

## REACTIVITY OF $[\text{HFe}_3(\text{CO})_{11}]^-$ TOWARD ALKYNES

### II \*. REACTIONS WITH MONOSUBSTITUTED ALKYNES: FROM ALKYLIDYNE TO ACETYLIDE LIGANDS ON A TRIIRON UNIT

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#### Summary

The reactions of monosubstituted alkynes  $\text{RC}\equiv\text{CH}$  ( $\text{R} = \text{C}_6\text{H}_5, \text{C}_3\text{H}_7, \text{C}(\text{O})\text{CH}_3$  or  $\text{C}(\text{O})\text{OCH}_3$ ) with  $[\text{P}(\text{C}_6\text{H}_5)_4][\text{HFe}_3(\text{CO})_{11}]$  have been studied. Depending on the reaction conditions the complexes  $[\text{P}(\text{C}_6\text{H}_5)_4][\text{Fe}_3(\text{CO})_{10}(\mu_3\text{-CCH}_2\text{R})]$  (**A**) and  $[\text{P}(\text{C}_6\text{H}_5)_4][\text{HFe}_3(\text{CO})_9(\mu_3\text{-C=CHR})]$  (**B**) can be isolated. The two types of complexes are interrelated by the addition (**B**  $\rightarrow$  **A**) or loss (**A**  $\rightarrow$  **B**) of carbon monoxide. In two cases ( $\text{R} = \text{C}_6\text{H}_5, \text{C}_3\text{H}_7$ ) the transformation **B**  $\rightarrow$   $[\text{P}(\text{C}_6\text{H}_5)_4][\text{Fe}_3(\text{CO})_9(\mu_3\text{-C}\equiv\text{CR})]$  has been observed in refluxing 2-methoxyethanol, and for these two alkynes all the transformations  $\text{RC}\equiv\text{CH} \xrightarrow{+\text{H}} \text{RCH}_2\text{C}\begin{smallmatrix} \diagup \\ \diagdown \end{smallmatrix} \xrightarrow{-\text{H}} \text{RCH}=\text{C}\begin{smallmatrix} \diagup \\ \diagdown \end{smallmatrix} \xrightarrow{-\text{H}} \text{RC}\equiv\text{C}-$  have been observed on a triiron unit.

#### Introduction

We showed recently that the cluster anion  $[\text{HFe}_3(\text{CO})_{11}]^-$  is very reactive toward disubstituted alkynes and acetylene [1,2]. Thus  $[\text{P}(\text{C}_6\text{H}_5)_4][\text{HFe}_3(\text{CO})_{11}]$  (**1**) reacts with acetylene at room temperature to give  $[\text{P}(\text{C}_6\text{H}_5)_4][\text{Fe}_3(\text{CO})_{10}(\mu_3\text{-CCH}_3)]$  (**2**) and  $[\text{P}(\text{C}_6\text{H}_5)_4][\text{Fe}_2(\text{CO})_6(\mu\text{-CO})(\mu\text{-CHCH}_2)]$  (**3**). To establish whether this ready transformation of acetylene to an alkylidyne ligand also takes place with monosubstituted alkynes we have extended the study of the reactions of the monosubstituted alkynes  $\text{RC}\equiv\text{CH}$  with  $\text{R} = \text{C}_6\text{H}_5, \text{C}_3\text{H}_7, \text{C}(\text{O})\text{CH}_3$  and  $\text{C}(\text{O})\text{OCH}_3$ , the substituents being chosen to cover a range of steric and electronic properties. In most cases the reactions observed at room temperature are, in fact, more complex than with acetylene, and we report here the results obtained at various temperatures.

\* For part I see ref. 2.

