Arenecarbonylcobalt clusters with functional substituents in a side chain

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(Received September 25th, 1987)

Abstract

Arenecarbonyl clusters of the type $(arene)Co_4(CO)_9$ (II) [arene $= Me_3C_6H_3$, C_6Me_6, $Mes(CH_2)_3Mes$ ($Mes = 2,4,6\text{-}Me_3C_6H_2$), $Ph(CH_2)_nCH=CH_2$ ($n = 2, 3$), $Ph(CH_2)_2Br$, $Mes(CH_2)_3OH$, $C_6Me_5CH_2OH$, $Mes(CH_2)_3COOEt$, $Ph(CH_2)_n Fe(CO)_2Cp$ ($n = 1, 2$) and $Mes(CH_2)_3MesCr(CO)_3$] were prepared by reaction of $Co_2(CO)_8$ with various arenes in hexane. The compounds obtained are rather unstable with respect to strong nucleophilic agents. The complex $Me_3C_6H_3Co_4(CO)_8PPh_3$ (III) was prepared via photochemical substitution of CO by triphenylphosphine and its properties were investigated. Complexes II react with electrophiles in a different manner depending on the nature of the latter. Some properties of arenecarbonylcobalt clusters and arenechromium tricarbonyls are compared.

Introduction

We have previously obtained a number of arene π-complexes of transition metals of the type I with ω-functional groups in a side chain, and have studied their chemical transformations [1–7] (in some cases they particularly were converted into chelates)

(I)
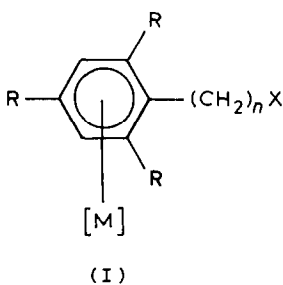
R = H, CH$_3$; n = 0–4; X = Cl, Br, OH, CH=CH$_2$, COCH$_3$, COOH, COOEt, CN, etc. [M] = Cr(CO)$_3$ [1–4]; Mo(CO)$_3$ [5,6]; W(CO)$_3$ [6]; Mn(CO)$_2$CN [7].

Here we describe the synthesis of similar compounds with a tetracobaltnona-carbonyl group:

(II)
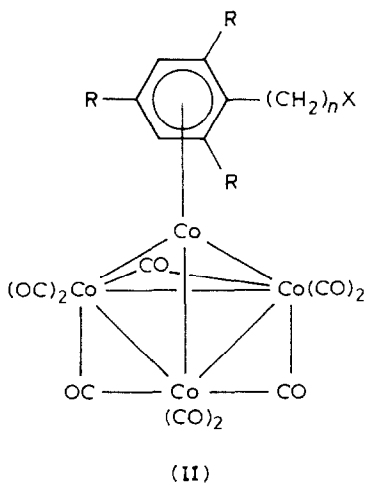
There are only a few reports in the literature on the preparation of similar clusters derived from benzene, its homologues, and alkoxy-substituted benzenes, the yields not being higher than 35% [8–10].

Results and discussion

To obtain complexes II, we somewhat modified the procedure previously described [8] and prepared the compounds with various arenes including those with functional groups in a side chain [11]:

$$\text{arene} + 2 \, Co_2(CO)_8 \xrightarrow[\text{–7 CO}]{\text{hexane; } \Delta} \text{arene } Co_4(CO)_9$$

(IIa–l)
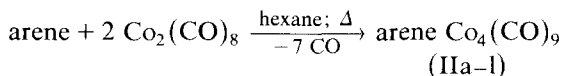
The best results were achieved upon reflux of cobalt carbonyl with a two to three-fold excess of arene in hexane, to give products in 30–60% yields (see Table 1). It should be noted that electron-withdrawing groups in the aromatic ring hinder the reaction. Only traces of the benzophenone derivative are formed. In the case of CN and COOH substituents in a side chain, the reactions are more complex, and the expected products were not isolated. By contrast, reactions with arenes containing organometallic groups σ-Fe(CO)$_2$C$_5$H$_5$ and π-Me$_3$C$_6$H$_2$Cr(CO)$_3$ in a side chain, took place smoothly to give the appropriate complexes of the type II.

