

distances found for the cations are comparable to those found for *trans*-Cl and *trans*-N atoms in other complexes of Rh^{III} (Siripaisarnpipat & Schlemper, 1984; Muir *et al.*, 1987; Acharya *et al.*, 1984). The average C—N, N—N and C—C distances and bond angles for the two cations are different but are within the ranges found in other nitrogen heterocyclic compounds (Carmona, Lahoz, Oro, Pilar Lamata & Buzarra, 1991).

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Structure of [SbCl{Mn(CO)₂(η^5 -C₅H₅)}{Co(CO)₃(PPh₃)₂}]₂·C₆H₅Cl

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Abstract. Octacarbonyl-1 κ^2 C,2 κ^3 C,3 κ^3 C-chloro-4 κ Cl-[1(η^5 -cyclopentadienyl)]bis(triphenylphosphine)-2 κ P,3 κ P-antimonydicobaltmanganese(2Co—Sb, Mn—Sb) chlorobenzene solvate, [Co₂MnSbCl(C₅H₅)(C₁₈H₁₅P)₂(CO)₈].C₆H₅Cl, $M_r = 1256.32$, monoclinic, $P2_1/c$, $a = 16.632$ (4), $b = 16.906$ (3), $c = 19.332$ (3) Å, $\beta = 93.15$ (2)°, $V = 5428$ (2) Å³, $Z = 4$, $D_x = 1.54$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 15.21$ cm⁻¹, $F(000) = 2512$, $T = 298$ K, $R = 0.062$ for 2821 unique observed reflections. The title complex contains an Sb atom in a slightly distorted tetrahedral environment, coordinated to two Co atoms [Sb—Co 2.569 (3) and 2.574 (3) Å], one Mn atom [Sb—Mn 2.460 (4) Å] and one Cl atom [Sb—Cl 2.439 (6) Å].

Experimental. The title complex was obtained in low yield from a reaction between Mn(CO)₂(thf)(η -C₅H₅) and the crude product of a reaction between SbCl₃ and two equivalents of K[Co(CO)₃(PPh₃)] in thf

solution. Infrared data: $\nu(\text{CO})$ (thf solution) 2046w, 2025m, 1986s, 1965m, 1921m and 1871m cm⁻¹. Analysis calculated for C₄₉H₃₅ClCo₂MnO₈P₂Sb: C 51.5; H 3.1%. Analysis found: C 51.9; H 3.1%. Dark purple crystals were obtained from the crude reaction mixture by solvent diffusion from chlorobenzene/hexane. A crystal of dimensions ca 0.4 × 0.5 × 0.4 mm (systematic absences: $k = 2n + 1$ in $0k0$; $l = 2n + 1$ in $h0l$) was used for data collection on an Enraf-Nonius CAD-4F diffractometer with graphite monochromator and in the $\theta/2\theta$ -scan mode. Cell parameters were refined by least-squares methods from setting angles of 22 independent reflections ($\theta \approx 10^\circ$). Intensities were measured to $\theta = 22.5^\circ$ over hkl range 0 to 17, 0 to 18, -20 to 20. Standard reflections $4\bar{2}8$, $91\bar{1}$ and $7\bar{1}\bar{1}$ were measured every 2 h and showed 30% decay over 118.2 h of data collection. 7687 data were measured, of which 7097 were independent. 2821 data with $I > 2.0\sigma(I)$ were considered observed and used in structure determination and refinement. R_{int} before absorption correction was 0.035. Data were corrected for decomposition,

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Table 1. Fractional coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