## Results and discussion

### Reactions at room temperature

The reactions of **1** with monosubstituted alkynes generally gave complex mixtures, as evidenced by infrared spectroscopy, and it was not possible to isolate any pure products. Only for  $R = C(O)CH_3$  was a single product (**4**) isolated in good yield and fully identified. Its infrared spectrum in the  $\nu(CO)$  stretching region closely resembles that of complex **2**, and the proton NMR spectrum shows in addition to the phenyl resonances of the cation) two singlets in 2/3 ratio at 5.58 and 2.45 ppm. The  $[P(C_6H_5)_4][Fe_3(CO)_{10}(CCH_2COCH_3)]$  formulation is consistent with the elemental analysis and the presence of an ethylidyne ligand  $\mu_3$ -bonded is firmly demonstrated by the  $^{13}C$  NMR spectrum (Table 1), in which there is a resonance for the  $C-CH_2COCH_3$  carbon at 280.6 ppm, in good agreement with the value found for **2** (289 ppm) [2]. In a solution of **4** the infrared spectrum shows, in addition to terminal carbonyl frequencies, two absorptions at 1760 and 1675  $cm^{-1}$  characteristic of  $\mu_2$ - and  $\mu_3$ -bridging carbonyl groups, and this indicates that, as for **2**, two forms are present in equilibrium in solution, and these are shown in Fig. 1. It should be noted that in the solid state spectrum of **4** there is only one absorption at 1735  $cm^{-1}$  for the bridging carbonyl group, and this favours a structure with a doubly bridging carbonyl group. (For **2** only the structure with a triply-bridging carbonyl group was observed in the solid state). In the light of these initial results, which indicated that at room temperature the reaction of **1** with monosubstituted alkynes was generally more complex than with acetylene, we have carried out the reactions at higher temperatures.

### Reactivity of **1** toward $RC\equiv CH$ in boiling acetone

Complex **1** reacts in boiling acetone with all the monosubstituted alkynes studied, to give the same type of product, as shown by infrared spectroscopy. For  $R = C_6H_5$  or  $C(O)OCH_3$ , boiling ethyl acetate was used in order to increase the rate of the reaction but this did not affect the nature of the products. The products isolated, **5a** ( $R = C_6H_5$ ), **5b** ( $R = CH_3(CH_2)_2$ ), **5c** ( $R = C(O)OCH_3$ ) and **5d** ( $R = C(O)CH_3$ ) have similar infrared spectra in the  $\nu(CO)$  stretching region; there is the expected shift towards lower frequencies with increase in the electron donating power of the R groups (Table 1). These infrared spectra closely resemble that of  $[P(C_6H_5)_4][HFe_3(CO)_3(CCH_2)]$  (**6**) the product of thermal rearrangement of **2** [2].

The  $^1H$  and  $^{13}C$  NMR data (Table 1) for the compounds **5** confirm this

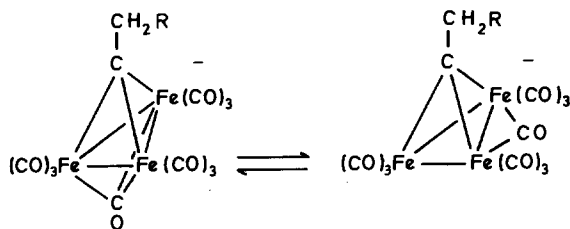


Fig. 1. Structures of complexes **4** and **7a-7d** (a,  $R = C_6H_5$ ; b,  $R = CH_3(CH_2)_2$ ; c,  $R = C(O)-OCH_3$ ; d,  $R = C(O)CH_3$ ) in solution.

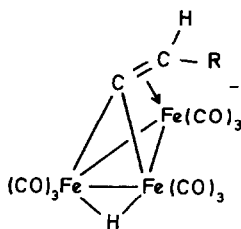


Fig. 2. Structure of complexes **5a–5d** (a, R = C<sub>6</sub>H<sub>5</sub>; b, R = CH<sub>3</sub>(CH<sub>2</sub>)<sub>2</sub>; c, R = C(O)OCH<sub>3</sub>; d, R = C(O)CH<sub>3</sub>).

similarity. Except in the case of **5a**, two doublets are observed in the <sup>1</sup>H NMR spectra (Table 1), one near –17 ppm characteristic of a hydride ligand and one at lower field near 5.6 ppm. These doublets have the same intensity, and comparison with the signals of the R groups shows that each represents one hydrogen. This suggests the formulation [P(C<sub>6</sub>H<sub>5</sub>)<sub>4</sub>][HFe<sub>3</sub>(CO)<sub>9</sub>CCHR], which is consistent with the chemical analysis and is confirmed by the <sup>13</sup>C NMR spectra, in which resonances characteristic of μ<sub>3</sub>-η<sup>2</sup>-bonded vinylidene ligands, CCHR, are observed between 80.3 and 97.3 ppm (coupled with one hydrogen for the CHR group) and between 278 and 294 ppm for the carbon bonded to two metallic centres [3]. By analogy with **2** we propose the structure shown in Fig. 2, in which the hydride ligand is located between the Fe(2) and Fe(3) atoms. Examination of the mother solution after crystallisation of complex **5** by infrared spectroscopy in the ν(CO) stretching region suggests the presence of [P(C<sub>6</sub>H<sub>5</sub>)<sub>4</sub>][Fe<sub>2</sub>(CO)<sub>6</sub>(μ-CO)(μ-CHCHR)] complexes [1], but these have not been isolated.

