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Reaction of $RCCo_3(CO)_9$ ($R = CH_3$, C_2H_5, C_6H_5) with $Na_2[Fe(CO)_4]$ in THF solution leads to the formation of the new mixed metal alkylidyne clusters $RCFeCo_2(CO)_9H$ ($R = CH_3$, C_2H_5, C_6H_5) in yields of 31-48%. Spectral data suggest that these have structures similar to those of the parent

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$RCCo_3(CO)_9$ clusters; i.e., an $FeCo_2$ triangle triply-bridged by the alkylidyne carbon. The possible location of the hydride ligand is discussed on the basis of electron-count, infrared, ^1H and ^{13}C NMR spectra. The reaction of $HCCo_3(CO)_9$ with $Na_2[Fe(CO)_4]$, which gave a complex mixture of products, is also discussed.

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Synthesis and Characterization of the Mixed-Metal Alkylidyne Clusters $RCFeCo_2(CO)_9H$ ($R = CH_3, C_2H_5, C_6H_5$)

by

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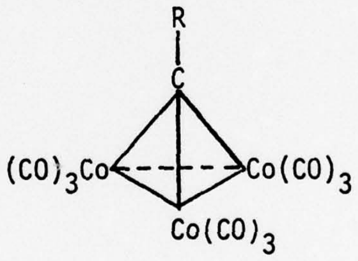
Introduction

Reaction of closed metal carbonyl trimers with carbonylmetalates has been shown[1-4] to be a useful synthetic route to tetranuclear clusters in which the carbonylmetalate adds to the face of the original trimer. This type of reaction has been particularly useful for the synthesis of mixed-metal clusters. In principle, clusters with more than four framework atoms could be prepared by the addition of carbonylmetalates to the triangular faces of tetranuclear and larger clusters.[5] In order to test this hypothesis we attempted to prepare pentanuclear clusters with an $FeCo_3C$ skeleton by the addition of $[Fe(CO)_4]^{2-}$ to a series of alkylidyne $RCCo_3(CO)_9$ (R = H, CH_3, C_2H_5, C_6H_5) clusters of structure 1.[6-9]



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However, the pentanuclear $FeCo_3C$ clusters were not isolated from these reactions, but rather the first examples of a new class of mixed metal alkylidyne clusters, $RCFeCo_2(CO)_9H$ (R = CH_3, C_2H_5, C_6H_5), were obtained. The synthesis and characterization of these clusters is described herein.

Experimental Section

The complexes $RCCo_3(CO)_9$ (R = H,[10] CH_3,[11] C_2H_5,[12] C_6H_5[11]) were prepared by published procedures. $Na_2[Fe(CO)_4]\cdot C_4H_8O_2$, hereafter referred to as $Na_2[Fe(CO)_4]$, was obtained from Alfa Ventron Corporation and was used without further purification. Tetrahydrofuran (THF) and hexane were dried and deoxygenated by distillation from CaH_2 under N_2. All other solvents were deoxygenated either by evacuation through a liquid N_2 cooled trap or by rigorous bubbling of N_2 through the solvent. Reactant solutions were prepared in an N_2-filled glove box, and all reactions and manipulations of the compounds were conducted under an N_2 atmosphere.

Chromatography was carried out using the low-pressure chromatography apparatus previously described.[2] The column was thoroughly flushed with deoxygenated hexane prior to chromatography of the reaction mixtures.

Spectral Measurements. Infrared spectra were recorded on a Perkin-Elmer 580 grating infrared spectrophotometer using 0.5 mm pathlength NaCl solution IR cells. Values reported are ± 1 cm^{-1}. The cells were fitted with serum caps and purged with N_2 in order to record spectra of the air-sensitive compounds. Mass spectra were obtained using an AEI-MS 9 spectrometer with a 70 eV ionization source. Electronic absorption spectra were measured on a Cary 17 spectrophotometer using 1-cm pathlength quartz cells. A JEOL PS-100-FT Fourier transform NMR spectrometer was used to obtain both ^1H and ^{13}C NMR spectra. [Cr(acetylacetonate)$_3$] (10^{-3} M) was added as a shiftless relaxation agent for the ^{13}C NMR measurements.

