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REGIOSPECIFIC SUBSTITUTION OF CARBONYL BY PHOSPHINE LIGANDS IN HYDROCARBYL-SUBSTITUTED CARBONYL CLUSTERS. SYNTHESIS AND SPECTROSCOPIC CHARACTERIZATION OF $Fe_3(CO)_8(PPh_3)(\mu_3\text{-}\eta^2\text{-}\perp\text{-}EtC_2Et)$ AND $(\eta^5\text{-}C_5H_5)NiFe_2(CO)_5(PPh_3)(\mu_3\text{-}\eta^2\text{-}\perp\text{-}C_2Bu^t)$

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Summary

The complexes $Fe_3(CO)_8(PPh_3)(\mu_3\text{-}\eta^2\text{-}\perp\text{-}EtC_2Et)$ and $(\eta^5\text{-}C_5H_5)NiFe_2(CO)_5(PPh_3)(\mu_3\text{-}\eta^2\text{-}\perp\text{-}C_2Bu^t)$ have been obtained by treating $Fe_3(CO)_9(C_2Et_2)$ or $(Cp)NiFe_2(CO)_6(C_2Bu^t)$ with PPh_3 under mild conditions; the substituted clusters have been characterized spectroscopically. Structures are proposed in which the phosphine is on the unique metal atom σ-bonded to the alkyne or acetylide moiety. Replacement of CO by PPh_3 ligands rather than by addition, is observed for the formally unsaturated $Fe_3(CO)_9(C_2Et_2)$. Reorientation of the acetylide was expected for $(Cp)NiFe_2(CO)_6(C_2Bu^t)$ upon substitution, but was not observed.

Introduction

Terminal and internal alkynes react readily with transition metal carbonyl clusters [1]; in particular, the interaction of one alkyne molecule or of one alkyne-derived acetylide with triangular metal clusters give rise to M_3C_2 frames which can adopt two different geometries. When the ligand is coordinated $(2\sigma + 1\pi)$ and the C_2 vector is parallel to a M–M edge the alkyne is designated $\mu_3\text{-}\eta^2\text{-}\|\text{-}$ [2] and *nido* [3], square pyramidal, 7 skeletal electron pairs (SEP), 5 vertexes structures are formed; these are notably the more common for both homo- and hetero-metallic clusters with alkyne substituents [4]. By contrast, when the alkyne or acetylide ligand, coordinated $(1\sigma + 2\pi)$, is perpendicular to a M–M edge, it is designated as $\mu_3\text{-}\eta^2\text{-}\perp\text{-}$ and gives rise to *closo*, trigonal bipyramidal, 6 SEP, 5 vertexes structures. These different arrangements are schematically represented in Fig. 1.

Different viewpoints can be adopted when describing the electronic situation of the above M_3C_2 clusters; thus the alkyne ligand may be viewed simply as one among the other ligands [5], behaving as a four-electron donor. This is generally