The compounds obtained are dark-green, almost black crystalline solids, stable in the air and soluble in organic solvents and result in dark-green or brown-green solutions. Their IR spectra in heptane contain five ν(C≡O) bands of Co$_4$(CO)$_9$ (one of which is bridging, see Table 1), whereas iron- and chromium-containing complexes IIj, IIk and IIl have additional bands corresponding to the carbonyl ligands. A spectrum of IIi (in KBr) has an ester band at 1730 cm^{-1}. It should be noted that
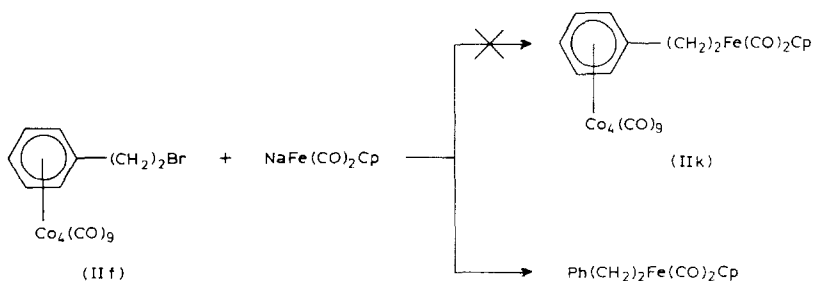
Table 1

Com-pound	Arene	Reaction time (h)	Yield (%)	M.p. (°C)	Analysis (Found (calc) (%))			Empirical	ν(C≡O) (heptane) (cm^{-1})
					C	H	Co		
IIa	1,3,5-Me$_3$C$_6$H$_3$	22	55	200–205 [a]	36.04 (35.56)	1.21 (1.99)	38.56 (38.77)	C$_{18}$H$_{12}$Co$_4$O$_9$	1833m [b], 1997w, 2011m, 2029s, 2071m
IIb	C$_6$Me$_6$	14	60	150–155 (dec.)	38.86 (38.80)	2.87 (2.79)	35.51 (36.26)	C$_{21}$H$_{18}$Co$_4$O$_9$	1817 [h], 1997, 2010, 2025, 2070 [c]
IIc	Mes(CH$_2$)$_3$Mes	14	52	120–125 [a]	47.34 (46.90)	3.85 (3.67)	30.63 (30.68)	C$_{30}$H$_{28}$Co$_4$O$_9$	1828 [b], 1996, 2010, 2028, 2070
IId	Ph(CH$_2$)$_2$CH=CH$_2$	14	55	85–87	36.98 (36.80)	1.93 (1.95)	37.64 (38.02)	C$_{19}$H$_{12}$Co$_4$O$_9$	1833 [b], 1999, 2013, 2032, 2072
IIe	Ph(CH$_2$)$_3$CH=CH$_2$	14	44	60–62	38.08 (37.88)	2.31 (2.23)	37.31 (37.18)	C$_{20}$H$_{14}$Co$_4$O$_9$	1834 [b], 1999, 2014, 2031, 2073
IIf	Ph(CH$_2$)$_2$Br	14 [d]	42	90–92 (dec.)	30.35 (30.34)	1.39 (1.35)	35.07 (35.03)	C$_{17}$H$_9$BrCo$_4$O$_9$	1845 [b], 2000, 2015, 2031, 2074
IIg	Mes(CH$_2$)$_3$OH	14 [d]	25 [e]	130–135 (dec.)	37.55 (37.87)	2.59 (2.72)	35.06 (35.39)	C$_{21}$H$_{18}$Co$_4$O$_{10}$	1828 [b], 1994, 2010, 2026, 2070
IIh	C$_6$Me$_5$CH$_2$OH	14 [d]	36 [e]	142–147 [a]	38.38 (37.87)	2.88 (2.72)	35.28 (35.39)	C$_{21}$H$_{18}$Co$_4$O$_{10}$	1817 [b], 1995, 2011, 2027, 2069
IIi	Mes(CH$_2$)$_3$COOEt	7	36	95–97 (dec.)	39.88 (39.91)	3.04 (3.07)	32.11 (32.64)	C$_{24}$H$_{22}$Co$_4$O$_{11}$	1827 [b], 1994, 2009, 2027, 2070
IIj	PhCH$_2$Fe(CO)$_2$Cp	22 [d]	41	120–125 [a]	36.61 (36.55)	1.56 (1.60)	30.95 (31.19)	C$_{23}$H$_{12}$Co$_4$FeO$_{11}$	1831m [b], 1965m, 2001m, 2013s, 2032s, 2074m
IIk	Ph(CH$_2$)$_2$Fe(CO)$_2$Cp	22 [d]	37	102–104 (dec.)	37.55 (37.44)	1.77 (1.83)	30.44 (30.62)	C$_{24}$H$_{14}$Co$_4$FeO$_{11}$	1834m [b], 1964m, 2001m, 2014s, 2032s, 2074m
IIl	Mes(CH$_2$)$_3$MesCr(CO)$_3$	22 [d]	30	105–110 (dec.)	44.31 (43.83)	3.53 (3.12)	25.57 (26.07)	C$_{33}$H$_{28}$Co$_4$CrO$_{12}$	1830m [b], 1901s, 1968s, 1997w, 2012m, 2029s, 2073m