Preparation of $C_2H_5CFeCo_2(CO)_9H$. A 50-mL THF solution of $C_2H_5CCo_3(CO)_9$ (0.93 g, 2 mmol) was added dropwise over a 15 min period to a refluxing 125-mL THF solution of $Na_2[Fe(CO)_4]$ (1.40 g, 4 mmol). The color of the reactant solution changed from yellow-orange to deep red-brown during the addition, and the solution was refluxed for 1 h after the $C_2H_5CCo_3(CO)_9$ addition was complete. Removal of solvent by evaporation under vacuum gave a dark red-brown solid to which was added 50 mL of deoxygenated hexane. The hexane remained colorless until 50 mL of deoxygenated 40% aqueous H_3PO_4 was added, and then it acquired a red-black color as the residue dissolved and extracted. After stirring for 10 min the hexane was removed under N_2. The extraction was repeated 2-3 times and when complete the acid layer was red-pink in color.

The hexane extract was concentrated under vacuum and chromatographed on silica gel. Elution with deoxygenated hexane gave a red-purple band of $C_2H_5CCo_3(CO)_9$ followed by a green band of $Fe_3(CO)_{12}$. An immobile red fraction remained at the top of the column and was subsequently removed by elution with deoxygenated methanol. Evaporation of the methanol under vacuum followed by acidification and extraction as before gave a maroon solid.. Purification by sublimation at 50°C (10^{-3} mm Hg) gave a 46% yield (0.417 g, 0.91 mmol) of $C_2H_5CFeCo_2(CO)_9H$. Anal. Calcd. for $C_2H_5CFeCo_2(CO)_9H$: C, 30.80%; H, 1.64%; Co, 25.19%. Found, C, 31.10%; H, 1.28%, Co, 24.19%.

When the reaction of $C_2H_5CCo_3(CO)_9$ (0.140 g, 0.30 mmol) and $Na_2[Fe(CO)_4]$ (0.180 g, 0.52 mmol) was conducted at room temperature for 1.25 h using the workup described above, a 23% yield (0.031 g, 0.07 mmol) of $C_2H_5CFeCo_2(CO)_9H$ was obtained.

Preparation of $CH_3CFeCo_2(CO)_9H$. Reaction of $CH_3CCo_3(CO)_9$ (0.103 g, 0.23 mmol) and $Na_2[Fe(CO)_4]$ (0.070 g, 0.20 mmol) in refluxing THF for 1.25 h followed by work-up in a manner similar to that described above gave a 43% yield (0.045 g, 0.10 mmol) of $CH_3CFeCo_2(CO)_9H$ after sublimation at 50°C (10^{-3} mm Hg). Reaction at room temperature of $CH_3CCo_3(CO)_9$

(0.462 g, 1.01 mmol) with $Na_2[Fe(CO)_4]$ (0.526 g, 1.52 mmol) gave a 19% yield (0.084 g, 0.19 mmol) of $CH_3CFeCo_2(CO)_9H$.

Preparation of $C_6H_5CFeCo_2(CO)_9H$. Reaction of $C_6H_5CCo_3(CO)_9$ (0.446 g, 0.86 mmol) and $Na_2[Fe(CO)_4]$ (0.650 g, 1.88 mmol) in refluxing THF for 2 h followed by the work-up described above gave a 31% yield (0.140 g, 0.27 mmol) of $C_6H_5CFeCo_2(CO)_9H$. The corresponding reaction at room temperature of $C_6H_5CCo_3(CO)_9$ (0.135 g, 0.26 mmol) and $Na_2[Fe(CO)_4]$ (0.100 g, 0.29 mmol) gave a 19% yield (0.025 g, 0.05 mmol) of $C_6H_5CFeCo_2(CO)_9H$.

Results

General Synthetic Procedure. The synthesis of each of the $RCFeCo_2(CO)_9H$ alkylidyne clusters was carried out by the dropwise addition of a THF solution of the appropriate $RCCo_3(CO)_9$ cluster to a THF solution of $Na_2[Fe(CO)_4]$ under an N_2 atmosphere. After the specified reaction time (1-2 h), the solvent was removed under vacuum, and the residue was acidified with 40% H_3PO_4 and extracted into hexane. The hexane solution was then chromatographed on silica gel.

These $RCFeCo_2(CO)_9H$ clusters are somewhat acidic and readily deprotonate on silica gel during chromatography, remaining at the top of the column as a dark-red band. They can be removed from the silica as anions by changing the eluting solvent to methanol after the other components have been eluted with hexane. The nature of the accompanying cation is unknown. Removal of the methanol by evaporation under vacuum followed by protonation and extraction of the residue as before gives the neutral $RCFeCo_2(CO)_9H$ clusters. Mass spectra of the reaction mixtures before chromatography exhibited mass peaks due to the $RCFeCo_2(CO)_9H$ clusters, indicating their presence prior to chromatographic separation. Identical chromatographic behavior was shown by the isolated and purified compounds.

