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THE REACTIONS OF COBALT AND RHODIUM CARBONYL CLUSTERS WITH OLEFINS

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Summary

Dodecacarbonyltetracobalt, $Co_4(CO)_{12}$, and dodecacarbonyltetrarhodium, $Rh_4(CO)_{12}$, react with olefins to give new olefin complexes, such as $Rh_6(CO)_{14}$(Diene), $Rh_2(CO)_2(NBD)_3$ (NBD = norbornadiene), $Co_4(CO)_9$ (CHT), $Co_4(CO)_6(CHT)_2$ (CHT = cycloheptatriene), $Co_2(CO)_4$(COT) and $Rh_4(CO)_8(COT)_2$ (COT = cyclooctatetraene), in which the basic structures of the parent cluster carbonyls are either retained or broken down. Plausible structures for these complexes are discussed on the basis of IR, NMR, and mass spectrometric data.

Introduction

Recently, many metal cluster complexes have been synthesized and their reactions described. However, there have been only a few studies on cobalt and rhodium carbonyl clusters. Substitution reactions of dodecacarbonyltetracobalt, $Co_4(CO)_{12}$, and dodecacarbonyltetrarhodium, $Rh_4(CO)_{12}$, with phosphorus ligands [1 - 4] and with alkynes [4 - 7] have been reported. Hexadecacarbonylhexarhodium, $Rh_6(CO)_{16}$, has been reported to react with thiols, halogens and carboxylic acids [8]. We have now investigated the reactions of $Co_4(CO)_{12}$ and $Rh_4(CO)_{12}$ with olefins. They react with diolefins, cycloheptatriene and cyclooctatetraene to give several new olefin complexes, and we describe below the preparation and characterization of these complexes.

Results and discussion

Reactions with diolefins

Although the reactions of $Co_4(CO)_{12}$ and $Rh_4(CO)_{12}$ with monoolefins, such as 1-hexene, acrylonitrile and dimethyl maleate did not afford any olefin complexes, they reacted with diolefins to give several diene complexes. Reactions of $Rh_4(CO)_{12}$ with diolefins, such as 1,5-cyclooctadiene, norbornadiene, 1,4-cyclohexadiene and 2,3-dimethyl-1,3-butadiene in refluxing n-hexane gave crystalline complexes. These complexes were also obtained in better yields

TABLE 1

PHYSICAL PROPERTIES AND ANALYTICAL DATA FOR $Rh_6(CO)_{14}(Diene)$

Compound	Physical properties		Analysis, found (cald.) (%)		Mol. wt.[e]
	Colour	M.p. (°C)	C	H	found (calcd.)
$Rh_6(CO)_{14}(1,5\text{-COD})$ [a](I)	Dark brown	ca. 140 (dec.)	23.65 (23.64)	1.04 (1.08)	1118[f] (1118)
$Rh_6(CO)_{14}(NBD)$ [b](II)	Reddish brown	ca. 145 (dec.)	23.13 (22.89)	0.64 (0.73)	1020 (1101)
$Rh_6(CO)_{14}(1,4\text{-CHD})$ [c] (III)	Dark brown	ca. 110 (dec.)	22.52 (22.04)	0.67 (0.74)	1033 (1090)
$Rh_6(CO)_{14}(2,3\text{-DM-}1,3\text{-BD})$ [d] (IV)	Black	ca. 135 (dec.)	22.20 (22.00)	0.98 (0.92)	1063 (1091)

[a] 1,5-COD = 1,5-cyclooctadiene. [b] NBD = norbornadiene. [c] 1,4-CHD = 1,4-cyclohexadiene. [d] 2,3-DM-1,3-BD = 2,3-dimethyl-1,3-butadiene. [e] VPO method in CH_2Cl_2. [f] Mass spectroscopy.

from the reactions of $Rh_6(CO)_{16}$ with diolefins in tetrahydrofuran under reflux. Their physical properties and analytical data are shown in Table 1. The mass spectrum of the 1,5-cyclooctadiene complex (I) showed a molecular ion peak at *m/e* 1118. As the mass spectra of the other diene complexes were not obtained, their molecular weights were measured by the vapor pressure osmometry method, which indicated values of about 1000. The IR spectra of the complexes showed absorptions at 1780 - 1790 cm^{-1} indicating the presence of triply-bridging carbonyls, in addition to absorptions at 2000 - 2100 cm^{-1} attributable to terminal carbonyls (Table 2). This pattern resembles that of $Rh_6(CO)_{16}$.

