1201. *Chemistry of the Metal Carbonyls. Part XXXIII.*[1] *Synthesis of Tetracarbonyl-σ-tetrafluoroethylcobalt, and Related Reactions*

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Tetracarbonylhydridocobalt adds to tetrafluoroethylene to afford $(HCF_2{\cdot}CF_2)Co(CO)_4$, which with triphenylphosphine yields *trans*-$(HCF_2{\cdot}CF_2)Co(CO)_3(PPh_3)$. Similarly, $(CH_3{\cdot}CF_2{\cdot}CF_2)Mn(CO)_5$ and triphenylphosphine gives *trans*-$(CH_3{\cdot}CF_2{\cdot}CF_2)Mn(CO)_4(PPh_3)$ which can also be prepared from *cis*-$CH_3Mn(CO)_4(PPh_3)$ and tetrafluoroethylene. The compound $(C_6F_5)Co(CO)_4$ has been characterised.

Tetracarbonylhydridocobalt reduces chlorotrifluoroethylene and hexafluorobut-2-yne to 1-chloro-1,2,2-trifluoroethane and *trans*-hexafluorobut-2-ene, respectively. Several reductions of fluoro- and chloro-ethylenes by pentacarbonylhydridomanganese are described, and further studies on the reaction between the hydride and 1,1-dichloro-2,2-difluoroethylene are reported.

ADDITION of tetracarbonylhydridocobalt to olefins has been well studied [2] as the first step of the oxo reacton. Herein is reported addition of the hydride to tetrafluoroethylene, a reaction of the type observed previously with several other metal carbonyl hydrides.[3] Tetracarbonyl-σ-tetrafluoroethylcobalt, $(HCF_2{\cdot}CF_2)Co(CO)_4$, is a volatile orange-yellow liquid, and can be isolated from the reaction between $HCo(CO)_4$ and $CF_2{:}CF_2$ in pentane at —20°. Addition also occurs in the absence of pentane, and under all conditions an approximately equivalent amount of 1,1,2,2-tetrafluoroethane is obtained. Tetracarbonylhydridocobalt does not add to tetrafluoroethylene in aqueous solution at 0°, in contrast to the behaviour of the hydridopentacyanocobaltate(III) ion.[4] This difference may be due

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[1] Part XXXII, P. W. Jolly, F. G. A. Stone, and K. McKenzie, *J.*, 1965, **6416**.

[2] R. F. Heck and D. S. Breslow, *J. Amer. Chem. Soc.*, 1961, **83**, 4023, and references cited therein.

[3] (*a*) P. M. Treichel and F. G. A. Stone, *Adv. Organometallic Chem.*, 1964, **1**, 143; (*b*) J. B. Wilford and F. G. A. Stone, *Inorg. Chem.*, 1965, **4**, 93.

[4] M. J. Mays and G. Wilkinson, *Nature*, 1964, **203**, 1167.

to the virtually complete dissociation of $HCo(CO)_4$ in water,[5] so that a four-centre addition to the double bond is not possible.

The infrared spectrum of $(HCF_2{\cdot}CF_2)Co(CO)_4$ in the 7—16 μ region, as well as the proton and fluorine-19 n.m.r. spectra, is typical of a $(HCF_2{\cdot}CF_2)$Metal group, confirming the expected structure.[3b] If a trigonal bipyramidal molecule $LM(CO)_4$ has C_{3v} symmetry, three infrared-active carbonyl stretching frequencies are predicted, and this behaviour might be expected for $(HCF_2{\cdot}CF_2)Co(CO)_4$. However, the complex shows four terminal carbonyl stretching frequencies. A similar effect is observed in the spectrum of $(HCF_2{\cdot}CF_2)Mn(CO)_5$ which shows five bands, rather than the three expected for C_{4v} symmetry. The appearance of additional bands in these complexes may be ascribed to a lowering of the overall molecular symmetry through the presence of a non-axially symmetric fluorocarbon–metal group.[6] We have also synthesised tetracarbonyl-σ-perfluorophenylcobalt. This complex exhibits higher symmetry than $(HCF_2{\cdot}CF_2)Co(CO)_4$, showing three infrared-active carbonyl stretches in its spectrum. Interestingly, similar behaviour is shown by manganese; the complex $(C_6F_5)Mn(CO)_5$ has three carbonyl stretching frequencies in its spectrum and is therefore of higher symmetry than $(HCF_2{\cdot}CF_2)Mn(CO)_5$.