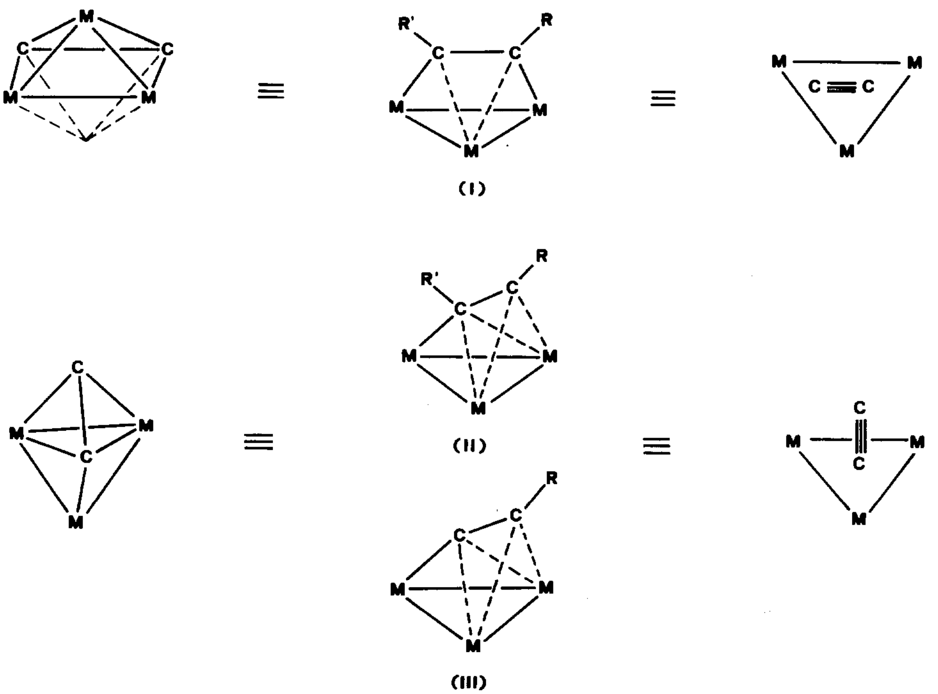

Fig. 1. Schematic representation of the structures of M_3C_2 cores obtained upon interaction of alkynes or acetylides with triangular metal clusters.

accepted for the *nido* clusters (I) (see Fig. 1), which may reach a 48 electron count by means of other ligands and of the cluster metals. Alternatively the alkynes or acetylides can be considered as skeletal constituents of the clusters, behaving as six- or five-electron donors respectively; indeed, in the Wade's theory, the CR groups and the C units contribute 3 or 2 electrons, respectively, to the bonding of the cluster frame [3]. On the latter picture the *closo* clusters II and III are 48 electron molecules; otherwise, these must be considered as 46 electron, unsaturated structures.

Recently, in some papers dealing with these complexes, the 46 electron hypothesis has been favoured [6], and electrochemical reduction of complexes II has been shown to be possible; surprisingly, however, no addition reactions have been attempted with clusters II or III in order to demonstrate their electronic unsaturation.

A report on the reactions of some clusters II or III with PPh_3 is given here; $Fe_3(CO)_9(\mu_3\text{-}\eta^2\text{-}\perp\text{-}EtC_2Et)$ (complex **1**) [7–9] undergoes substitution of CO instead of addition under relatively mild conditions, and high yields of the monosubstituted derivative $Fe_3(CO)_8(PPh_3)(\mu_3\text{-}\eta^2\text{-}\perp\text{-}EtC_2Et)$ (complex **1a**) are obtained. An improved synthesis of $(\eta^5\text{-}C_5H_5)NiFe_2(CO)_6(\mu_3\text{-}\eta^2\text{-}\perp\text{-}C_2Bu^t)$ (complex **2**) [10] allowed the study of its reactivity towards PPh_3; once again, high yields of the monosubstituted complex $(\eta^5\text{-}C_5H_5)NiFe_2(CO)_5(PPh_3)(\mu_3\text{-}\eta^2\text{-}\perp\text{-}C_2Bu^t)$ (complex **2a**) were obtained, without modification of the cluster-acetylide interactions.

Complexes **1a** and **2a** have been identified and characterized by analytical and spectroscopic techniques. The behaviour of clusters I–III with regard to the preference for addition or substitution reactions is discussed.

Experimental

$Fe_3(CO)_{12}$ was prepared by published reports [11]; $Fe_2(CO)_9$ (Strem Chemicals), $(\eta^5\text{-}C_5H_5)_2Ni_2(CO)_2$ (Pressure Chemicals), diethylacetylene, t-butylacetylene and triphenylphosphine (Fluka) were commercial products, used as received. The hydrocarbon solvents were distilled over sodium. Reactions were carried out under dry nitrogen. The reaction solutions, filtered under N_2, were taken to dryness in vacuo and the residues dissolved in CH_3Cl and subjected to TLC (Kieselgel P.F. Merck; diethyl ether and light petroleum as eluants). When possible, the products were crystallized from hexane or hexane/chloroform solutions kept under N_2; complex **1a** could be obtained only as microcrystalline powder.