From the above data, the diene complexes are formulated as $Rh_6(CO)_{14}$-(Diene) [(I)–(IV)].

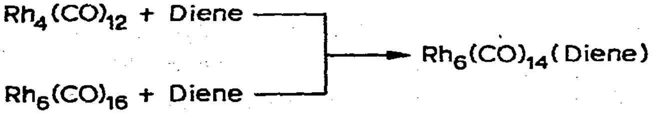


TABLE 2

IR AND NMR DATA FOR RHODIUM COMPLEXES

Compound	IR data	NMR data (δ ppm)[a]	
	ν(CO) (cm^{-1})	Olefinic protons	Other protons
(I)	2105 m, 2087 (sh), 2072 vs, 2045 m, 2037 m, 1813 w (br), 1795 m (br), 1781 s (br), 1778 s (br) [b]	4.70 (4H)	1.98 (4H) 2.60 (4H)
(II)	2103 m, 2070 vs, 2059 vs, 2035 s, 2024 s, 1815 m, 1796 s (br), 1781 s (br), 1768 s (br) [c]		
(III)	2103 m, 2070 vs, 2045 m, 2037 m, 2013 w, 1820 (sh, br), 1792 s (br)[b]		
(IV)	2105 m, 2083 (sh), 2072 vs, 2045 m, 2037 m, 1816 (sh, br), 1801 m (br), 1777 s (br) [b]	3.55 (4H)	2.19 (6H)
(V)	1828 vs[c]		
(IX)	2002 vs (br), 1763 s (br), 1741 s (br) [d]	4.96 (8H) 5.05 (4H) 6.12 (4H)	

[a] In $CDCl_3$ solution, TMS internal standard. [b] In CCl_4 solution. [c] Nujol mull. [d] In CH_2Cl_2 solution.

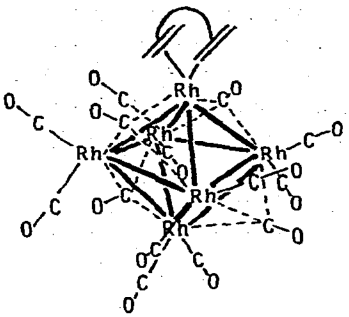


Fig. 1. Proposed structure of $Rh_6(CO)_{14}$ (Diene).

From scale models, it seems that the dienes in $Rh_6(CO)_{14}$(Diene) should be coordinated to one rhodium atom. A plausible structure consistent with the spectroscopic data is shown in Fig. 1. In these reactions, rearrangements of the metal atom cluster from tetranuclear to hexanuclear were observed.

The reaction of $Rh_4(CO)_{12}$ with norbornadiene at room temperature gave a dinuclear complex which was formulated as $Rh_2(CO)_2(C_7H_8)_3$ (V) from elemental analysis and mass spectral data. The mass spectrum showed a parent molecular ion peak $[Rh_2(CO)_2(C_7H_8)_3]^+$, together with peaks corresponding to the stepwise loss of two molecules of carbon monoxide and one molecule of norbornadiene giving $[Rh_2(C_7H_8)_3(CO)_n]^+$ ($n = 0, 1$) and $[Rh_2(C_7H_8)_2]^+$. The IR spectrum showed the presence of bridging carbonyls only at 1828 cm^{-1} (Table 2). The NMR spectrum was not obtained because of the low solubility of the complex. A plausible structure obtained from the above data is shown in Fig. 2. Other diolefins gave no corresponding complexes.