Of the various spectroscopic techniques used to identify the products of the reactions, the most useful were infrared and mass spectroscopy. Previously described compounds were identified mainly by comparison to their reported infrared spectra. The new $RCFeCo_2(CO)_9H$ clusters were primarily characterized by their mass spectra which showed parent ions with the correct m/e values and the expected isotopic distribution. In each case, ions corresponding to successive loss of nine carbonyls were observed. $C_2H_5CFeCo_2(CO)_9H$ was further characterized by elemental analysis, but $CH_3CFeCo_2(CO)_9H$ and $C_6H_5CFeCo_2(CO)_9H$ proved too thermally unstable to analyze. Spectroscopic data for the new clusters are set out in Tables I and II.

Preparation of $C_2H_5CFeCo_2(CO)_9H$. Reaction of $C_2H_5CCo_3(CO)_9$ with $[Fe(CO)_4]^{2-}$ proceeded cleanly to give $C_2H_5CFeCo_2(CO)_9H$ as the only detectable product. Unreacted $C_2H_5CCo_3(CO)_9$ and $Fe_3(CO)_{12}$, resulting from protonation of $[Fe(CO)_4]^{2-}$, were also isolated. When the reaction was conducted in refluxing THF, the yield of $C_2H_5CFeCo_2(CO)_9H$, based on the initial quantity of $C_2H_5CCo_3(CO)_9$ employed, was 46%. The yield decreased to 23% with a concommitant increase in the amount of unreacted $C_2H_5CCo_3(CO)_9$ recovered when the reactants were maintained at room temperature (∿22-25°C).

Preparation of $CH_3CFeCo_2(CO)_9H$. $CH_3CFeCo_2(CO)_9H$ was prepared by allowing $CH_3CCo_3(CO)_9$ to react with $[Fe(CO)_4]^{2-}$. Stirring these reactants at room temperature for 1-2 h gave five compounds upon chromatography of the acidified reaction mixture. These were, in order of elution, red-purple $CH_3CCo_3(CO)_9$ (5%), a pink compound, green $Fe_3(CO)_{12}$, blue $Co_4(CO)_{10}(CH_3C{\equiv}CCH_3)$[13](9%), and maroon $CH_3CFeCo_2(CO)_9H$ (19%). The mass spectrum of the pink compound exhibited a parent peak at m/e 452. The position of the parent ion and the isotopic distribution correspond to a compound with the molecular formula $CH_3CFe_2Co(CO)_9H_2$, but insufficient material was isolated for complete characterization.

The yield of $CH_3CFeCo_2(CO)_9H$ was increased to 43% by carrying out the reaction in refluxing THF. $CH_3CCo_3(CO)_9$, $Fe_3(CO)_{12}$, the pink compound, and a trace of an unidentified yellow compound were also isolated. No $Co_4(CO)_{10}(CH_3C{\equiv}CCH_3)$ was detected.

Preparation of $C_6H_5CFeCo_2(CO)_9H$. Unreacted $C_6H_5CCo_3(CO)_9$, $Fe_3(CO)_{12}$ and $C_6H_5CFeCo_2(CO)_9H$ in 19% yield were the only compounds isolated from the reaction of $C_6H_5CCo_3(CO)_9$ with $[Fe(CO)_4]^{2-}$ at room temperature. When conducted in refluxing THF, the yield of $C_6H_5CFeCo_2(CO)_9H$ increased to 31%, and trace amounts of two unidentified products were isolated in addition to unreacted $C_6H_5CCo_3(CO)_9$ and $Fe_3(CO)_{12}$.

Reaction of $HCCo_3(CO)_9$ with $[Fe(CO)_4]^{2-}$. The reaction of $HCCo_3(CO)_9$ with $[Fe(CO)_4]^{2-}$ gave a complex mixture of products. Prior to chromatography, the product mixture from a reaction maintained at room temperature exhibited a peak at m/e 780 in its mass spectrum. Peaks due to the stepwise loss of 11 carbonyls from this species were clearly evident with four additional carbonyl loss peaks partially obscured by other ions. The mass position, isotopic distribution of the m/e 780 ion, and the carbonyl loss pattern suggest the presence of a species with the formula $H_3Fe_3Co_3(CO)_{15}C$. We were never able to isolate this compound, however, as it presumably decomposed during the chromatographic separation of the product mixture.