For aryl C atoms *U* is the isotropic displacement parameter, and for other atoms $U = (1/3)\sum_{i=1}^3 U_{ij} a_i^* a_j^* a_i \cdot a_j$.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i>
Sb	0.24275 (7)	0.02145 (8)	0.12548 (7)	0.049
Co(1)	0.37547 (14)	0.05000 (15)	0.19528 (13)	0.049
Co(2)	0.12330 (14)	0.05862 (15)	0.19819 (13)	0.047
Mn	0.23083 (19)	0.05930 (22)	0.00248 (17)	0.083
P(1)	0.4895 (3)	0.0859 (3)	0.2503 (3)	0.051
P(2)	0.0210 (3)	0.0991 (3)	0.2574 (3)	0.053
Cl(1)	0.2390 (4)	-0.1225 (3)	0.1334 (4)	0.098
Cl(2S)	0.6038 (8)	0.0472 (6)	0.5399 (5)	0.221
O(1)	0.3035 (8)	0.2000 (9)	0.2314 (9)	0.089
O(2)	0.4600 (9)	0.0157 (13)	0.0716 (8)	0.127
O(3)	0.3581 (11)	-0.0891 (10)	0.2809 (10)	0.106
O(4)	0.2084 (10)	-0.0003 (12)	0.3229 (8)	0.121
O(5)	0.1461 (8)	0.2184 (8)	0.1458 (8)	0.071
O(6)	0.0261 (10)	-0.0541 (11)	0.1173 (11)	0.142
O(7)	0.3015 (11)	0.2104 (10)	0.0428 (10)	0.109
O(8)	0.0662 (10)	0.1153 (11)	0.0137 (8)	0.117
C(1)	0.3284 (11)	0.1398 (13)	0.2153 (10)	0.053
C(2)	0.4256 (12)	0.0292 (15)	0.1179 (13)	0.086
C(3)	0.3633 (13)	-0.0364 (14)	0.2494 (12)	0.076
C(4)	0.1752 (11)	0.0245 (14)	0.2739 (12)	0.067
C(5)	0.1393 (10)	0.1532 (15)	0.1660 (10)	0.059
C(6)	0.0650 (12)	-0.0082 (15)	0.1453 (12)	0.088
C(7)	0.2746 (13)	0.1558 (15)	0.0312 (12)	0.071
C(8)	0.1332 (14)	0.0922 (16)	0.0104 (12)	0.092
C(11)	0.3250 (16)	0.0218 (24)	-0.0580 (14)	0.155
C(12)	0.2643 (15)	0.0572 (13)	-0.1004 (12)	0.126
C(13)	0.1938 (13)	0.0129 (26)	-0.0952 (17)	0.147
C(14)	0.2109 (17)	-0.0499 (16)	-0.0495 (8)	0.141
C(15)	0.2920 (11)	-0.0444 (22)	-0.0265 (17)	0.178
C(111)	0.5459 (7)	0.0028 (11)	0.2871 (13)	0.058 (6)
C(112)	0.5632 (13)	-0.0052 (7)	0.3582 (11)	0.070 (6)
C(113)	0.6019 (10)	-0.0730 (11)	0.3840 (5)	0.092 (8)
C(114)	0.6234 (7)	-0.1328 (9)	0.3388 (11)	0.077 (7)
C(115)	0.6061 (12)	-0.1248 (8)	0.2678 (9)	0.075 (6)
C(116)	0.5674 (9)	-0.0570 (13)	0.2419 (7)	0.065 (6)
C(121)	0.5595 (6)	0.1355 (6)	0.1982 (5)	0.052 (5)
C(122)	0.5287 (6)	0.1901 (12)	0.1496 (10)	0.066 (6)
C(123)	0.5810 (6)	0.2361 (11)	0.1124 (10)	0.108 (8)
C(124)	0.6640 (6)	0.2276 (6)	0.1238 (5)	0.087 (7)
C(125)	0.6947 (6)	0.1730 (12)	0.1725 (10)	0.084 (7)
C(126)	0.6425 (6)	0.1270 (11)	0.2096 (10)	0.070 (6)
C(131)	0.4785 (16)	0.1546 (10)	0.3221 (11)	0.057 (5)
C(132)	0.5264 (10)	0.2219 (6)	0.3309 (9)	0.081 (7)
C(133)	0.5143 (10)	0.2735 (10)	0.3856 (6)	0.103 (8)
C(134)	0.4543 (13)	0.2579 (9)	0.4315 (9)	0.118 (9)
C(135)	0.4065 (7)	0.1906 (7)	0.4227 (8)	0.103 (8)
C(136)	0.4186 (13)	0.1389 (11)	0.3680 (7)	0.083 (7)
C(211)	-0.0382 (6)	0.1789 (11)	0.2080 (12)	0.054 (5)
C(212)	-0.0742 (10)	0.2368 (12)	0.2573 (6)	0.065 (6)
C(213)	-0.1212 (11)	0.2954 (6)	0.2245 (10)	0.070 (6)
C(214)	-0.1322 (6)	0.2962 (10)	0.1525 (10)	0.078 (6)
C(215)	-0.0961 (10)	0.2383 (10)	0.1132 (5)	0.075 (6)
C(216)	-0.0492 (11)	0.1796 (5)	0.1459 (11)	0.057 (5)
C(221)	0.0541 (16)	0.1366 (17)	0.3428 (6)	0.062 (6)
C(222)	0.1075 (10)	0.2002 (12)	0.3455 (7)	0.101 (8)
C(223)	0.1398 (10)	0.2272 (10)	0.4092 (7)	0.15 (1)
C(224)	0.1188 (13)	0.1905 (14)	0.4703 (6)	0.13 (1)
C(225)	0.0654 (8)	0.1268 (10)	0.4676 (7)	0.13 (1)
C(226)	0.0331 (13)	0.0999 (13)	0.4039 (7)	0.094 (7)
C(231)	-0.0518 (9)	0.0246 (9)	0.2760 (4)	0.054 (5)
C(232)	-0.0239 (7)	-0.0514 (12)	0.2918 (11)	0.085 (7)
C(233)	-0.0779 (9)	-0.1103 (10)	0.3091 (10)	0.092 (7)
C(234)	-0.1598 (8)	-0.0933 (8)	0.3106 (4)	0.087 (7)
C(235)	-0.1876 (7)	-0.0173 (11)	0.2949 (10)	0.087 (7)
C(236)	-0.1337 (10)	0.0416 (9)	0.2775 (10)	0.077 (7)
C(411S)	0.6843 (9)	0.0787 (13)	0.5019 (10)	0.117 (9)
C(412S)	0.7590 (11)	0.0419 (11)	0.5117 (11)	0.14 (1)
C(413S)	0.8267 (11)	0.0748 (16)	0.4834 (15)	0.19 (2)
C(414S)	0.8197 (8)	0.1447 (11)	0.4452 (9)	0.14 (1)
C(415S)	0.7450 (11)	0.1815 (13)	0.4354 (13)	0.16 (1)
C(416S)	0.6773 (11)	0.1486 (17)	0.4637 (16)	0.13 (1)