The reactions of $Co_4(CO)_{12}$ which had the same structure as $Rh_4(CO)_{12}$ were also investigated. Reactions of $Co_4(CO)_{12}$ with diolefins, such as norbornadiene, 1,3-cyclohexadiene, 1,4-cyclohexadiene and 2,3-dimethyl-1,3-butadiene in n-hexane under reflux gave the complexes $Co_2(CO)_4$(Diene)$_2$ which were identical with those previously prepared from $Co_2(CO)_8$ and diolefins [9]. The breakdown of the metal atom cluster to a dinuclear framework was observed in these cases.

$$Co_4(CO)_{12} + \text{Diene} \longrightarrow Co_2(CO)_4(\text{Diene})_2$$

Reactions with cycloheptatriene

It has recently been reported that $Co_4(CO)_{12}$ reacts with arenes to give $Co_4(CO)_9$(Arene), in which arenes coordinate to one cobalt atom [10]. The

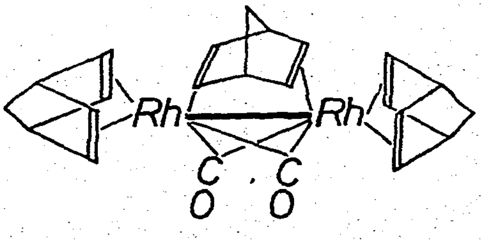
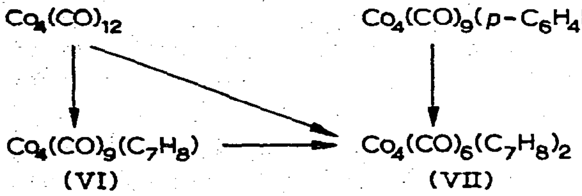


Fig. 2. Proposed structure of $Rh_2(CO)_2(C_7H_8)_3$.

reaction of $Co_4(CO)_{12}$ or $Rh_4(CO)_{12}$ with cycloheptatriene is expected to give either complexes similar to the above arene complexes or those in which cycloheptatriene coordinates to three cobalt or rhodium atoms beneath the basal M_3 (M = Co or Rh) triangle of $M_4(CO)_{12}$. The reaction of $Co_4(CO)_{12}$ with cycloheptatriene in n-hexane under reflux gave two kinds of cycloheptatriene complexes, which were formulated as $Co_4(CO)_9(C_7H_8)$ (VI) and $Co_4(CO)_6(C_7H_8)_2$ (VII) respectively. The longer the reaction time, the higher the yield of (VII), but the yield of (VI) was almost unchanged. The reaction of (VI) with cycloheptatriene gave (VII), indicating the formation of (VII) via (VI). Complex (VII) was also prepared in better yield in the reaction of $Co_4(CO)_9(p\text{-}C_6H_4Me_2)$ [10] with cycloheptatriene.



In the mass spectra, the parent molecular ions $[Co_4(CO)_9(C_7H_8)]^+$ and $[Co_4(CO)_6(C_7H_8)_2]^+$, together with peaks corresponding to the stepwise loss of nine or six carbonyl groups giving $[Co_4(C_7H_8)]^+$ or $[Co_4(C_7H_8)_2]^+$ are readily identifiable. These results indicated that both complexes retained the parent metal atom cluster, and that three or six carbonyl groups had been replaced by one or two cycloheptatriene molecules, respectively. The IR and NMR spectral data for these complexes are shown in Table 3. The NMR spectrum of complex (VI) is very similar to that of $(C_7H_8)Cr(CO)_3$ which has signals at δ 6.01 (2H), 4.83 (2H), 3.4 (2H), 2.9 (1H) and 1.73 (1H) ppm in CCl_4 [11]. The carbonyl stretching frequencies are also similar to those observed in the spectrum of $Co_4(CO)_9(m\text{-}C_6H_4Me_2)$ [2080 m, 2040 vs, 2022 vs, 1999 s, 1993 s, 1967 w and 1818 vs cm^{-1} in CCl_4]. These results suggest that cycloheptatriene coordinates to one cobalt atom as a tridentate ligand. A plausible structure is shown in Fig. 3. Complex (VII) was moderately stable in the solid state under nitrogen, but decomposed gradually in solution. The IR spectrum showed the presence of both terminal and bridging carbonyl groups