Separation of this mixture by silica gel chromatography gave five distinct bands. The first was red-purple and corresponded in color and chromatography characteristics to unreacted $HCCo_3(CO)_9$. However, the infrared and mass spectrum of the material isolated from this band revealed it to be a mixture of $RCCo_3(CO)_9$ clusters with R = H, CH_3, C_2H_5 and C_3H_7 with the former two in greater abundance. The mass spectrum of the pink material isolated in trace quantity from the second band also indicated it to be a mixture of compounds. The mass positions and isotope patterns are consistent with the formula $RCFe_2Co(CO)_9H_2$ with R = C_2H_5, C_3H_7, and possibly CH_3. Further characterization was not possible because of the very small amount of material isolated. The third band was green $Fe_3(CO)_{12}$ and the fourth contained a trace quantity of an unidentified pink compound.

The chromatography characteristics and infrared spectrum of the final red band were similar to the other $RCFeCo_2(CO)_9H$ clusters described herein. However, we were unable to obtain a satisfactory mass spectrum for this material. The mass spectrum exhibited a complex series of peaks up to m/e 500 with the only identifiable patterns at m/e 454 and 440, possibly due to $CH_3CFeCo_2(CO)_9H$ and $HCFeCo_2(CO)_9H$. It may be that one of the compounds initially present in the sample ($HCFeCo_2(CO)_9H$?) pyrolyzes on the mass probe even at the relatively low temperature used (∿100°C). The ^1H NMR spectrum of this fraction exhibited a doublet at 2.57 ppm (J_{M-H} = 2.5 Hz) attributable to $CH_3CFeCo_2(CO)_9H$, Table 1, and singlets at 4.03, 2.20, 0.95, and 0.37 ppm which could not be assigned.

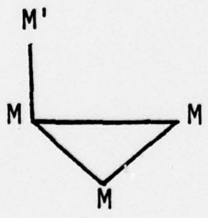
When this reaction was carried out in refluxing THF for 1-2 h, the same five bands resulted upon chromatography of the acidified reaction mixture, in addition to trace amounts of an unidentified yellow fraction between bands 1 and 2 and a brown fraction just leading the $Fe_3(CO)_{12}$ band. The mass spectrum of this brown fraction exhibited peaks at m/e 452, 466, and 480 consistent with the formulation $RCFe_3(CO)_9H_3$ with R = CH_3, C_2H_5, and C_3H_7, but insufficient material was isolated for further characterization.

Properties. The $RCFeCo_2(CO)_9H$ clusters are dark-maroon solids which give orange-red or orange solutions. They are soluble in most organic solvents, although they are somewhat unstable in alcohols and halogenated hydrocarbons. Solid samples and solutions of the clusters can be handled in air for short periods of time; solutions show signs of decomposition within 15-30 min and solids within 30-45 min. The compounds slowly decompose even under an N_2 atmosphere and are best stored under an atmosphere of CO at 0°C. Under these conditions, $C_2H_5CFeCo_2(CO)_9H$ showed no signs of decomposition after several weeks but some decomposition of $C_6H_5CFeCo_2(CO)_9H$ was evident after a period of several days. $CH_3CFeCo_2(CO)_9H$ and $C_2H_5CFeCo_2(CO)_9H$ can be purified by sublimation at 50°C (10^{-3} mm Hg), but $C_6H_5CFeCo_2(CO)_9H$ is less stable under the sublimation conditions and is not readily purified. Satisfactory elemental analysis was obtained only for $C_2H_5CFeCo_2(CO)_9H$ and only when a sample was mailed under an atmosphere of CO. The other two compounds apparently decomposed even under these conditions during shipment.

Discussion

Synthesis. The mixed metal alkylidyne clusters $RCFeCo_2(CO)_9H$ ($R = CH_3, C_2H_5, C_6H_5$) described herein resulted from the reaction of $[Fe(CO)_4]^{2-}$ with the appropriate $RCCo_3(CO)_9$ clusters. The spectral data summarized below indicates that the structures of these clusters are similar to those of the parent $RCCo_3(CO)_9$ compounds,[6-9] possessing an $FeCo_2$ triangle bridged by an alkylidyne carbon atom.

The mechanism by which these mixed-metal alkylidyne clusters result from the reaction of $[Fe(CO)_4]^{2-}$ with $RCCo_3(CO)_9$ is not well understood. Similar reactions of $[Fe(CO)_4]^{2-}$ with $Ru_3(CO)_{12}$, $Ru_2Os(CO)_{12}$ and $RuOs_2(CO)_{12}$ followed by protonation gave the tetranuclear clusters $H_2FeRu_3(CO)_{13}$, $H_2FeRu_2Os(CO)_{13}$, and $H_2FeRuOs_2(CO)_{13}$.[1,2] It was suggested that the first step in these reactions is nucleophilic attack of the carbonylmetalate at the electropositive carbon of a coordinated carbonyl ligand.[2,3] Elimination of CO would give a species such as 2 which could collapse through 3 to give the tetrahedral cluster 4.