[a] Decomposes without melting. [b] Bridging CO. [c] Peak intensities in IIb–i correspond to those in IIa. [d] Optimal reaction time is given, although unchanged cobalt carbonyl remains in solution. The yields decrease, when cobalt carbonyl has been completely consumed. [e] For crystallized product.

dissolution of complexes II in THF leads to the formation of some unstable coordinative products as is evidenced from IR spectra in the CO bridging region, in which one common band for all solvents (~ 1830 cm^{-1}), and two additional bands at lower frequencies are found. After THF solvent had been evaporated off, an IR spectrum of the product was found to be the same as that of initial one, i.e. THF adduct formation is reversible. This phenomenon was not observed in other solvents (ether, chloroform, dioxane, acetone).

A study of the chemistry of complexes II has shown their marked sensitivity towards several nucleophilic agents which cause the cleavage of arene–cobalt bonds. Attempted independent preparation of complex IIk, by the reaction of IIf with $NaFe(CO)_2C_5H_5$, yielded only $Ph(CH_2)_2Fe(CO)_2C_5H_5$ which was identified from its IR and mass spectra, and from its R_f on Silufol:

Even the more stable compound, IIa, was decomposed by this nucleophile at $-78°$ C. It is important that the reaction with the chromium tricarbonyl analogue, $Ph(CH_2)_2BrCr(CO)_3$, gives a product in which the $Fe(CO)_2Cp$ replaces bromine, the Cr–arene bond being unaffected [4]. The arene–M bond in clusters II is thus less resistant towards nucleophiles than those in arenechromium tricarbonyls. Aromatic ring in complexes II can be replaced by various other arenes. For example, after a prolonged reflux of IIa in benzene, the benzene derivative $C_6H_6Co_4(CO)_9$ can be separated from the starting material by column chromatography on silica gel. Complexes II are also decomposed by alcoholic alkali. At the same time reactions with phosphines and amines to give decomposition products are much slower at room temperature (time of complete decomposition varies from 5 h to several days). The decomposition strongly increases on heating.
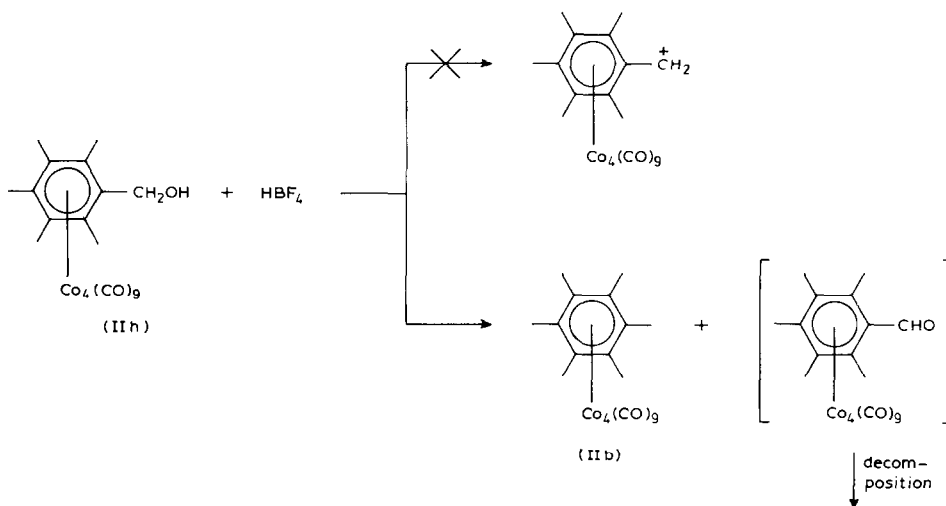
A photochemical reaction of IIa with PPh_3 in benzene solution even after 4–5 h leads to complete replacement of one CO group by triphenylphosphine:
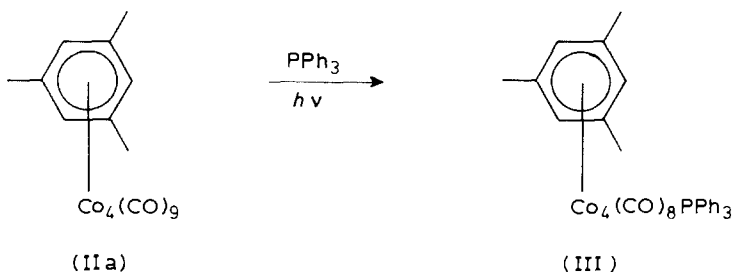
A product with two CO ligands replaced by PPh_3 was not detected even after