TABLE 3

IR AND NMR DATA FOR COBALT COMPLEXES

Compund	IR data	NMR data (δ ppm)[a]	
	ν(CO) (cm^{-1})	Olefinic protons	Other protons
(VI)	2082 m, 2066 w, 2043 vs, 2025 vs, 2000 s, 1995 s, 1855 w, 1820 vs[b]	3.85 (2H) 5.53 (2H) 6.85 (2H)	1.18 (1H) 2.46 (1H)
(VII)	2080 w, 2065 w, 2009 m, 1985 vs (br), 1736 s (br)[b]		
(VIII)	2078 (sh), 2065 s, 2033 vs, 2015 vs, 1999 vs, 1986 m, 1965 w, 1837 vs[c]	4.29 (8H)	

[a] In $CDCl_3$ solution, TMS internal standard. [b] In CCl_4 solution. [c] In n-hexane solution.

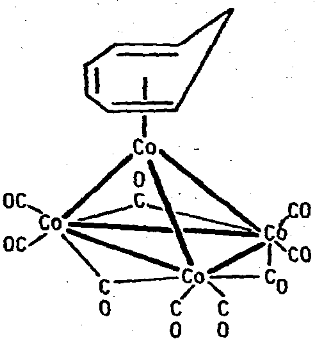


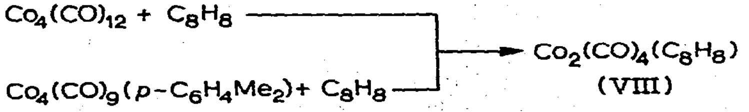
Fig. 3. Proposed structure of $Co_4(CO)_9(C_7H_8)$.

(Table 3). The NMR spectrum could not be obtained because of the instability of the complex in solutions, and so the coordination mode of the cycloheptatriene could not be determined.

The reaction of $Rh_4(CO)_{12}$ with cycloheptatriene in refluxing benzene gave no corresponding cycloheptatriene complexes, and $Rh_6(CO)_{16}$ was the only isolated product.

Reactions with cyclooctatetraene

It has been reported that the reaction of $PhCCo_3(CO)_9$ with cyclooctatetraene gave $PhCCo_3(CO)_6(C_8H_8)$, which has the ligand bound to three cobalt atoms beneath the basal Co_3 triangle [12, 13]. Both $Co_4(CO)_{12}$ and $Rh_4(CO)_{12}$ have the similar basal M_3 (M = Co or Rh) triangles, and it may be expected that their reactions with cyclooctatetraene lead to the formation of complexes similar to $PhCCo_3(CO)_6(C_8H_8)$. When the reaction of $Co_4(CO)_{12}$ with cyclooctatetraene was carried out in refluxing benzene, a black crystalline complex was isolated. This complex was also obtained in a better yield in the reaction of $Co_4(CO)_9(p\text{-}C_6H_4Me_2)$ with cyclooctatetraene. From elemental analysis and mass spectral data, the complex was found to be the dinuclear complex $Co_2(CO)_4(C_8H_8)$ (VIII).



