table 2. *Bond distances* (Å) *and bond angles* (°)

Sn—Br	2·615 (1)	P(1)—C(121)	1·842 (6)
Sn—Mn(1)	2·727 (1)	P(1)—C(131)	1·840 (5)
Sn—Mn(2)	2·718 (1)	P(2)—C(211)	1·863 (5)
Sn—Mn(3)	2·708 (1)	P(2)—C(221)	1·834 (6)
Mn(1)—C(1)	1·813 (7)	P(2)—C(231)	1·846 (4)
Mn(1)—C(2)	1·822 (7)	P(3)—C(311)	1·832 (5)
Mn(1)—C(3)	1·829 (7)	P(3)—C(321)	1·834 (4)
Mn(1)—C(4)	1·817 (7)	P(3)—C(331)	1·855 (5)
Mn(1)—P(1)	2·304 (2)	C(1)—O(1)	1·155 (9)
Mn(2)—C(5)	1·824 (8)	C(2)—O(2)	1·157 (9)
Mn(2)—C(6)	1·809 (8)	C(3)—O(3)	1·143 (8)
Mn(2)—C(7)	1·834 (8)	C(4)—O(4)	1·154 (9)
Mn(2)—C(8)	1·796 (8)	C(5)—O(5)	1·15 (1)
Mn(2)—P(2)	2·274 (2)	C(6)—O(6)	1·16 (1)
Mn(3)—C(9)	1·814 (6)	C(7)—O(7)	1·16 (1)
Mn(3)—C(10)	1·818 (6)	C(8)—O(8)	1·19 (1)
Mn(3)—C(11)	1·818 (8)	C(9)—O(9)	1·149 (7)
Mn(3)—C(12)	1·837 (7)	C(10)—O(10)	1·157 (8)
Mn(3)—P(3)	2·281 (2)	C(11)—O(11)	1·16 (1)
P(1)—C(111)	1·837 (5)	C(12)—O(12)	1·15 (1)