Tetracarbonyl-σ-perfluorophenylcobalt was prepared by treating perfluorobenzoyl chloride with tetracarbonylcobaltate(—I) anion. The intermediate perfluorobenzoyl cobalt complex spontaneously decarbonylates during this reaction, being similar in this respect to the tetracarbonyl-σ-perfluoroacylcobalt complexes.[7] The reaction between $[Co(CO)_4]^-$ and $C_6F_5{\cdot}COCl$ also affords $(C_6F_5)_2CO$. The latter is the major product if impure octacarbonyldicobalt is used to prepare the anion $[Co(CO)_4]^-$. Tetracarbonyl-σ-perfluorophenylcobalt, a low-melting solid, is quite stable in air when pure.

Treatment of $(HCF_2{\cdot}CF_2)Co(CO)_4$ with triphenylphosphine affords the compound $(HCF_2{\cdot}CF_2)Co(CO)_3(PPh_3)$. The infrared spectrum of the latter in the carbonyl region shows one intense E absorption, which is broad, and one weak A_1 absorption at higher frequency (Table 1). This is the pattern expected for C_{3v} symmetry corresponding to the formation of $(HCF_2{\cdot}CF_2)Co(CO)_3(PPh_3)$ as the *trans*-isomer. The *cis*-isomer (C_s) would be expected to have a spectrum with three carbonyl absorptions of comparable intensity. We have also observed that $(CH_3{\cdot}CF_2{\cdot}CF_2)Mn(CO)_5$ reacts with triphenylphosphine to give *trans*-$(CH_3{\cdot}CF_2{\cdot}CF_2)Mn(CO)_4(PPh_3)$, and that this same complex may be prepared by adding $CH_3Mn(CO)_4(PPh_3)$ to tetrafluoroethylene, using ultraviolet irradiation. The spectrum of *trans*-$(CH_3{\cdot}CF_2{\cdot}CF_2)Mn(CO)_4(PPh_3)$ in the carbonyl region shows as expected one weak A_1 absorption and a strong E absorption at lower frequency. The *cis*-isomer would

TABLE 1

Infrared spectra of some fluorocarbon–metal compounds

Compound	Fundamental C≡O stretching bands * (cm.$^{-1}$)	Other bands † (cm.$^{-1}$)
$(C_6F_5)Co(CO)_4$	2122w, 2060s, 2040vs	1069m, 1061w, 968s, 765m
$(HCF_2{\cdot}CF_2)Co(CO)_4$	2126m, 2060s, 2052vs, 2048vs	2950m, 1360s, 1180m, 1115vs, 1090vs, 1030vs, 935m, 785m, 650s
$(HCF_2{\cdot}CF_2)Co(CO)_3(PPh_3)$	2066m, 1997vs, b	3065w, 3020w, 2960w, 1480m, 1470m, 1440m, 1362s, 1310w, 1192w, 1180m, 1105s, d, 1085m, 1035sh, 1020vs, 935m, 790w, 850w, 750s, 712s, 695s, 645m
$(HCCl_2{\cdot}CF_2)Mn(CO)_5$	2131w, 2072w, 2043vs, 2037vs, 2013s	2950w, 2905w, 1235m, 1215w, 1095w, 1085m, 975vs, 965sh, 922w, 812s, 790m, 680w, 645s
$(CH_3{\cdot}CF_2{\cdot}CF_2)Mn(CO)_4(PPh_3)$	2075w, 1990vs	3060w, 3012w, 3005w, 2950w, 1380m, 1330w, 1270m, 1200w, 1160s, 1090m, 1025s, 1000w, 980s, 970s, 940w, 840s, 765m, 695s, 670vs, 640s

* Cyclohexane solution, grating spectrophotometer. † Carbon disulphide solution.

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[5] H. W. Sternberg, I. Wender, R. A. Friedel, and M. Orchin, *J. Amer. Chem. Soc.*, 1953, **75**, 2717.

[6] J. B. Wilford and F. G. A. Stone, *Inorg. Chem.*, 1965, **4**, 389.