The mass spectrum showed a parent molecular ion $[Co_2(CO)_4(C_8H_8)]^+$, together with peaks corresponding to the stepwise loss of four carbonyl groups giving $[Co_2(C_8H_8)]^+$. The NMR spectrum consisted of one sharp line (δ 4.29 ppm) suggesting fluxional behavior. Because of its instability, further investigation of its fluxionality was difficult. Since $(C_8H_8)_2Ru_3(CO)_4$ [14,15] and $(C_8H_8)Fe_2(CO)_5$ [16] are both fluxional molecules, it is not surprising that in complex (VIII), in which there is similar metal—ring bonding, fluxional behavior is also observed. The IR spectrum indicated the presence of terminal and bridging carbonyl groups (Table 3). Structure (a) in Fig. 4 is suggested by the above observations. However, the IR spectrum in n-hexane showed seven absorptions in the terminal carbonyl stretching region, which is not explicable in terms of structure (a) alone. It can be accounted for by postulating the presence of isomeric structures (b), (c) and (d) in solution.

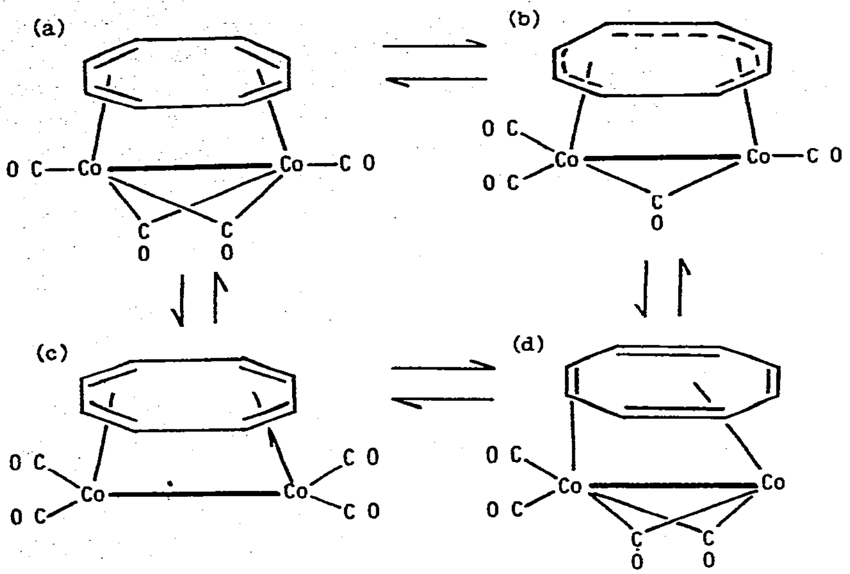


Fig. 4. Isomeric structures of $Co_2(CO)_4(C_8H_8)$ in solution.

The reaction of $Rh_4(CO)_{12}$ with cyclooctatetraene in benzene under reflux gave a dark brown complex. Although its mass spectrum could not be obtained, the complex was formulated as $Rh_4(CO)_8(C_8H_8)_2$ (IX) from elemental analysis and the molecular weight measurement by vapor pressure osmometry. The NMR spectrum in Table 2 indicates that the two cyclooctatetraene ligands coordinate to the rhodium cluster in two different ways. It is thought that one cyclooctatetraene molecule is bonded to the rhodium cluster through the 1,5-diene system [δ 6.12 (4H) and 5.05 (4H) ppm], while the other is bonded through the 1,3-diene system and exhibits fluxional behavior [δ 4.96