prolonged irradiation of IIa in the presence of a 1000% excess triphenylphosphine. Interaction of IIb with triphenylphosphine proceeds more slowly than in the case of IIa, the product formed being so unstable that even its IR spectrum could not be recorded. Such a decrease in stability of triphenylphosphine derivatives on going from the mesityl to hexamethylbenzene π-complex has also been observed in an arenechromium carbonyl family [12–14]. It is important to note that compounds derived from II by substituting tolane or maleic anhydride for CO were not obtained. Starting compounds were only recovered in 60–80% yield.

Studies on the reactivity of complexes II towards some electrophilic agents exemplified by IIa,b have revealed that IIb do not undergo protonation by trifluoroacetic acid in CH_2Cl_2 and their IR spectra in the metallocarbonyl region remain unchanged. Complexes of the type II with benzylic or 3,5-dimethylbenzylic alcohols are very unstable. The protonation of alcohol derivatives was therefore studied using the more stable complex IIh with pentamethylbenzylic alcohol. Under the action of the acid, it apparently behaves like a free ligand [15], i.e. it does not afford the carbenium ion but disproportionates. The only product isolated was IIb. Evidently the aldehyde complex which formed simultaneously, decomposed rapidly because of its instability, in a way similar to that of benzophenone complex (see above):

CH$_2$OH + HBF$_4$ $\longrightarrow$ CH$_2^+$ / Co$_4$(CO)$_9$; (IIh) Co$_4$(CO)$_9$; (IIb) Co$_4$(CO)$_9$ + [CHO Co$_4$(CO)$_9$] decomposition

Complexes IIa,b react rather slowly with $HgCl_2$ at room temperature, and a light-grey adduct precipitates after 20–30 min of reflux in CH_2Cl_2. After treatment with water, only 5–10% of the starting compound was found to be still reversed, i.e. the adducts are very labile. Under the action of $SnCl_4$ on IIa,b a light-blue adduct precipitates after 4–5 min. The action of water restores 50% of the initial complex, but the adduct decomposes immediately in air. The adducts were identified from IR spectra of their solutions in CH_2Cl_2: 2020 and 2105 cm^{-1} (with $HgCl_2$) and 2050, 2130 cm^{-1} (with $SnCl_4$). Complex III reacts with $HgCl_2$ even at room temperature, while with $SnCl_4$ the reaction occurs instantaneously with vigorous decomposition. The adducts were also identified from IR spectra of their CH_2Cl_2 solutions (1976s, 2014m, 2020m, 2103m in the case of $HgCl_2$ and 2012s, 2060s, 2133m in the case of $SnCl_4$). The observed high-frequency shift of the carbonyl bands is typical of carbonyl π-complexes of transition metals [16].