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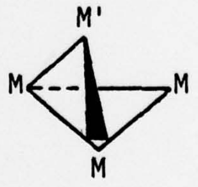

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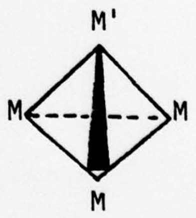

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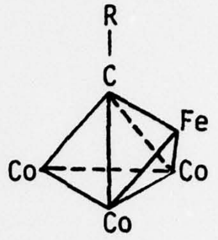
The formation of the mixed metal alkylidyne clusters reported herein can be rationalized in a similar manner, but with the alkylidyne carbon atom as an additional site of attack. It does not appear that the preferred site of addition is on the Co_3 face, since clusters with an

$FeCo_3C$ framework were not isolated, but rather on one of the Co_2C faces to produce an intermediate of possible skeletal structure 5.



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Elimination of a cobalt-carbonyl fragment would lead from 5 to the observed $RCFeCo_2(CO)_9H$ clusters, and indeed $HCo(CO)_4$ was detected in the acidified reaction mixture. It may be that a cluster with structure 5 persists as an anion, but that it readily fragments upon protonation.

In the reactions of $[Fe(CO)_4]^{2-}$ with $CH_3CCo_3(CO)_9$ and $HCCo_3(CO)_9$, compounds with an Fe_2CoC skeleton were detected by mass spectroscopy, but were formed in insufficient quantities to allow full characterization. These clusters could result by the reaction of $[Fe(CO)_4]^{2-}$ with the initially formed $[RCFeCo_2(CO)_9]^-$ anions in a manner similar to that described above followed by elimination of another cobalt-carbonyl fragment. Further reaction of $[Fe(CO)_4]^{2-}$ with the Fe_2CoC clusters would account for the $RCFe_3(CO)_9H_3$ clusters that were detected, but not conclusively identified, in one of the reactions employing $HCCo_3(CO)_9$.

The $H_3Fe_3Co_3(CO)_{15}C$ carbide cluster which was detected in the mass spectrum of the reaction mixture resulting from addition of $[Fe(CO)_4]^{2-}$ to $HCCo_3(CO)_9$ is similar to $[Co_6(CO)_{15}C]^{2-}$ which was isolated by Chini and coworkers[14,15] from the reaction of $ClCCo_3(CO)_9$ with $[Co(CO)_4]^-$. The carbide carbon in $[Co_6(CO)_{15}C]^{2-}$ was shown[15] to derive from the

the alkylidyne carbon atom of $ClCCo_3(CO)_9$. The alkylidyne carbon atom in $C_2H_5CFeCo_2(CO)_9H$ and $C_6H_5CFeCo_2(CO)_9H$ should be less susceptable to attack than in $HCCo_3(CO)_9$ and $CH_3CCo_3(CO)_9$ due to the larger substituents. This may explain why the reactions of the former two clusters with $[Fe(CO)_4]^{2-}$ proceeded to give fewer products than the reactions of the latter two.

We do not at all understand the mechanism by which alkylidyne clusters with R = CH_3, C_2H_5, and C_3H_7 result from the reaction of $[Fe(CO)_4]^{2-}$ with $HCCo_3(CO)_9$, but we are currently examining these reactions for their possible relevance to CO hydrogenation.

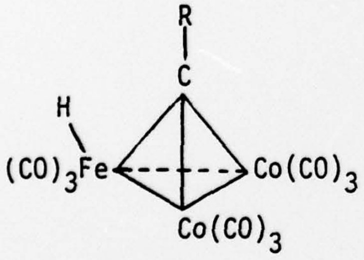
Spectroscopic Characterization. The molecular formulas of the mixed metal alkylidyne clusters were established by their mass spectra and for $C_2H_5CFeCo_2(CO)_9H$ by chemical analysis. The infrared, electronic absorption, and ^1H NMR spectra of these clusters are similar to those of the isoelectronic $RCCo_3(CO)_9$ clusters and suggest that the two series of compounds have similar structures.