In summary, in boiling acetone **1** reacts with monosubstituted alkynes to give as the main products the [P(C<sub>6</sub>H<sub>5</sub>)<sub>4</sub>][HFe<sub>3</sub>(CO)<sub>9</sub>(CCHR)] complexes containing a μ<sub>3</sub>-η<sup>2</sup>-bonded vinylidene ligand. By comparison with the results observed for the reaction of **1** with acetylene [1,2] this suggests that the first step of the reaction is the formation of [P(C<sub>6</sub>H<sub>5</sub>)<sub>4</sub>][Fe<sub>3</sub>(CO)<sub>10</sub>CCH<sub>2</sub>R], which then rearranges to **5a–5d** in boiling acetone.

As [P(C<sub>6</sub>H<sub>5</sub>)<sub>4</sub>][HFe<sub>3</sub>(CO)<sub>9</sub>(CCH<sub>2</sub>)] (**6**) quickly rearranges to [P(C<sub>6</sub>H<sub>5</sub>)<sub>4</sub>][Fe<sub>3</sub>(CO)<sub>10</sub>(CCH<sub>3</sub>)] (**2**) under the action of carbon monoxide at atmospheric pressure we examined the reactions of complexes **5a–5d** with carbon monoxide.

#### Reactions of complexes **5a–5b** with carbon monoxide

In contrast to the result with **6**, there is no apparent reaction between complexes **5a–5d** and carbon monoxide at atmospheric pressure. However, under 20 atm of carbon monoxide the complexes **5a–5d** are slowly converted into new compounds (**7a–7d**), which were isolated by crystallization. The infrared spectra in the ν(CO) stretching region closely resemble that of **2**, and moreover **7d** has the same infrared spectrum as **4**. This indicates that as for **6**, the addition of one molecule of carbon monoxide induces the transformation of the vinylidene ligand CCHR into the alkylidyne ligand CCH<sub>2</sub>R. Chemical analyses are consistent with the [P(C<sub>6</sub>H<sub>5</sub>)<sub>4</sub>]-Fe<sub>3</sub>(CO)<sub>10</sub>(CCH<sub>2</sub>R)] formulation, and the <sup>1</sup>H and <sup>13</sup>C NMR spectra confirm the presence of the CCH<sub>2</sub> group, in particular resonances characteristic of the carbon bonded to the three iron atoms [2,3] are present in the 280–310 ppm region (Table 1). Complexes **7a–7c** show the same behaviour as that of **2** and **4**. In solution there

TABLE 1  
SPECTROSCOPIC DATA FOR THE ISOLATED COMPLEXES<sup>a</sup>

Compound	IR, $\nu(\text{CO})$ ( $\text{cm}^{-1}$ )	<sup>1</sup> H NMR <sup>b</sup> $\delta$ (ppm)	<sup>13</sup> C NMR <sup>b</sup> $\delta$ (ppm)
<b>5a</b>	2047m, 2005s, 1980s, 1960m	-17.1 d ( <i>J</i> 2.8 Hz)	278, 215.3, 97.3
<b>5b</b>	2045m, 2000s, 1975s, 1955m	5.59 dt ( <i>J</i> 2.8 Hz; <i>J</i> 5.8 Hz) 2.15 m, 1.66 m, 1.02 t ( <i>J</i> 7.3 Hz) -17.3 d ( <i>J</i> 2.8 Hz)	280.4, 216.1, 100.1, 43.8, 25.6, 13.8
<b>5c</b>	2055m, 2010s, 1990s, 1965m	5.69 d ( <i>J</i> 1.5 Hz), 3.73, -17.1 d ( <i>J</i> 1.5 Hz)	294.0, 215.8, 171.5, 80.3, 50.8
<b>5d</b>	2055m, 2010s, 1985s, 1965m	5.84 d ( <i>J</i> 2.9 Hz), 2.24, -17.0 d ( <i>J</i> 2.9 Hz)	293.0, 216.3, 199.9, 89.9, 29.2
<b>7a</b>	2053w, 2000vs, 1985sh, 1965s, 1755w, 1660w; 1675 in the solid state	5.95	298.8, 220.9, 60.9
<b>7b</b>	2050w, 1995vs, 1962s 1935sh, 1760w, 1665w; 1770 in the solid state		
<b>7c</b>	2055w, 2000vs, 1967s, 1940sh, 1770vw, 1715w, 1665w; 1735, 1710sh in the solid state		309.0, 221.3, 57.7, 40.0, 23.4, 14.2
<b>4 (7d)</b>	2052w, 1998vs, 1965s, 1935sh, 1760w, 1720w, 1675w, 1735; 1715sh in the solid state	5.21, 3.70	280.1, 220.8, 172.3, 58.7, 51.1
<b>8a</b>	2045m, 1990s, 1980s, 1960m, 1935m	5.58, 2.45	
<b>8b</b>	2042m, 1983s, 1975s, 1953m, 1938m	- 3.07t ( <i>J</i> 6.9 Hz), 1.95m, 1.23t ( <i>J</i> 7.1 Hz)	216.9, 216.4, 194.9, 101.9 217.1, 216.9, 186.3, 104.8, 34.5, 26.3, 13.7