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

Sb—Co(1)	2.569 (3)	Sb—Co(2)	2.574 (3)
Sb—Mn	2.460 (4)	Sb—Cl(1)	2.439 (6)
Co(1)—P(1)	2.209 (6)	Co(1)—C(1)	1.76 (3)
Co(1)—C(2)	1.79 (3)	Co(1)—C(3)	1.81 (3)
Co(2)—P(2)	2.211 (6)	Co(2)—C(4)	1.75 (3)
Co(2)—C(5)	1.74 (3)	Co(2)—C(6)	1.78 (3)
Mn—C(7)	1.86 (3)	Mn—C(8)	1.73 (3)
Mn—C(11)	2.10 (3)	Mn—C(12)	2.09 (3)
Mn—C(13)	2.10 (4)	Mn—C(14)	2.12 (3)
Mn—C(15)	2.12 (4)		
Co(1)—Sb—Co(2)	109.5 (1)	Co(1)—Sb—Mn	118.7 (2)
Co(1)—Sb—Cl(1)	100.3 (2)	Co(2)—Sb—Mn	116.2 (2)
Co(2)—Sb—Cl(1)	100.7 (2)	Mn—Sb—Cl(1)	108.6 (2)
Sb—Co(1)—P(1)	174.4 (2)	Sb—Co(1)—C(1)	84.1 (7)
Sb—Co(1)—C(2)	87.2 (7)	Sb—Co(1)—C(3)	91.8 (8)
P(1)—Co(1)—C(1)	92.1 (7)	P(1)—Co(1)—C(2)	91.7 (8)
P(1)—Co(1)—C(3)	93.6 (8)	C(1)—Co(1)—C(2)	126.0 (11)
C(1)—Co(1)—C(3)	120.1 (10)	C(2)—Co(1)—C(3)	113.4 (11)
Sb—Co(2)—P(2)	175.9 (2)	Sb—Co(2)—C(4)	90.9 (7)
Sb—Co(2)—C(5)	83.8 (7)	Sb—Co(2)—C(6)	86.8 (7)
P(2)—Co(2)—C(4)	91.6 (7)	P(2)—Co(2)—C(5)	92.2 (7)
P(2)—Co(2)—C(6)	94.8 (8)	C(4)—Co(2)—C(5)	121.5 (10)
C(4)—Co(2)—C(6)	120.0 (11)	C(5)—Co(2)—C(6)	117.8 (11)
Sb—Mn—C(7)	86.0 (8)	Sb—Mn—C(8)	91.4 (8)
Sb—Mn—C(11)	115.8 (9)	Sb—Mn—C(12)	154.4 (7)
Sb—Mn—C(13)	140.7 (12)	Sb—Mn—C(14)	103.5 (6)
Sb—Mn—C(15)	91.5 (9)		

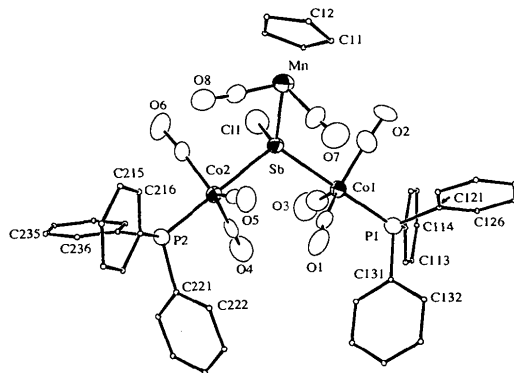


Fig. 1. Molecular structure and atomic labelling scheme for [SbCl{Mn(CO)₂(η^5 -C₅H₅)}]{Co(CO)₃(PPh₃)₂}. Thermal ellipsoids are shown at the 30% probability level, and aryl and cyclopentadienyl C atoms are shown as spheres of arbitrary radii.