The elemental analyses were carried out with a F&M 165 C, H, N analyzer and those for the metals and phosphorus by the F. Pascher laboratories (Bonn, W. Germany). The mass spectra (E.I. technique, 70 eV) were obtained on a Kratos MS-50. The IR spectra were recorded on a Perkin–Elmer 580B instrument; the ^1H, ^{13}C and ^{31}P NMR spectra were recorded on a JEOL JNM GX 270 FT spectrometer.

Synthesis of 1 and improved synthesis of 2. Complex **1** was obtained as already reported [8,9] (analysis and mass spectrum, see ref. 8 and 12 respectively). IR ν(CO) (C_6H_{14}): 2080m, 2036vs, 2028vs, 2008s, 1995m, 1991m cm^{-1}. ^1H NMR (δ(ppm), $CDCl_3$, 298 K): 3.60 q (2H, CH_2), 1.72 t (3H, CH_3), 1.63 q (2H, CH_2), 0.49 t (3H, CH_3).

The yields reported for the original synthesis of **2** were very low [10]. These can be improved as follows: $(Cp)_2Ni_2(CO)_2$ is refluxed in heptane under N_2 with a 1.5 to 1 molar excess of t-butyl-acetylene for 40 min, until the initial red colour of the solution turns dark green and the ν(CO) bond of the nickel complex in the IR spectra of the solution has disappeared. To the resulting solution, containing $(Cp)_2Ni_2(HC_2Bu^t)$, is added a 2 to 1 molar excess of $Fe_2(CO)_9$ (calculated on the starting nickel complex), and the reflux is continued for 20 min. Yields of 30–35% of **2** are obtained, after two TLC procedures to separate the product from $Fe_3(CO)_{12}$ and $(\eta^5\text{-}C_5H_5)_2Ni_2Fe_2(CO)_6(HC_2Bu^t)$ [13] also formed in the reaction. Analysis and mass spectrum of **2**, see ref. 10 and 14 respectively. IR ν(CO)(C_6H_{14}): 2062s, 2021vs, 2012m(sh), 1993vs, 1989s(sh), 1973m(b), 1962m(b) cm^{-1}. ^1H NMR (δ ppm, $CDCl_3$, 298 K): 5.24 s (5H, Cp), 1.56 s (9H, Bu^t).

Reactions of 1 and 2 with phosphines. Refluxing of a solution of **1** (100 mg, 0.20 mmol) and PPh_3 (100 mg, 0.45 mmol) in hexane under nitrogen for 20 min yields about 85% of **1a** together with five unidentified trace products and some decomposition. A similar procedure with PCy_3 gives moderate yields of products tentatively identified as the mono- and bi-substitution products of **1**; PPh_2H and $PPh_2(OEt)$ give low yields of bisubstitution products (tentative identification) and mostly decomposition.

Complex **1a**, brown-black, crystalline powder. Analysis. Found: C, 38.9; H, 5.21; Fe, 27.7; P, 5.40. $C_{32}Fe_3H_{28}PO_8$ calcd.: C, 39.08; H, 5.00; Fe, 28.68; P, 5.30%. The complex decomposes in the mass spectrometer. IR ν(CO) (C_6H_{14}): 2082m, 2079m, 2073m(sh), 2024vs, 2015s(sh), 1996s(sh), 1990vs, 1948s(b), cm^{-1}. ^1H NMR (δ (ppm), $CDCl_3$, 298 K): 7.47–7.43 m (15H, Ph), 2.98 q (2H, CH_2), 2.42 q (2H,

CH_2), 1.32 t (3H, CH_3), 1.20 t (3H, CH_3). ^{31}P NMR (δ(ppm), $CDCl_3$, 298 K: H_3PO_4): +68.38 s.