<sup>a</sup> For compounds **a**, **R** = C<sub>6</sub>H<sub>5</sub>; **b**, **R** = C<sub>3</sub>H<sub>7</sub>; **c**, **R** = C(O)CH<sub>3</sub>; **d**, **R** = C(O)OCH<sub>3</sub>. <sup>b</sup> Except phenyl resonances.

is an equilibrium between two forms (Fig. 1) containing a bridging carbonyl ligand,  $\mu_2$ - or  $\mu_3$ -bonded, as evidenced by the infrared spectrum in the  $\nu(\text{CO})$  stretching region (Table 1). However, in the solid state the situation is quite different: **7a** contains a  $\mu_3$ -bridging carbonyl ligand, while **7b**, **7c**, contain a  $\mu_2$ -bridging carbonyl ligand in the solid state.

In summary the vinylidene complexes **5a–5d** react with carbon monoxide to give the alkylidyne compounds  $[\text{P}(\text{C}_6\text{H}_5)_4][\text{Fe}_3(\text{CO})_{10}\text{CCH}_2\text{R}]$ , but under more forcing conditions than for **6** since 20 atm of CO are necessary. The reason for this lower reactivity of complexes **5a–5d** is not clear; the greatest difference is the steric hindrance by the R groups, which perhaps interferes with the approach of a CO ligand to the iron atoms.

We have confirmed that the transformation is reversible and by showing that complexes **7a–7d** revert to compounds **5a–5d** in boiling acetone.

This fairly easy carbon-hydrogen activation resulting in conversion of an alkylidyne ligand into a vinylidene ligand, suggested that under more forcing conditions vinylidene ligands might be converted to acetylidyne ligands. For this reason we have studied the behaviour of complexes **5a–5d** in a solvent with a higher boiling point.

#### *Rearrangement of complexes 5a–5d in boiling 2-methoxyethanol*

Solution of complexes **5a–5d** and **6** in 2-methoxyethanol were refluxed. Except for **5a** and **5b** this leads only to decomposition of the compounds. With **5a** and **5b** the similar complexes **8a** and **8b** are isolated as shown by infrared spectroscopy in the  $\nu(\text{CO})$  stretching region (Table 1). Furthermore these spectra are very similar to that of  $[\text{P}(\text{C}_6\text{H}_5)_4][\text{Fe}_3(\text{CO})_9(\mu_3\text{-C}\equiv\text{CCH}_3)]$ , which was obtained by reduction of  $\text{Fe}_3(\text{CO})_9(\mu_3\text{-CCH}_3)(\mu_3\text{-COC}_2\text{H}_5)$  [4] (2044m, 1982s, 1976s, 1955s, 1930m  $\text{cm}^{-1}$ ). This similarity is confirmed by the  $^1\text{H}$  NMR spectrum, which shows only the signals from the  $\text{P}(\text{C}_6\text{H}_5)_4$  cation and the R groups. The  $^{13}\text{C}$  NMR spectra are also typical of  $[\text{P}(\text{C}_6\text{H}_5)_4][\text{Fe}_3(\text{CO})_9(\mu_3\text{-C}\equiv\text{CR})]$  complexes, showing two resonances in the 2/1 ratio for the carbonyl ligands and resonances characteristic of the  $\text{C}_\alpha$  and  $\text{C}_\beta$  carbons of the  $\mu_3$ -bonded acetylidyne ligands [4,5] (Table 1). Complex **8b** has since been obtained in the reaction of  $\text{C}_3\text{H}_7\text{C}\equiv\text{CH}$  with  $[\text{HFe}_4(\text{CO})_{13}]^-$  [6]. Chemical analyses are consistent with the proposed formulations, and the structure proposed is shown in Fig. 3. Such a structure has recently been established by single crystal X-ray diffraction for  $[\text{Fe}_3(\text{CO})_9\text{CCOC}(\text{O})\text{CH}_3]^-$  which shows very similar spectroscopic characteristics [7]. It thus appears that in refluxing 2-methoxyethanol there is carbon-hydrogen activation of the CH group of the vinylidene ligand but the hydride ligands are not retained and the isolated complexes are formed from **5a** and **5b** by elimination of molecular hydrogen. This is consistent with the weakness of iron–hydrogen bonds compared with iron–carbon monoxide bonds.