We attempted to obtain chelates IV from complexes II:

(IId , n = 2 ,
IIe , n = 3)

(IV)
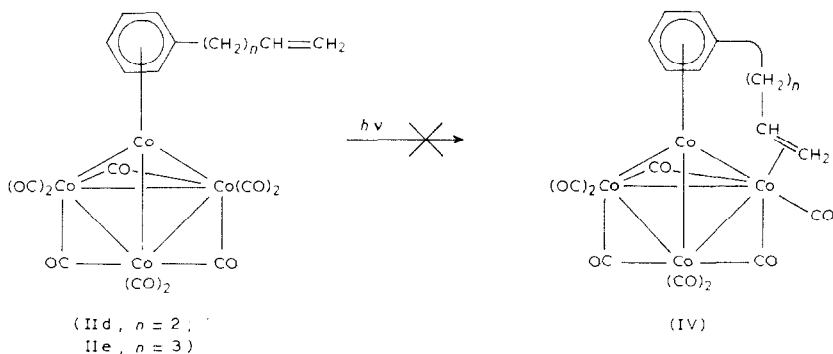
It turned out, however, that even under prolonged irradiation of IId,e in heptane, benzene or ether, both at room temperature and on heating do not lead to novel products and only the starting compounds were recovered in 70–80% yield. Irradiation in THF leads to vigorous decomposition, probably indicative of the formation of an intermediate unstable chelate. At the same time the corresponding unstable chelate is apparently formed on irradiation of complex IIi in THF, since a small band at 1590 cm^{-1} appears in the IR spectrum after 5–6 h, which is observed together with the bands from the starting compound and can be ascribed to the coordinated ester group. An attempt to separate the product from the starting compound resulted in decomposition.

The metal-containing complexes IIj,k and III show different behaviour on UV irradiation. The Fe–C σ-bond is cleaved on irradiation of IIj,k in THF, for example:
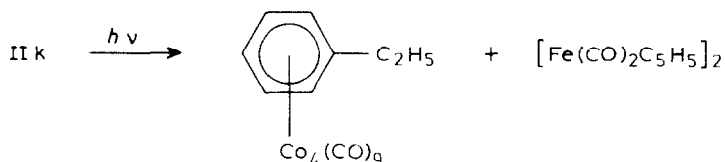
The same reaction, only slower, takes place when the complexes are irradiated in heptane or ether solutions. At the same time III loses the $Cr(CO)_3$ group to give IIc even when irradiated in heptane. This result is important, since it demonstrates the greater photochemical stability of the arene–Co_4 bond than that of the arene–Cr bond. It has been indicated that the trend is the reverse with respect to nucleophiles.

A study of IR and NMR spectra of $Me_3C_6H_3Co_4(CO)_9$ (IIa) and $Me_3C_6H_3Co_4(CO)_8$-PPh_3 (III)

An IR spectrum of III (in the $\nu(C\equiv O)$ region) contains an enhanced number of bands, namely five bands from terminal CO ligands and two from bridging ones, reflecting a decrease in symmetry of the molecule owing to the replacement of one CO unit by PPh_3. The oscillation frequencies are shifted to higher wavelengths by 20–40 cm^{-1} compared with starting IIa, and the shifts are considerably lower than for those of the mesitylenechromium carbonyl analogues (45–75 cm^{-1} [17]).