The electronic absorption spectra of the $RCFeCo_2(CO)_9H$ clusters exhibit broad bands in the visible spectral region, Table I, similar to those previously observed[16] in the spectra of the parent $RCCo_3(CO)_9$ clusters. These latter bands were attributed to transitions localized within the triangular Co_3 framework.[16] The presence of a similar spectral feature in the mixed-metal clusters strongly suggests that these also possess a triangular array of metals.

The carbonyl-region infrared spectra of the $RCFeCo_2(CO)_9H$ clusters are also similar to those of the $RCCo_3(CO)_9$ clusters, except that the former exhibit a greater number of bands due to their lower symmetry, Figure 1 and Table I. No bands attributable to bridging carbonyls were observed in any of the spectra.

^1H NMR data for the $RCFeCo_2(CO)_9H$ clusters are summarized in Table I. Each cluster shows the expected resonances for the substituent on the alkylidyne carbon and a broad resonance 9-10 ppm upfield of TMS, indicative of a metal hydride ligand. A coupling of approximately 2.5 hz was observed between the hydride ligand and the hydrogen atoms on the carbon adjacent to the alkylidyne carbon, as illustrated by the spectrum of $C_2H_5CFeCo_2(CO)_9H$, Figure 2. Similar coupling was observed in the spectrum of $HCOs_3(CO)_9H_3$ (J = 1.1 hz)[17] but not in the spectrum of $CH_3CRu_3(CO)_9H_3$.[18] The ^{13}C NMR spectrum of $CH_3CFeCo_2(CO)_9H$[19] exhibits a broad singlet at 179.2 ppm (width at half-height = 1.7 ppm) which remains invariant through the temperature range 30°C to -50°C, the lowest temperature investigated. The carbonyl ligands are likely undergoing rapid exchange within this temperature range, as has been previously observed[20] for a number of $RCCo_3(CO)_9$ clusters.

The location of the hydride ligand cannot be deduced from the available spectroscopic data, and the four structures 6-9 must be considered.



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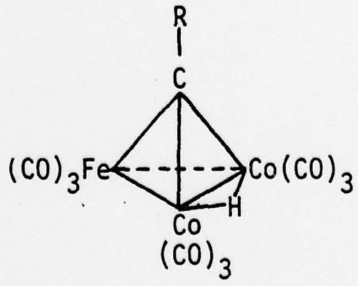

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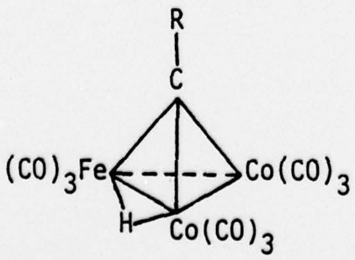

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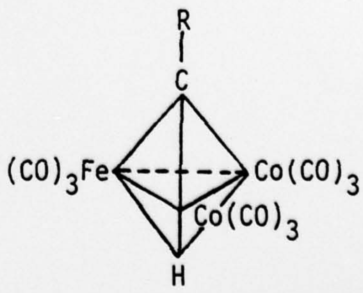

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In order for each metal atom in the $RCFeCo_2(CO)_9H$ cluster to possess a filled 18-electron valence shell, the hydride ligand should be terminally bound to iron as in structure 6. However, the hydride resonances in the ^1H NMR spectra are quite broad with widths at half-heights of approximately 0.9 ppm. The broadening is presumably due to coupling with the cobalt nuclei which have a quadrupole moment and a nuclear spin of 7/2. Such coupling would be expected for the static structures 7-9 in which the hydride bridges an iron-cobalt or a cobalt-cobalt bond, or the $FeCo_2$ face. Coupling could also result if the hydride ligand were fluxional at room temperature. Recent x-ray[21] and neutron[22] diffraction studies of $HFeCo_3(CO)_9\{P(OCH_3)_3\}_3$ have established that the hydride ligand bridges the Co_3 face in this compound, and presumably the unsubstituted cluster $HFeCo_3(CO)_{12}$ also possesses a face-bridging hydride. This latter cluster has been shown[16] to be electronically similar to the $RCCo_3(CO)_9$ clusters and hence a face-bridging position for the hydride in the $RCFeCo_2(CO)_9H$ complexes discussed herein seems likely. Unfortunately, ^1H NMR resonances due to the metal hydride of the parent[23] and substituted[21] $FeCo_3$ clusters have not been observed, and a comparison to the spectra reported herein cannot be made.