[7] W. R. McClellan, *J. Amer. Chem. Soc.*, 1961, **83**, 1598.

be predicted to show four carbonyl absorptions. Interestingly, *cis*-$CH_3Mn(CO)_4(PPh_3)$ also reacts with carbon monoxide to give the *trans*-isomer of $(CH_3{\cdot}CO)Mn(CO)_4(PPh_3)$.[8]

It was mentioned above that 1,1,2,2-tetrafluoroethane is formed in the reaction between tetracarbonylhydridocobalt and tetrafluoroethylene. Reduction also occurs when chlorotrifluoroethylene is treated with $HCo(CO)_4$. In this reaction, 1-chloro-1,2,2-trifluoroethane is produced, but no organo-cobalt complex could be isolated. Similarly, hexafluorobut-2-yne is reduced by tetracarbonylhydridocobalt to *trans*-hexafluorobut-2-ene. The previously reported [9] acetylene complex $(CF_3{\cdot}C{\vdots}C{\cdot}CF_3)Co_2(CO)_6$ is also formed. The latter is undoubtedly produced by reaction between the butyne and some octacarbonyldicobalt formed by decomposition of the hydride.[5]

The observed formation of reduction products of tetrafluoro- and chlorotrifluoroethylene, and hexafluorobut-2-yne, prompted a further study of some reactions of pentacarbonylhydridomanganese previously subjected to only cursory examination.[10] It was reported that several fluoroethylenes (*e.g.*, $CF_2{:}CH_2$) did not react with $HMn(CO)_5$ at room temperature. However, we have now observed that at elevated temperatures all olefins of type $C_2F_nH_{4-n}$ $(0 \leqslant n < 4)$ afford fluoroethanes on treatment with $HMn(CO)_5$. Interestingly, reduction of ethylene with the hydride has been previously mentioned, but no details have been given.[11a] Some molecular hydrogen was also produced in the reactions studied, presumably by concomitant pyrolysis of the manganese hydride. However, control experiments in which decacarbonyldimanganese and hydrogen were heated with $CF_2{:}CH_2$ yielded no reduced gas. Moreover, the hydride is known to be a reducing agent.[11b]

In view of recent results [12] obtained with chlorotrifluoroethylene and pentacarbonyl hydridomanganese, the reaction between this hydride and 1,1-dichloro-2,2-difluoroethylene, previously reported to give only $(HCCl_2{\cdot}CF_2)Mn(CO)_5$,[10] was reinvestigated. The products depend both on the reaction time and the amount of olefin taken for reaction. With short reaction times (4 hr. or less) and between room temperature and 40°, addition took place to give a mixture of the two isomers $(HCCl_2{\cdot}CF_2)Mn(CO)_5$ and $(HCF_2{\cdot}CCl_2)Mn(CO)_5$. Reduction of the olefin also occurred, because 1,1-dichloro-2,2-difluoroethane was isolated and decacarbonyldimanganese was invariably produced. With reaction times of several days, or with a large excess of the olefin, only a small yield $(<10\%)$ of the isomer $(HCCl_2{\cdot}CF_2)Mn(CO)_5$ was obtained, together with $Mn(CO)_5Cl$ and $Mn_2(CO)_{10}$.

The compound $(HCF_2{\cdot}CCl_2)Mn(CO)_5$ proved to be very unstable in solution, and could not be isolated analytically pure. However, the infrared and n.m.r. spectral evidence for its presence in the reaction products was conclusive. After several hours at 40° a mixture of $(HCCl_2{\cdot}CF_2)Mn(CO)_5$ and $(HCF_2{\cdot}CCl_2)Mn(CO)_5$ affords 1-chloro-2,2-difluoroethylene and pentacarbonylchloromanganese. Thus, $(HCCl_2{\cdot}CF_2)Mn(CO)_5$ is more stable than $(HCF_2{\cdot}CCl_2)Mn(CO)_5$, and both isomers decompose by chlorine-atom migration.

Contrasting with the reaction between $CF_2{:}CCl_2$ and $HMn(CO)_5$, which occurs readily at room temperature, the hydride does not react with $CFCl{:}CFCl$ under similar conditions. At 60° the major products are decacarbonyldimanganese and 1,2-dichloro-1,2-difluoroethane. However, traces of $Mn(CO)_5Cl$ and an adduct, very probably $(HCFCl{\cdot}CFCl)Mn(CO)_5$, were obtained on repeated recrystallisation of the residue; the amount of adduct was insufficient for identification other than by infrared spectroscopy.