Refluxing of a solution of **2** (100 mg, ca. 0.20 mmol) and PPh_3 (100 mg, 0.45 mmole) in hexane, under N_2 for 20 min yields about 10% of $(Cp)_2Ni_2Fe_2(CO)_6$-(HC_2Bu^t) and about 70% of **2a**, together with small amounts of an unidentified product and some decomposition products. Complex **2a**, dark green-black crystals. Analysis. Found: C, 57.0; H, 4.20; Fe, 15.8; Ni, 8.10; P, 4.40%. $C_{43}Fe_2H_{29}NiPO_5$ calcd.: C, 56.79; H, 4.06; Fe, 15.35; Ni, 8.16; P, 4.31%. The complex decomposes in the mass spectrometer. IR ν(CO) (C_6H_{14}): 2072m, 2027vs, 2021vs, 1982vs, 1960s(sh,b), 1950vs(b) cm^{-1}. ^1H NMR (δ (ppm), $CDCl_3$, 298 K): 7.55–7.35 m (15H, Ph), 5.00 s (5H, Cp), 1.60 s (9H, Bu^t). ^{31}P NMR (δ (ppm), $CDCl_3$, 298 K: H_3PO_4): +74.17 s.

Results and discussion

Spectroscopic characterization and hypothesized structures of clusters 1a, 2a. The analytical data are consistent with the formulation of **1a** and **2a** as monosubstituted products. The IR spectra are similar to one another, and also show similarities with that of $HRu_3(CO)_8(PPh_3)(\mu_3\text{-}\eta^2\text{-}\perp\text{-}C_2Bu^t)$ [15]; CO-absorptions observed are due to terminal carbonyls only.

The chemical shifts and integrated intensities in the ^1H NMR also are consistent with the proposed formulation. For complexes **1** and **1a** two quadruplets and two triplets are observed for the ethyl substituents on the alkyne; we tentatively attribute the downfield signals (3.60 q, 1.72 t) to the Et on C(α) of **1**, by analogy with data for isostructural derivatives [6]. In the spectrum of **1a** the downfield signals are shifted to higher field (2.98 q, 1.32 t), whereas the other set of resonances is shifted downfield (1.63 q, 0.48 t for **1** against 2.42 q, 1.20 t for **1a**).

In the ^{31}P NMR spectra of **1a** and **2a** sharp singlets are observed at room temperature; these fall in the expected range for coordinated phosphines. The ^{13}C NMR spectra of **1**, **1a**, **2** and **2a** are listed in Table 1.

The singlets attributed to C(α) and C(β) in the parent complexes appear as doublets for **1a** and **2a**; the assignments have been made by comparison with established complexes. Typical values of ^{13}C chemical shifts for acetylenes and acetylides coordinated in $\mu_3\text{-}\eta^2\text{-}\perp$-fashion are listed in Table 2.

In the CO region of the ^{13}C NMR spectrum complex **1a** shows, at room temperature, two singlets each corresponding to three CO's and two doublets each corresponding to one CO; this indicates the presence of two groups of three equivalent CO's coordinated to two different metal atoms. Two other CO's apparently rigid, are located on the metal atom bearing the phosphine; the downfield position of the signals from them indicates that they are on the iron atom σ-bound to the alkyne [15,18,20,21]. Also, the values of J(P–C) (17.35 and 13.4 Hz) are comparable with that for the alkyne C(α) (12.1 Hz).

Complex **2** shows CO fluxionality at room temperature; for **2a** two singlets (3 and 2 carbonyls respectively) are observed, which indicates the presence of two groups of CO's on different metal atoms.

On the basis of the spectroscopic data, we propose for **1a** and **2a** the structures shown in Fig. 2.