refinement on *F* included anisotropic thermal parameters for all non-H atoms except aryl C atoms. H atoms were included at calculated positions (C—H = 1.0 Å) with fixed contributions to the structure factors. The phenyl and cyclopentadienyl groups were refined as rigid groups with idealized geometry (Ph C—C 1.395 Å, Cp C—C 1.40 Å). A solvation molecule of chlorobenzene was found with full occupancy. $\sum w(|F_o| - |F_c|)^2$ was minimized with $w = [\sigma^2(F_o)]^{-1}$. Maximum $\Delta/\sigma = 0.07$, average $\Delta/\sigma = 0.004$; maximum $\Delta\rho = 0.74$, minimum $\Delta\rho = -0.56 \text{ e } \text{Å}^{-3}$; final $R = 0.062$, $wR = 0.073$, using 337 parameters; $S = 2.72$. Atomic scattering factors were obtained from *International Tables for X-ray Crystallography* (1974, Vol. IV). Calculations were carried out on a MicroVAX 3600 computer using the Glasgow *GX* suite of programs (Mallinson & Muir,

Lp and absorption [DIFABS (Walker & Stuart, 1983), maximum and minimum values of applied absorption correction 1.18 and 0.70]. The structure was solved by direct methods (MITHRIL; Gilmore, 1984), and subsequent full-matrix least-squares

1985). Final positional parameters are given in Table 1, with selected bond distances and angles in Table 2.* The atomic labelling scheme and molecular structure are shown in Fig. 1.

Related literature. For other Co/Sb complexes see Clegg, Compton, Errington, Hockless, Norman, Ramshaw & Webster (1990), and Norman, Webster & Farrugia (1992). Other related complexes containing Sb—Mn bonds include [SbCl{Mn(CO)₂(η^5 -C₅H₄Me)}{Mn(CO)₅}₂] (Lombard, Huttner & Zsolnai, 1988), [SbCl{Mn(CO)₂(η^5 -C₅H₅)}{Mn₂(CO)₄(η^5 -C₅H₅)₂}] (von Seyerl, Wohlfahrt & Huttner, 1980), [SbBr{Mn(CO)₂(η^5 -C₅H₄Me)}{Fe(CO)₂(η^5 -C₅H₅)₂}] and [SbBr{Mn(CO)₂(η^5 -C₅H₄Me)}{Fe₂(CO)₄(η^5 -C₅H₅)₂}] (Zsolnai, 1984). For other monohalo metallastibane or stibate(1-) species see Ferrer, Rossell, Seco & Braunstein (1989),

* Lists of structure factors, anisotropic thermal parameters, H-atom positional parameters and complete bond lengths and bond angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55401 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI1004]

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Structure of 4-Piperidone Derivatives. III. 1,3,3-Trimethyl-2,6-diphenyl-4-piperidone and 2,6-Bis(*p*-chlorophenyl)-1,3,5-trimethyl-4-piperidone

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Abstract. C₂₀H₂₃NO (I), *M_r* = 293.4, triclinic, *P* $\bar{1}$, *a* = 6.486 (2), *b* = 10.921 (2), *c* = 12.542 (1) Å, α = 79.90 (1), β = 88.87 (2), γ = 73.50 (2)°, *V* = 838.2 (3) Å³, *Z* = 2, *D_x* = 1.16 g cm⁻³, $\lambda(\text{Cu } K\alpha)$ = 1.5418 Å, μ = 5.15 cm⁻¹, *F*(000) = 316, *T* = 295 K, *R* = 0.051, *wR* = 0.070 for 2641 unique observed reflections [*I* > 3 σ (*I*)]. C₂₀H₂₁Cl₂NO (II), *M_r* = 362.3,

Trinh-Toan & Dahl (1971), Panster & Malisch (1976) and Malisch & Panster (1975, 1976).

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orthorhombic, *Pnma*, *a* = 7.420 (2), *b* = 23.118 (1), *c* = 10.624 (2) Å, *V* = 1822.4 (2) Å³, *Z* = 4, *D_x* = 1.32 g cm⁻³, $\lambda(\text{Cu } K\alpha)$ = 1.5418 Å, μ = 32.95 cm⁻¹, *F*(000) = 760, *T* = 295 K, *R* = 0.042, *wR* = 0.048 for 1221 unique observed reflections [*I* > 3 σ (*I*)]. The 4-piperidone ring has a slightly distorted chair conformation in both compounds and the mean torsion angles are 54.0 (2) and 52.7 (3)° for (I) and (II), respectively. In (I) the puckering is enhanced in the area of N1 and in (II) it is enhanced in the area of

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