Reaction between $HMn(CO)_5$ and $CCl_2{:}CCl_2$ at 70° afforded trichloroethylene (42%), traces of 1,1- and 1,2-dichloroethylene, and the saturated hydrocarbons obtained by reduction of these olefins. A similar reaction involving 1,1-dichloroethylene gave vinyl chloride and ethylene in small yield. The non-gaseous residues of both reactions were mostly decacarbonyldimanganese and the carbonyl halides $Mn(CO)_5Cl$ and $[Mn(CO)_4Cl]_2$.

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[8] R. J. Mawby, F. Basolo, and R. G. Pearson, *J. Amer. Chem. Soc.*, 1964, **86**, 5043.

[9] J. L. Boston, D. W. A. Sharp, and G. Wilkinson, *J.*, 1962, 3488.

[10] P. M. Treichel, E. Pitcher, and F. G. A. Stone, *Inorg. Chem.*, 1962, **1**, 511.

[11] (*a*) W. Hieber and G. Wagner, *Annalen*, 1958, **618**, 24; (*b*) *Z. Naturforsch.*, 1958, **13***b*, 338.

[12] J. B. Wilford, P. M. Treichel, and F. G. A. Stone, *J. Organometallic Chem.*, 1964, **2**, 119.

It may be concluded that, whereas $HCo(CO)_4$ and $HMn(CO)_5$ add to tetrafluoroethylene to afford stable metal carbonyl complexes, with the less highly fluorinated ethylenes and with chloroethylenes, particularly those containing more than one chlorine atom, addition is followed by decomposition, *e.g.*,

$$CF_2{:}CH_2 + HMn(CO)_5 \xrightarrow{130^\circ} \underset{\text{unstable}}{(H_3C_2F_2)Mn(CO)_5} \xrightarrow{HMn(CO)_5} Mn_2(CO)_{10} + CH_3{\cdot}CF_2H$$

$$CCl_2{:}CCl_2 + HMn(CO)_5 \xrightarrow{70^\circ} \underset{\text{unstable}}{(HC_2Cl_4)Mn(CO)_5} \longrightarrow Mn(CO)_5Cl + CCl_2{:}CClH$$

$$\xrightarrow{HMn(CO)_5} (H_2C_2Cl_3)Mn(CO)_5 \longrightarrow \begin{cases} \longrightarrow Mn(CO)_5Cl + CH_2{:}CCl_2 + CHCl{:}CHCl \\ \xrightarrow{HMn(CO)_5} Mn_2(CO)_{10} + CH_2Cl{\cdot}CHCl_2 \end{cases}$$

From $CCl_2{:}CF_2$ somewhat unstable pentacarbonylmanganese complexes can be isolated.

EXPERIMENTAL

Reactions involving olefins and metal carbonyl hydrides were carried out using a high-vacuum system. Tetracarbonylhydridocobalt,[5] pentacarbonylhydridomanganese,[13] and *cis*-tetracarbonyl-σ-methyl(triphenylphosphine)manganese [14] were prepared as previously described. Tetrafluoroethylene, chlorotrifluoroethylene, and *trans*-1,2-dichloro-1,2-difluoroethylene were prepared by standard procedures; other reagents were commercial samples. Fluorine-19 and proton n.m.r. spectra were obtained using a Perkin-Elmer R-10 spectrometer. Gases recovered from the various reactions were identified by their infrared spectra.

Reaction between Tetracarbonylhydridocobalt and Tetrafluoroethylene.—Tetracarbonylhydridocobalt [from octacarbonyldicobalt (3·0 g., 8·8 mmoles)] and tetrafluoroethylene (264 c.c., 11·8 mmoles) were held in a trap between −40° and −10° for 3½ hr., during which time a black deposit formed. Fractionation in the vacuum system afforded *tetracarbonyl-σ-tetrafluoroethylcobalt* (318 mg., 10%) as a yellow liquid (Table 2), 1,1,2,2-tetrafluoroethane (31·4 c.c., 12%), and unreacted tetrafluoroethylene (240 c.c., 10·7 mmoles). The residue consisted of octacarbonyldicobalt and dodecacarbonyltetracobalt (1·0 g.), identified spectroscopically. In a similar experiment, with pentane (20 ml.) as solvent, $(HCF_2{\cdot}CF_2)Co(CO)_4$ (352 mg., 11%) was again obtained.