Regiospecific substitution of a phosphine on the metal σ-bound to the acetylide

TABLE 1

PROTON DECOUPLED ^{13}C NMR SPECTRA [a] OF COMPLEXES **1**, **1a**, **2** AND **2a**

Attribution	**1** [9]	**1a**	**2**	**2a**
CH_3	15.4 s, 16.5 s	15.2 s, 17.2–18.6 d	33.80 s	33.82 s
CH_2	21.8 s, 39.5 s	25.0 s, 30.08–30.19 d	–	–
Cp	–	–	88.30 s	88.19 s
Ph	–	124.44–128.59 d, 130.46 s(b) 133.26–133.45 d	–	128.03–128.17 d, 129.9–129.97 d, 133.17–133.33 d
$C(\beta)$	106.8 s	133.8–134.4 d (J(P–C) 43 Hz)	141.0 s	135.79–136.43 d (J(P–C) 43 Hz)
CO	207.9 s [b] 212.2 s	210.97 s (3) [c], 213.77(3), 216.5–216.8 d(1) (J(P–C) 17.53 Hz) 221.3–221.4 d(1) [d] (J(P–C) 13.4 Hz)	212.0 m(b)	214.83 s (3), 219.56 s(b) (2)
$C(\alpha)$	221.9 s	222.3–222.5 d(1) [d] (J(P–C) 12.1 Hz)	192.0 s	219.37 d

[a] $CDCl_3$, 20°C, proton decoupled; ppm, downfield positive with respect to TMS. [b] CO (π). [c] In parentheses relative intensities. [d] Attribution can be reversed.

has been observed for $HRu_3(CO)_9(\mu_3\text{-}\eta^2\text{-}\perp\text{-}C_2Bu^t)$ [15] and is confirmed by the crystal structure of $HRu_3(CO)_8[PPh_2(OEt)]$ $(\mu_3\text{-}\eta^2\text{-}\perp\text{-}C_2Bu^t)$ [22]. The position of the phosphine in **2a**, where no reorientation of the acetylide with respect to the metals has occurred, has been confirmed in a preliminary X-ray study [23].

Some comments on the alkyne or acetylide reorientation upon substitution, nucleophilic addition or electrochemical reduction. Although replacement of CO by phosphine ligands is a well known process in metal carbonyl chemistry, only a limited number of examples of phosphine substitutions on hydrocarbyl clusters is available. When clusters I–III are considered, one observes that the *nido* derivatives I can be shown to have a different behaviour from the *closo* II and III; complexes I show fluxionality of the alkyne with respect of the metal core [24,25] and undergo

TABLE 2

^{13}C CHEMICAL SHIFTS FOR μ_3-η^2-$\perp$-ALKYNE OR -ACETYLIDE LIGANDS ON SELECTED EXAMPLES OF IRON AND RUTHENIUM DERIVATIVES

Complex	$C(\beta)$	$C(\alpha)$ [a,b]	References
$Fe_3(CO)_9(C_2Et_2)$	106.8 s	221.9 s	9
$Fe_3(CO)_8(PPh_3)(C_2Et_2)$	133.8–134.4 d (J(P–C) 43.0 Hz)	222.3–222.5 d (J(P–C) 12.1 Hz)	this work
$(Cp)_2W_2Fe(CO)_6(C_2(C_6H_4\text{-}Me\text{-}4)_2)$	153.8	168.5	16
$(Cp)WFe_2(CO)_8(C_2Me)$	108.3	165.7	17
$(Cp)NiFe_2(CO)_6(C_2Bu^t)$	141.0 s	192.0 s	this work
$(Cp)NiFe_2(CO)_5(PPh_3)(C_2Bu^t)$	135.8–136.4 d (J(P–C) 43.0 Hz)	219.4 d	this work
$HRu_3(CO)_9(C_2Bu^t)$	110.6 s	164.2 s	18
$HRu_3(CO)_8[PPh_2(OEt)](C_2Bu^t)$	112.2 s	165.7 s	19
$(PPh_2)Ru_3(CO)_9(C_2Ph)$	64.3 s	199.3 d (J(P–C) 33.4 Hz)	19

[a] $C(\alpha)$ is the ligand atom interested to the σ-bonding with the metal. [b] All values in ppm, downfield positive with respect of TMS.