With regard to the ^1H NMR spectrum of IIa (see Table 2), signals of not only aromatic, but also of methyl protons were observed further downfield than those of

Table 2

^{1}H NMR spectral data (δ, ppm) ^{a}

Compound	Me	H$_{aromatic}$	PPh$_3$
Me$_3$C$_6$H$_3$Co$_4$(CO)$_9$ (IIa)	2.53	6.23	–
Me$_3$C$_6$H$_3$Co$_4$(CO)$_8$PPh$_3$ (III)	2.54	6.10	7.13–7.79m
Me$_3$C$_6$H$_3$Cr(CO)$_3$	2.19	5.18	–

^{a} Signals are singlets, unless otherwise stated.

Table 3

^{13}C NMR spectral data (δ, ppm) ^{a}

Compound	Me	C–H	C–CH$_3$	CO	PPh$_3$
IIa	19.6	93.8	107.3	206.58	–
III	19.7	95.6	106.5	206.6	128.5 130.0 133.1
Me$_3$C$_6$H$_3$Cr(CO)$_3$ ^{b}	21.0	92.4	111.5	235.1	–

^{a} Signals are singlets, unless otherwise stated. ^{b} Ref. 19.

Me$_3$C$_6$H$_3$Cr(CO)$_3$. The effect can be connected with a stronger accepting ability of Co$_4$(CO)$_9$ compared with Cr(CO)$_3$. The anisotropy of the metal–arene bond in the complexes may, however, also play a role. It is noteworthy that although the arene ligand and triphenylphosphine are bound to the different cobalt atoms, the proton signals of the arene are sensitive to the presence of the phosphine and the signals are shifted upfield by 0.13 ppm (see Table 2). The shift is much lower than that found for mesitylenechromium carbonyls (0.6 ppm [18]). Signals from the mesitylene carbons of IIa and III in ^{13}C NMR spectra do not differ markedly and are close to those of mesitylenechromium carbonyl [19] (see Table 3). It should be pointed out here that carbonyl carbon signals appear as one signal at ~ 206.6 ppm, indicative of rapid interconversions between all CO ligands, and of the small influence effected by PPh$_3$ on the signal positions. Other groups, who have investigated the ^{13}C NMR spectra of the same compound, IIa [20], and that of the toluene analogue, C$_6$H$_5$CH$_3$Co$_4$(CO)$_9$ [21], recorded the spectra only at low temperature (−35 to −70°C). Under such conditions the carbonyl carbons gave three signals of equal intensity, but the averaged value of their chemical shift in IIa (205.72 ppm [20]) agrees well with that obtained by us. At the same time these workers were unable to record ^{13}C NMR spectra at room temperature.

Experimental

All operations for the synthesis and isolation of the compounds studied were carried out in absolute solvents under argon. A PRK-4 lamp was used for UV irradiation. IR spectra were recorded on a UR-20 and IKS-14A spectrophotometers, NMR spectra were recorded on a Bruker WP-200SY spectrometer in CD$_3$COCD$_3$ with HMDS as internal standard (^{1}H) and in CH$_2$Cl$_2$ (^{13}C). Mass spectra were obtained on a AEI MS-30 apparatus. Organic ligands were prepared by standard

procedures, $Ph(CH_2)_nFe(CO)_2Cp$ were obtained as described in [22]. A procedure for preparation of the novel complex, $Mes(CH_2)_3MesCr(CO)_3$, is given below.

Preparation of arene $Co_4(CO)_9$ (II)

A mixture of dicobalt octacarbonyl (0.34 g, 1 mmol) and ligand (2–3 ml for liquids or 0.3–0.5 g for solids) was refluxed in 70 ml of hexane (for reaction time see Table 1). After cooling, the solution together with precipitate was column chromatographed on silica gel (except for IIg and IIh) with a benzene/petroleum ether mixture (1/5). After separation of the first small band, which contained an excess of ligand and sometimes unchanged dicobalt octacarbonyl, the dark-green, product-containing band was collected. The solvent was removed in vacuo, and the residue was recrystallized from pentane. Yields, melting points and analytical data are given in Table 1.