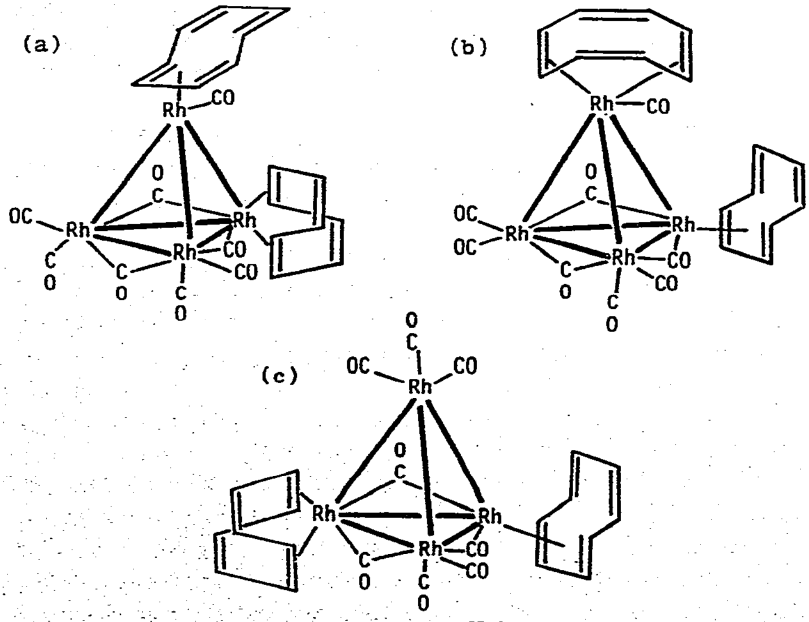


Fig. 5. Possible structures for $Rh_4(CO)_8(C_8H_8)_2$.

(8H) ppm]. For further investigation of the fluxionality, the temperature dependence of the NMR spectrum was studied, but no appreciable change of the signals was observed, even though the temperature was taken down to −70°. The IR spectrum indicated the presence of terminal and bridging carbonyl groups. However, no further information on the structure was obtained because of the broadness of the carbonyl absorptions (Table 2). From scale models, structures in which cyclooctatetraene coordinates to two rhodium metals as a bridge type ligand can be eliminated. Three structures may be postulated in the light of the available data (Fig. 5), and we cannot yet distinguish between them. X-Ray crystallography will be necessary to determine the structure unambiguously.

$$\underset{}{Rh_4(CO)_{12} + C_8H_8 \rightarrow \underset{\text{(IX)}}{Rh_4(CO)_8(C_8H_8)_2}}$$

Experimental

General comments

All reactions were carried out under dry nitrogen. The IR spectra of the products were recorded on a Jasco Model DS—402G grating spectrometer. NMR spectra were recorded on a Hitachi—Perkin—Elmer R-20 instrument with tetramethylsilane as internal standard, and mass spectra were recorded on a JEOL Model JMS 06 mass spectrometer. The molecular weights were measured in methylene dichloride solutions with a Knauer vapor pressure osmometer.

Reactions of diolefins

(a) With $Rh_4(CO)_{12}$. A typical procedure was as follows: A mixture of $Rh_4(CO)_{12}$ (0.3 g, 0.40 mmol) and 1,5-cyclooctadiene (0.9 g, 8.33 mmol) in n-hexane was refluxed for 3 h. Evaporation, and extraction of the residue with 1/3 benzene/n-hexane was followed by chromatography of the extract (silica gel). Elution with 1/3 benzene/n-hexane gave a dark brown fraction. Evaporation, followed by recrystallization from hot n-hexane, gave air-stable dark brown crystals of $Rh_6(CO)_{14}$(1,5-COD) (0.184 g, 62%). Subsequent elution with methylene dichloride gave $Rh_6(CO)_{16}$ (0.052 g, 18%), which was identified by its IR spectrum. $Rh_4(CO)_{12}$ reacted with norbornadiene, 1,4-cyclohexadiene, and 2,3-dimethyl-1,3-butadiene under similar conditions. The yields were 40, 51 and 88%, respectively.

(b) With $Rh_6(CO)_{16}$. A typical procedure was as follows. A mixture of $Rh_6(CO)_{16}$ (0.2 g, 0.188 mmol) and 1,5-cyclooctadiene (2 g, 18.5 mmol) in tetrahydrofuran was refluxed for 2 h. Evaporation, and extraction of the residue with 1/2 benzene/n-hexane was followed by chromatography (silica gel). Elution with 1/2 benzene/n-hexane gave a brown fraction. Evaporation, followed by recrystallization from hot n-hexane, gave air-stable dark brown crystals of $Rh_6(CO)_{14}$(1,5-COD) (0.034 g, 16%). This was identified by comparison of its IR spectrum and melting point with those of the complex obtained from the reaction of $Rh_4(CO)_{12}$ with 1,5-cyclooctadiene.