Treatment of $(HCF_2{\cdot}CF_2)Co(CO)_4$ (350 mg., 1·3 mmoles), with triphenylphosphine (300 mg., 1·1 mmoles) in refluxing cyclohexane (25 ml.), afforded, after evaporation of solvent, yellow *crystals* of $(HCF_2{\cdot}CF_2)Co(CO)_3(PPh_3)$ (427 mg., 77%) (from cyclohexane) (Table 2).

TABLE 2

		Found (%)			Required (%)		
Compound	M. p.	C	H	F	C	H	F
$(HCF_2{\cdot}CF_2)Co(CO)_4$	−9 to −8°	26·6	0·6	27·8	26·5	0·4	27·9
$(HCF_2{\cdot}CF_2)Co(CO)_3(PPh_3)$ *	142—143	54·9	3·3	—	54·6	3·2	—
$(C_6F_5)Co(CO)_4$ †	38—39	35·3	0·0	28·0	35·5	0·0	28·1
$(HCCl_2{\cdot}CF_2)Mn(CO)_5$	72—73	25·7	0·3	11·5	25·6	0·3	11·6
$(CH_3{\cdot}CF_2{\cdot}CF_2)Mn(CO)_4(PPh_3)$ ‡	>160 (decomp.)	55·7	3·8	—	55·2	3·3	—

* Mol. wt.: Found, 505 (in benzene) (Mechrolab Osmometer); Calc., 506. † Co: Found, 17·2; Calc., 17·4. ‡ Mol. wt.: Found, 540; Calc., 544.

Tetracarbonyl-σ-perfluorophenylcobalt.—A solution of sodium tetracarbonylcobaltate(−I), prepared from octacarbonyldicobalt (1·3 g., 3·8 mmoles) and excess of 1% sodium amalgam, in tetrahydrofuran (50 ml.) was slowly added to perfluorobenzoyl chloride (1·75 g., 7·6 mmoles) at −30°, the mixture being stirred for 1 hr. After evaporation of solvent, the residue was chromatographed on Florisil using ether–pentane mixtures as elutant. In this way *tetracarbonyl-σ-perfluorophenylcobalt* (1·92 g., 75%) (Table 2) and perfluorobenzophenone (50 mg.) (identified by comparing its infrared spectrum with that of an authentic sample) were successively eluted. The complex $(C_6F_5)Co(CO)_4$ was obtained as pale yellow needles by sublimation (20°/0·1 mm.).

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[13] R. B. King and F. G. A. Stone, *Inorg. Synth.*, 1963, **7**, 198.

[14] W. Hieber, G. Faulhaber, and F. Theubert, *Z. anorg. Chem.*, 1962, **314**, 125.

Synthesis of trans-$(CH_3\cdot CF_2\cdot CF_2)Mn(CO)_4(PPh_3)$.—(*a*) A sample (708 mg., 1·6 mmoles) of *cis*-$CH_3Mn(CO)_4(PPh_3)$, tetrafluoroethylene (271 c.c., 12·1 mmoles), and pentane (50 ml.) was irradiated (250w Hanovia lamp) for 4½ hr., to afford pale yellow *crystals* (528 mg., 61%) (from pentane) (Table 2).

(*b*) Freshly sublimed $(CH_3\cdot CF_2\cdot CF_2)Mn(CO)_5$ (555 mg., 1·79 mmoles) and triphenylphosphine (453 mg., 1·73 mmoles) were refluxed in cyclohexane (50 ml.) for 22 hr., the reaction being followed by infrared spectroscopy. After filtration and evaporation of solvent, the brown residue was extracted with pentane (2 × 30 ml.), to afford *trans*-$(CH_3\cdot CF_2\cdot CF_2)Mn(CO)_4(PPh_3)$ (540 mg., 57%), identical with the product from (*a*).

Reaction between Tetracarbonylhydridocobalt and Chlorotrifluoroethylene.—Tetracarbonylhydridocobalt [from octacarbonyldicobalt (3·6 g., 10·6 mmoles)] and chlorotrifluoroethylene (330 c.c., 14·7 mmoles) were mixed at −40° to −10° for 4 hr. Fractionation afforded 1-chloro-1,2,2-trifluoroethane (28 c.c., 1·25 mmoles), unreacted chlorotrifluoroethylene (298 c.c., 13·3 mmoles), and octacarbonyldicobalt.