Preparation of $Mes(CH_2)_3MesCr(CO)_3$

A mixture of $Cr(CO)_6$ (1 g) and 1,3-dimesitylpropane (5 g) was refluxed in a dibutyl ether (40 ml)/THF (4 ml) mixture for 18 h. The product was separated on a Al_2O_3 column; 0.72 g (38%) of $Mes(CH_2)_3MesCr(CO)_3$ was eluted with benzene. M.p. 114–115°C (from hexane). Found: C, 69.35; H, 6.79; Cr, 12.10. $C_{24}H_{28}CrO_3$ calc: C, 69.21; H, 6.78; Cr, 12.49%. ν(C≡O) (heptane): 1900, 1969 cm^{-1}.

Preparation of $Me_3C_6H_3Co_4(CO)_8PPh_3$ (III)

A solution of IIa (0.1 g, 0.165 mmol) and PPh_3 (0.1 g, 0.38 mmol) in 100 ml of benzene was irradiated with UV light at 10—20°C with stirring using a magnetic bar for 4–5 h. TLC showed the absence of the starting complex after this length of time. The solvent was removed in vacuo, and the residue was column chromatographed on silica gel eluting with a CH_2Cl_2/petroleum ether mixture (1/10). The excess of triphenylphosphine was removed at this stage. The product-containing brown band was eluted with methylene chloride. The solid, brown compound, $Me_3C_6H_3Co_4(CO)_8PPh_3$ (III) (0.09 g, 64%), was obtained after evaporation of the solvent in vacuo. Recrystallization from hexane gave nearly black, needle-like crystals which decompose without melting at 140–150°C. Found: C, 50.11; H, 3.24; Co, 28.49. $C_{35}H_{27}Co_4O_8P$, calc: C, 49.91; H, 3.23; Co, 27.99%. ν(C≡O) (heptane): 1804m, 1816m, 1969m, 1978m, 1988s, 2012s, 2043m cm^{-1}.

Protonation of $C_6Me_5CH_2OHCo_4(CO)_9$ (IIh)

To a stirred solution of IIh (0.133 g, 0.2 mmol) in 70 ml of benzene was added an excess of $HBF_4 \cdot OEt_2$ (0.2 ml), and the mixture was further stirred for 30 min. An excess of Et_2NH (0.3 ml) was then added and the stirring continued for 1 h. The solvent was removed in vacuo, and the residue column chromatographed on silica gel. The green band which was eluted with benzene contained 0.052 g (40%) of IIb. The product was identified from its IR spectrum in heptane (ν(C≡O)). R_f on Silufol and analytical data. Found: C, 38.50; H, 2.71. $C_{21}H_{18}Co_4O_9$ calc: C, 38.80; H, 2.79%.

Irradiation of $Ph(CH_2)_2Fe(CO)_2C_5H_5Co_4(CO)_9$ (IIk)

A solution of IIk (0.135 g) in 100 ml of THF was irradiated with UV light at 10–20°C for 6 h, after which the absence of starting complex was indicated by

TLC. The solvent was removed in vacuo, the residue separated by thin-layer chromatography on silica gel with a benzene/heptane mixture (1/1). In addition to the small amount of [Fe(CO)$_2$Cp]$_2$, 0.019 g (18%) of PhCH$_2$CH$_3$Co$_4$(CO)$_9$ was isolated; identified from mass spectral data (of $[M]^+ = 594$; $[M - 2CO] = 538$; $[M - 8CO] = 546$; the most intense peak belongs to ethylbenzene – 106). The compound decomposes without melting at 130–140°C. $\nu(C\equiv O)$ (heptane): 1835m, 2000w, 2013m, 2031s, 2072m cm^{-1}.

Irradiation of Mes(CH$_2$)$_3$MesCr(CO)$_3$Co$_4$(CO)$_9$ (III)

A solution of III (0.04 g) in 100 ml of heptane was irradiated with UV light at 10–20°C for 8 h. IR spectroscopy showed the absence of starting compound after this length of time. The solvent was removed in vacuo, and the only reaction product, IIc, was isolated by column chromatography on silica gel to yield 0.024 g (70%) of IIc identified by m.p., R_f and a complete IR spectrum.

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