$Rh_6(CO)_{16}$ reacted with norbornadiene, 1,4-cyclohexadiene, and 2,3-dimethyl-1,3-butadiene under similar conditions to give the corresponding Rh (CO)(Diene). The yields were 81, 58 and 88% respectively.

(c) With $Rh_4(CO)_{12}$. A mixture of $Rh_4(CO)_{12}$ (0.06 g, 0.08 mmol) and norbornadiene (2 g, 21.74 mmol) in benzene was stirred for 5 min at room temperature. The unreacted $Rh_4(CO)_{12}$ was removed by filtration. The red filtrate was allowed to stand for a few hours, and air-stable pale yellow needles of $Rh_2(CO)_2(NBD)_3$ (0.025 g, 30%), m.p. 185 - 189° (dec.), were deposited. They were filtered off, washed with n-hexane, dried in vacuo and recrystallized from hot benzene. (Found: C, 51.46; H, 4.46%; mol. wt. in CH_2Cl_2 540; M^+, 538. $C_{23}H_{24}Rh_2O_2$ calcd.: C, 51.32; H, 4.50%; mol. wt., 538.)

(d) With $Co_4(CO)_{12}$. A typical procedure was as follows: A mixture of $Co_4(CO)_{12}$ (0.318 g, 0.556 mmol) and 1,4-cyclohexadiene (1.78 g, 22.3 mmol) in n-hexane was refluxed for 7 h. The resulting dark brown solution was chromatographed on a column of silica gel. The unreacted $Co_4(CO)_{12}$ (dark brown band) was removed by the first elution with n-hexane. Further elution with 1/4 benzene/n-hexane gave a yellow fraction. Evaporation, followed by the recrystallization from benzene/hexane, gave orange-yellow crystals of $Co_2(CO)_4(1,4\text{-CHD})_2$ (0.09 g, 21%), which were identified by IR spectroscopy.

$Co_4(CO)_{12}$ reacted with norbornadiene, 1,3-cyclohexadiene or 2,3-dimethyl-1,3-butadiene under similar conditions to give orange-yellow crystalline complexes. The yields were 48, 63 and 4% respectively.

Reaction of cycloheptatriene with $Co_4(CO)_{12}$

A mixture of $Co_4(CO)_{12}$ (0.93 g, 1.63 mmol) and cycloheptatriene (5 g, 54.35 mmol) in n-hexane was refluxed for 9 h. After removal of the solvent, the black residue was chromatographed on a column of silica gel. Elution with n-hexane gave a reddish brown fraction, but this was too unstable to be isolated. Further elution with 1/4 benzene/n-hexane gave a greenish brown fraction. Evaporation, followed by recrystallization from benzene/hexane, gave air-stable black crystals of $Co_4(CO)_9(C_7H_8)$ (0.1 g, 11%), m.p. 103 - 105° (dec.). (Found: C, 33.38; H, 1.35%; mol. wt., in CH_2Cl_2, 606; M^+, 580. $C_{16}H_8Co_4O_9$ calcd.: C 33.13; H, 1.39%; mol. wt., 580.) Elution with benzene then gave an air-sensitive green fraction. After evaporation was followed by recrystallization from benzene/n-hexane, dark green crystals of $Co_4(CO)_6(C_7H_8)_2$ (0.148 g, 23%) were obtained. (Found: C, 40.86; H, 2.84%; mol. wt., in CH_2Cl_2, 611; M^+, 588. $C_{20}H_{16}Co_4O_6$ calcd.: C, 40.85; H, 2.74%; mol. wt., 588.)

The reaction of $Co_4(CO)_9(p\text{-}C_6H_4Me_2)$ with cycloheptatriene under similar conditions gave $Co_4(CO)_6(C_7H_8)_2$ (yield 47%).