Reaction between Pentacarbonylhydridomanganese and 1,1-*Difluoroethylene.*—Preliminary experiments showed that below 130° no reaction occurred between these compounds. Pentacarbonylhydridomanganese (843 mg., 4·3 mmoles) and 1,1-difluoroethylene (186 c.c., 8·3 mmoles) were distilled into a thick-walled Pyrex bulb (100 ml.) and heated at 130° for 25 hr.; a copious deposit of decacarbonyldimanganese appeared. On opening the bulb to the vacuum line, hydrogen (67 c.c., 3·0 mmoles), unreacted difluoroethylene (159 c.c., 7·1 mmoles), and 1,1-difluoroethane (22·4 c.c., 46%) were recovered. Solvent extraction of the residue gave decacarbonyldimanganese (670 mg., 80%). In similar experiments with pentacarbonylhydridomanganese, reduction of $CF_2{:}CFH$ (80°), $CFH{:}CH_2$ (130°), and $CH_2{:}CH_2$ (60°) occurred, to yield the corresponding fluoroethanes and ethane, respectively.

Reaction between Pentacarbonylhydridomanganese and 1,1-*Dichloro*-2,2-*difluoroethylene.*—Samples of $HMn(CO)_5$ (1·16 g., 5·9 mmoles) and $CCl_2{:}CF_2$ (320 c.c., 14·3 mmoles) were distilled into a 1-litre bulb. A rapid reaction occurred on warming to room temperature, and it was completed by heating (3 hr./40°). Unreacted $CCl_2{:}CF_2$ (246 c.c., 11·0 mmoles) and 1,1-dichloro-2,2-difluoroethane (20 c.c., 0·9 mmole) were recovered. Rapid extraction of the residue with pentane (20 ml.) afforded a yellow solid, shown spectroscopically to contain $Mn_2(CO)_{10}$, and a mixture of $(HCCl_2\cdot CF_2)Mn(CO)_5$ and $(HCF_2\cdot CCl_2)Mn(CO)_5$ in which the latter predominated. The crude solid was dissolved in pentane (3 ml.). After centrifugation, this was chromatographed on a Florisil column (8 × 2 cm.). Elution with pentane afforded, successively, decacarbonyldimanganese and $(HCCl_2\cdot CF_2)Mn(CO)_5$ (10 mg.). No trace of $(HCF_2\cdot CCl_2)Mn(CO)_5$ was detected, even on elution with ether.

From a similar reaction using 885 mg. (4·25 mmoles) of $HMn(CO)_5$ and 1497 c.c. (66·8 mmoles) of $CCl_2{:}CF_2$, a yellow solid (880 mg.) was obtained, m. p. 55—70°. Repeated recrystallisation from pentane under nitrogen, followed by sublimation (20°/0·01 mm.), afforded pure $(HCCl_2\cdot CF_2)Mn(CO)_5$ (39 mg.) (Table 2). The residues contained $Mn_2(CO)_{10}$ and $[Mn(CO)_4Cl]_2$.

TABLE 3

Fluorine-19 and proton chemical shifts and spin coupling constants (c./sec.); measured for 10% solutions in trichlorofluoromethane or hexafluorobenzene

Compound	δ(H) * (τ)	δ(α-CF₂) † (p.p.m.)	δ(β-CF₂) † (p.p.m.)	*J*(H—Fα)	*J*(H—Fβ)	*J*(Fα—Fβ)
$(HCF_2\cdot CF_2)Co(CO)_4$	4·70 ‡	41·7	119·3	3·7	57·1	4·7
$(HCF_2\cdot CF_2)Co(CO)_3(PPh_3)$	4·25,‡ 2·55 §	48·2 ¶	121·1	5·5	57·0	5·5
$(HCCl_2\cdot CF_2)Mn(CO)_5$	3·74	47·2	—	11·0	—	—
$(HCF_2\cdot CCl_2)Mn(CO)_5$	3·91	—	105·2	—	61·0	—

* Tetramethylsilane as internal standard. † Relative to CCl_3F; increasing to high field. ‡ Triplets of triplets. § Unresolved peak due to phenyl protons. ¶ This resonance is split by the ^{31}P nucleus (J = 30·5 c./sec.).

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