Acknowledgement

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Table I. Spectroscopic Data

Compound	IR ν_{CO} (cm^{-1})	UV-VIS λ_{max}, nm (ε_{max}, L mol^{-1}cm^{-1})	^{1}H NMR[a] δ (ppm)
$CH_3CFeCo_2(CO)_9H$	2101 m, 2065 m, 2053 s, 2047 s, 2037 s, 2018 m, 2014 m, 1994 w, 1989 m	495 (1370) 375 sh (3190)	H_M: -9.82 CH_3: 2.57 ($J_{CH_3-H_M}$ = 2.5 Hz)
$C_2H_5CFeCo_2(CO)_9H$	2101 m, 2065 m, 2053 s, 2046 s, 2038 s, 2017 m, 2014 m, 1994 w, 1989 m	530 (1390) 390 sh (4270)	H_M: -9.97 CH_3: 0.90 ($J_{CH_3-CH_2}$ = 7.0 Hz) CH_2: 2.75 ($J_{CH_2-CH_3}$ = 7.0 Hz; ($J_{CH_2-H_M}$ = 2.6 Hz)
$C_6H_5CFeCo_2(CO)_9H$	2106 mw, 2101 m, 2067 ms, 2056 s, 2047 s, 2037 s, 2024 m, 2015 w, 1998 mw, 1990 m, 1982 mw	540 sh 400 sh	H_M: -9.00 C_6H_5: complex multiplet centered at 7.17 ppm[b]

[a] H_M = metal hydride resonance.

[b] Multiplet structure is identical to that centered at 6.03 ppm in the spectrum of $C_6H_5CCo_3(CO)_9$.

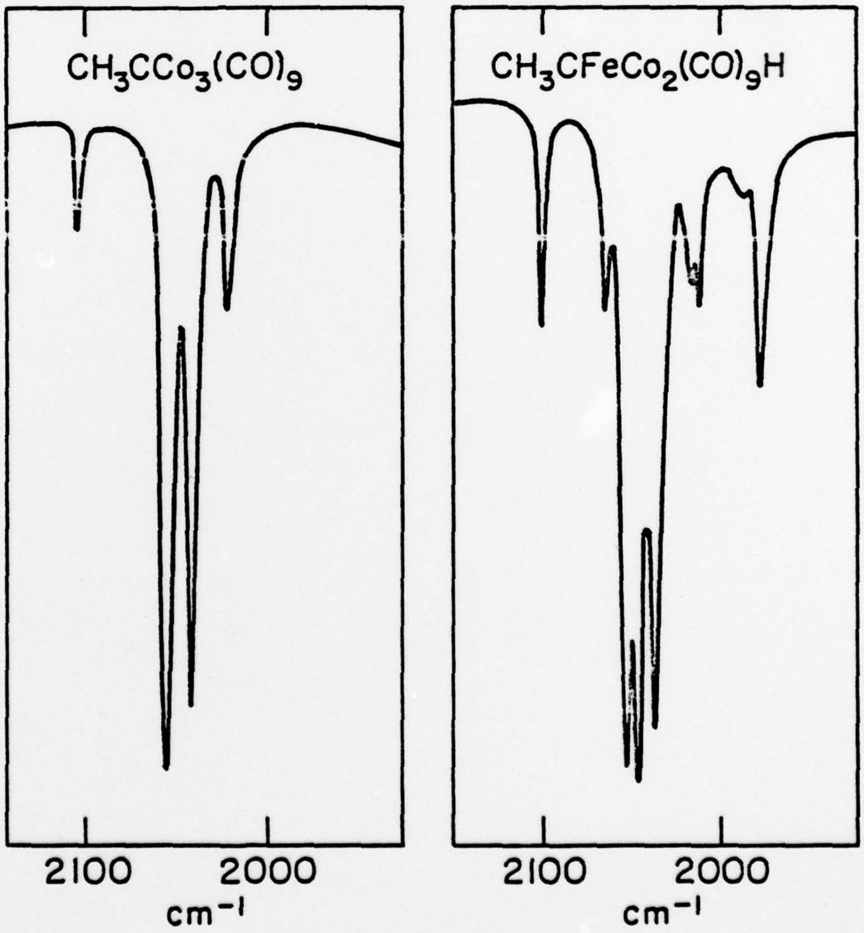
Table II. Mass Spectral Data

Compound	Parent Ion Isotopic Distribution (rel. intensity)	Other Principal Fragments
$CH_3CFeCo_2(CO)_9H$	452(12), 454(100) 455(18), 456(6)	426, 398, 370, 342, 314, 286, 258, 230, 202
$C_2H_5CFeCo_2(CO)_9H$	466(11), 468(100), 469(17) 470(5)	440, 412, 384, 356, 328, 300, 272, 244, 216
$C_6H_5CFeCo_2(CO)_9H$	514(9), 516(100) 517(20), 518(4)	488, 460, 432, 404, 376, 348, 320, 292, 264