Reactions of cyclooctatetraene

(a) With $Co_4(CO)_{12}$. A mixture of $Co_4(CO)_{12}$ (0.5 g, 0.87 mmol) and cyclooctatetraene (0.5 g, 4.80 mmol) in benzene was refluxed for 8 h. After removal of the solvent, the black residue was chromatographed on a column of silica gel. Elution with n-hexane gave a dark brown fraction, $Co_4(CO)_{12}$. Further elution with 1/3 benzene/n-hexane gave a purplish red fraction. Evaporation, followed by recrystallization from n-hexane, gave air-sensitive black crystals of $Co_2(CO)_4(C_8H_8)$ (0.011 g, 2%), m.p. 92-95° (dec.). (Found: C, 43.28; H, 2.60%; mol. wt., in CH_2Cl_2, 383; M^+, 334. $C_{12}H_8Co_2O_4$ calcd.: C, 43.15; H, 2.41%; mol. wt., 334.)

The reaction of cyclooctatetraene with $Co_4(CO)_9(p\text{-}C_6H_4Me_2)$ under similar conditions gave $Co_2(CO)_4(C_8H_8)$ (yield 26%).

(b) With $Rh_4(CO)_{12}$. A mixture of $Rh_4(CO)_{12}$ (0.2 g, 0.27 mmol) and cyclooctatetraene (0.3 g, 2.88 mmol) in benzene was refluxed for 3 h. Evaporation and extraction of the residue with 2/1 benzene/n-hexane was followed by chromatography (silica gel). Elution with 2/1 benzene/n-hexane gave a dark brown fraction. Evaporation, followed by recrystallization from benzene/n-hexane, gave dark brown crystals of $Rh_4(CO)_8(C_8H_8)_2$ (0.173 g, 77%), m.p. 148-152° (dec.). (Found: C, 34.04; H, 2.18%; mol. wt., in CH_2Cl_2, 816. $C_{24}H_{16}O_8Rh_4$ calcd.: C, 34.15; H, 1.91%; mol. wt., 8.44).

References

1 W. Hieber and R. Brea, Chem. Ber., 90 (1957) 1259.
2 G. Cetini, O. Grambina, R. Rossetti and P.L. Stanghellini, Inorg. Chem., 7 (1968) 609.
3 R. Whyman, Chem. Commun., (1970) 230; R. Whyman, J. Chem. Soc., Dalton, Trans., (1972) 1375.
4 B.L. Booth, M.J. Else, R. Frelds and R.N. Haszeldine, J. Organometal. Chem., 27 (1971) 119.
5 U. Krüerke and W. Hübel, Chem. Ber., 94 (1961) 2829.
6 R.S. Dickson and G.R. Tailby, Aust. J. Chem., 23 (1970) 229.
7 Y. Iwashita and F. Tamura, Bull. Chem. Soc. Japan, 42 (1970) 1517.
8 B.F.G. Johnson, J. Lewis and P.W. Robinson, J. Chem. Soc. A, (1970) 1100.
9 G. Winkhaus and G. Wilkinson, J. Chem. Soc., (1961) 602.
10 I.U. Khand, G.R. Knox, P.L. Pauson and W.E. Watts, Chem. Commun., (1971) 36.
11 M.A. Bennett, L. Pratt and G. Wilkinson, J. Chem. Soc., (1961) 2037.
12 B.H. Robinson and J. Spencer, J. Organometal. Chem., 33 (1971) 97.
13 M.D. Brice, R.J. Dellaca and B.R. Penfold, Chem. Commun., (1971) 72.
14 F.A. Cotton, A. Davison and A. Musco, J. Amer. Chem. Soc., 89 (1967) 6796.
15 M.J. Bennett, F.A. Cotton and P. Legzdins, J. Amer. Chem. Soc., 89 (1967) 6797.
16 F.B. Fleischer, A.L. Stone, R.B.K. Dewar, J.D. Wright, C.E. Keller and R. Pettit, J. Amer. Chem.Soc., 88 (1966) 3158.