Br—Sn—Mn(1)	101·63 (3)	C(8)—Mn(2)—P(2)	94·3 (2)
Br—Sn—Mn(2)	102·21 (3)	Sn—Mn(3)—C(9)	89·2 (2)
Br—Sn—Mn(3)	100·03 (3)	Sn—Mn(3)—C(10)	83·4 (3)
Mn(1)—Sn—Mn(2)	117·95 (4)	Sn—Mn(3)—C(11)	93·0 (3)
Mn(1)—Sn—Mn(3)	116·73 (3)	Sn—Mn(3)—C(12)	79·7 (2)
Mn(2)—Sn—Mn(3)	114·09 (3)	Sn—Mn(3)—P(3)	170·49 (6)
Sn—Mn(1)—C(1)	85·0 (3)	C(9)—Mn(3)—C(10)	169·3 (3)
Sn—Mn(1)—C(2)	85·7 (2)	C(9)—Mn(3)—C(11)	85·7 (3)
Sn—Mn(1)—C(3)	80·4 (2)	C(9)—Mn(3)—C(12)	89·5 (3)
Sn—Mn(1)—C(4)	93·3 (3)	C(9)—Mn(3)—P(3)	94·6 (3)
Sn—Mn(1)—P(1)	167·05 (6)	C(10)—Mn(3)—C(11)	87·0 (3)
C(1)—Mn(1)—C(2)	167·5 (3)	C(10)—Mn(3)—C(12)	96·7 (3)
C(1)—Mn(1)—C(3)	94·4 (3)	C(10)—Mn(3)—P(3)	93·9 (3)
C(1)—Mn(1)—C(4)	85·6 (3)	C(11)—Mn(3)—C(12)	171·3 (3)
C(1)—Mn(1)—P(1)	95·6 (2)	C(11)—Mn(3)—P(3)	96·0 (3)
C(2)—Mn(1)—C(3)	92·2 (3)	C(12)—Mn(3)—P(3)	91·6 (2)
C(2)—Mn(1)—C(4)	86·6 (3)	Mn(1)—P(1)—C(111)	120·4 (1)
C(2)—Mn(1)—P(1)	95·4 (2)	Mn(1)—P(1)—C(121)	113·4 (2)
C(3)—Mn(1)—C(4)	173·6 (3)	Mn(1)—P(1)—C(131)	112·9 (2)
C(3)—Mn(1)—P(1)	86·7 (2)	C(111)—P(1)—C(121)	101·6 (2)
C(4)—Mn(1)—P(1)	99·7 (3)	C(111)—P(1)—C(131)	103·0 (2)
Sn—Mn(2)—C(5)	84·1 (2)	C(121)—P(1)—C(131)	103·6 (2)
Sn—Mn(2)—C(6)	92·4 (2)	Mn(2)—P(2)—C(211)	119·1 (2)
Sn—Mn(2)—C(7)	89·4 (2)	Mn(2)—P(2)—C(221)	110·7 (2)
Sn—Mn(2)—C(8)	79·9 (2)	Mn(2)—P(2)—C(231)	117·1 (2)
Sn—Mn(2)—P(2)	173·62 (7)	C(211)—P(2)—C(221)	105·2 (3)
C(5)—Mn(2)—C(6)	174·4 (3)	C(211)—P(2)—C(231)	100·2 (2)
C(5)—Mn(2)—C(7)	87·5 (3)	C(221)—P(2)—C(231)	102·6 (3)
C(5)—Mn(2)—C(8)	90·9 (4)	Mn(3)—P(3)—C(311)	112·8 (2)
C(5)—Mn(2)—P(2)	93·4 (2)	Mn(3)—P(3)—C(321)	115·2 (2)
C(6)—Mn(2)—C(7)	88·0 (4)	Mn(3)—P(3)—C(331)	118·3 (2)
C(6)—Mn(2)—C(8)	92·8 (4)	C(311)—P(3)—C(321)	105·6 (2)
C(6)—Mn(2)—P(2)	90·6 (2)	C(311)—P(3)—C(331)	102·8 (2)
C(7)—Mn(2)—C(8)	169·3 (3)	C(321)—P(3)—C(331)	100·4 (2)
C(7)—Mn(2)—P(2)	96·4 (2)		

Discussion. The molecular structure of the title compound is shown in Fig. 1; a stereoview of the molecule is given in Fig. 2. The positional parameters and the equivalent values of the anisotropic temperature factors U_{ij} [$U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33} + 2U_{23} \cos \alpha + 2U_{13} \times \cos \beta + 2U_{12} \cos \gamma)$] 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.

With the exception of the distance Sn—Mn(1) and a few angles which deviate a little more than 3σ, distances and angles in [SnBr{Mn(C$_{18}$H$_{15}$P)(CO)$_4$}$_3$] predominantly agree within 3σ with those in [SnCl{Mn-C$_{18}$H$_{15}$P)(CO)$_4$}$_3$] (Preut & Haupt, 1981). The change from Cl to Br brings about only slight changes in the molecule. The crystal structures are isomorphous. As in [SnCl{Mn(C$_{18}$H$_{15}$P)(CO)$_4$}$_3$] the Sn—Mn distances in the title compound are significantly different and 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].

Intermolecular distances do not indicate interactions exceeding van der Waals forces.

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Tribromotris(4-methylpyridine)molybdenum(III)

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Abstract. [MoBr$_3$(C$_6$H$_7$N)$_3$], C$_{18}$H$_{21}$Br$_3$MoN$_3$, triclinic, P$\bar{1}$, a = 9·193 (1), b = 16·379 (2), c = 17·319 (3) Å, α = 61·82 (2), β = 80·08 (2), γ = 75·93 (2)°, Z = 4, D_o = 1·81 (3) (by flotation in dibromoethane/carbon tetrachloride), D_c = 1·836 Mg m^{-3}; R = 0·079 for 3408 reflections [$I > 5\sigma(I)$]. The