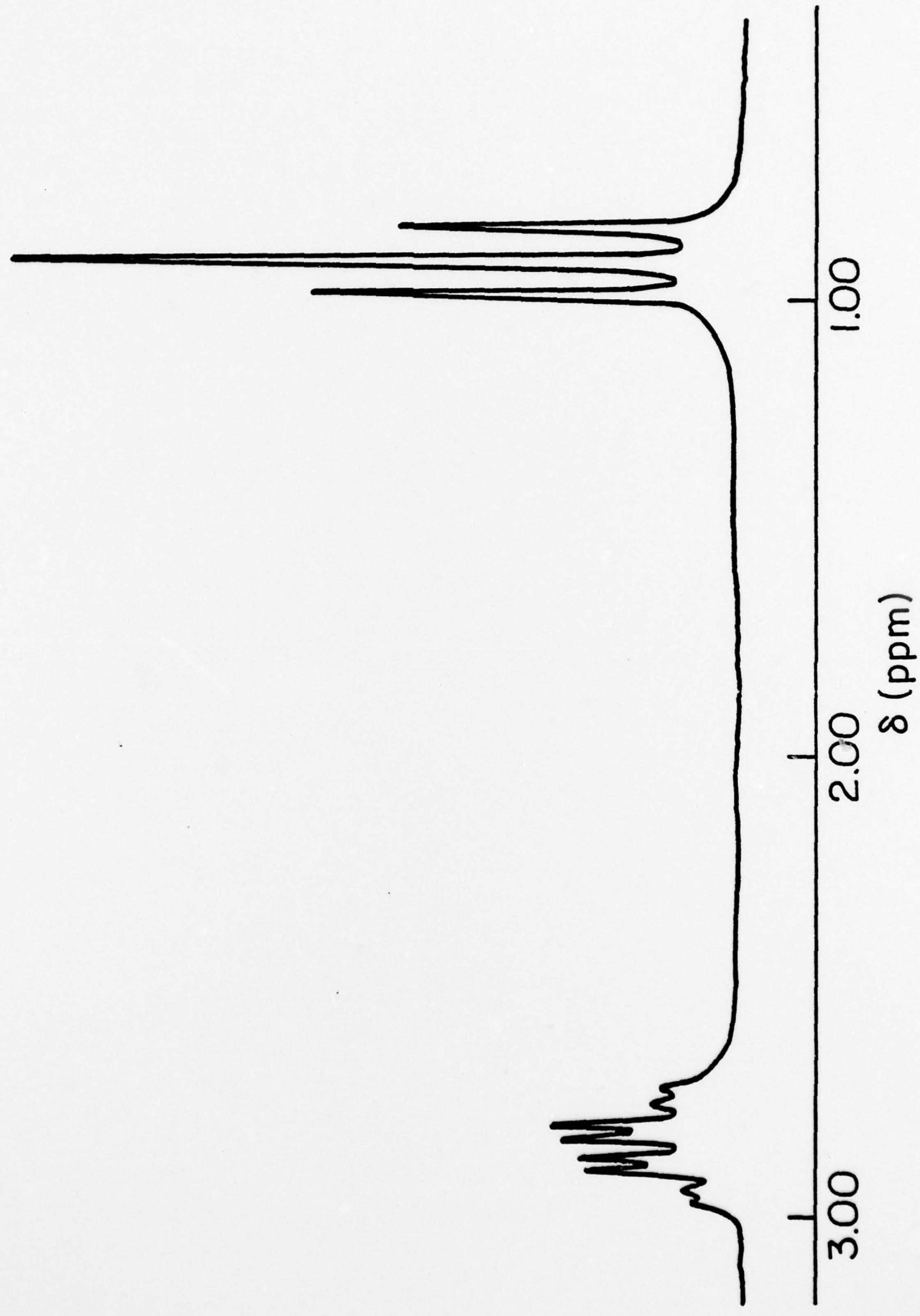
Figure Captions

Figure 1. Infrared spectra of $CH_3CCo_3(CO)_9$ and $CH_3CFeCo_2(CO)_9H$ in hexane solution.

Figure 2. ^1H NMR spectrum of $C_2H_5CFeCo_2(CO)_9H$ in C_6D_6 solution.



$CH_3CCo_3(CO)_9$
$CH_3CFeCo_2(CO)_9H$
2100
2000
cm⁻¹
2100
2000
cm⁻¹



1.00
2.00
3.00
δ (ppm)

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