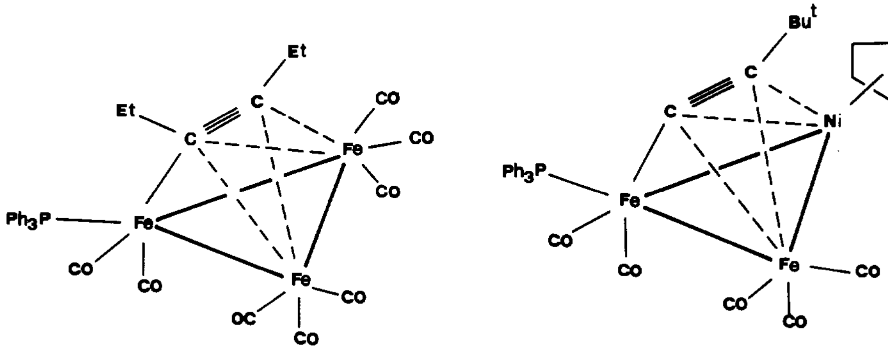

Fig. 2. Proposed structures for complexes **1a** and **2a**.

displacement of CO by phosphine under mild conditions, with reorientation of the alkyne vis-a-vis of the cluster edges. Examples are the heterometallic derivatives (Cp)NiFeCo(CO)$_5$(PPh$_3$)(C$_2$Ph$_2$) [24,26] and FeCo$_2$(CO)$_8$(PPh$_3$)(C$_2$Et$_2$) [27]. By contrast, complexes II and III are characterized by rigidity of the alkyne or acetylide with respect to the cluster, as shown by variable temperature NMR studies on Fe$_3$(CO)$_9$(MeC$_2$Ph) [6] which do not show free rotation below the decomposition temperature. This has been explained in terms of a higher energy barrier with respect of complexes I; only the latter have a vacancy on the "cluster surface" which can be associated with fluxionality [28].

On the other hand the acetylide complexes III show considerable nucleophilic reactivity at the acetylide C(α); this feature confirmed by UV-PES studies [9,29,30] leads to the formation of functionalized alkynes, parallel to one edge of the clusters, and hence to structures comparable with I [31]. The same happens when alkynes are obtained via reductive coupling of hydrides and acetylides [32] or via alkylidine-alkylidine coupling, as in the remarkable complex Fe$_3$(CO)$_9$(PPh$_3$)(MeCCOEt) [33] which is, at least formally, the unique product of addition to structure I. Finally carbon-carbon coupling upon electrochemical, one-electron reduction, lead to formation of the [Fe$_3$(CO)$_9$(MeCCO)]$^-$ anion with structure I [34]; electrochemical, two-electron reduction of cluster **1** gives the dianion [Fe$_3$(CO)$_9$(C$_2$Et)$_2$]$^{2-}$, also with structure I [6].

The above evidence clearly shows that the acetylide complexes III, at least, can readily accept further electronic density; however, neither **1** nor **2** undergo addition reactions in the presence of phosphines. Only substitution of CO occurs in good yields. A possible explanation for this apparent anomaly is that there are no vacant coordination sites on these *closo* structures, and in particular on the metal cores; the chemical and electrochemical reactivity of the complexes is essentially associated with the alkyne or acetylide ligands.

The behaviour of complex **2**, which shows unexpected metal-ligand interaction, accords with this hypothesis. Indeed, in **2**, the nickel atom π-bound to the acetylide is formally 19 electron and the iron σ-bound to the organic moiety is a 17 electron atom in the effective atom number formalism. Reorientation of the acetylide and formation of a σ-bond between nickel and acetylide would lead to a 18 electron count for all the metal atoms; however, complex **2** shows a remarkable stability and rigidity of the NiFe$_2$C$_2$ core. Studies on other homo- and hetero-metallic hydrocarbyl clusters are in progress.

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