That **5c**, **5d** and **6** do not give complexes of type **8** can be attributed to the fact

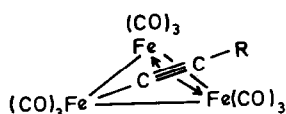
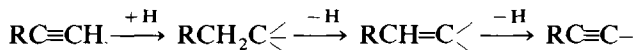


Fig. 3. Structure of complexes **8a** ( $\text{R} = \text{C}_6\text{H}_5$ ) and **8b** ( $\text{R} = \text{CH}_3(\text{CH}_2)_2$ ).

that these compounds are not thermally stable under the conditions necessary for CH activation. (We have synthesized  $[\text{P}(\text{C}_6\text{H}_5)_4][\text{Fe}_3(\text{CO})_9(\mu_3\text{-C}\equiv\text{CH})]$  independently [8].

To conclude, the study of the reactions of  $[\text{P}(\text{C}_6\text{H}_5)_4][\text{HFe}_3(\text{CO})_{11}]$  with mono-substituted alkynes has provided evidence for the transformation on a trinuclear iron unit:



Although such steps were well known for reactions of clusters of ruthenium or osmium [9,10] with alkynes, this is the first time that they have been observed with a triiron cluster unit.

## Experimental

All reactions were performed under nitrogen using standard Schlenk tube techniques. Infrared spectra were recorded with a Perkin-Elmer 225 spectrometer using dichloromethane solutions or KBr pellets.  $^1\text{H}$  NMR spectra were recorded with a Bruker WH90 spectrometer and  $^{13}\text{C}$  NMR spectra with a Bruker WM250 instrument.

$[\text{P}(\text{C}_6\text{H}_5)_4][\text{HFe}_3(\text{CO})_{11}]$  was prepared by exchange of the cation of  $[\text{N}(\text{C}_2\text{H}_5)_3\text{-H}][\text{HFe}_3(\text{CO})_{11}]$  [11] with  $\text{P}(\text{C}_6\text{H}_5)_4\text{Br}$ .

Microanalyses were performed in our laboratory.

### Preparation of **4**

A solution of **1** (1 g) and  $\text{CH}_3\text{C}(\text{O})\text{CCH}$  ( $0.110\text{ cm}^3$ ) in acetone ( $30\text{ cm}^3$ ) was stirred at room temperature for 2 h, then evaporated to dryness in vacuo. The residue was dissolved in a small amount of methanol, and the solution cooled at  $-20^\circ\text{C}$  to give 0.4 g (40% yield) of **4** as dark brown crystals.

Anal. Found: C, 52.18; H, 2.95.  $\text{C}_{38}\text{H}_{25}\text{O}_{11}\text{PFe}_3$  calcd.: C, 53.27; H, 2.92%.

### Preparation of **5a-5d**

A solution of **1** (1 g) and the stoichiometric quantity of the alkyne  $\text{RC}\equiv\text{CH}$  in ethyl acetate ( $\text{R} = \text{C}_6\text{H}_5$  or  $\text{CH}_3\text{OC}(\text{O})$ ) or acetone ( $\text{R} = \text{C}_3\text{H}_7$  or  $\text{CH}_3\text{C}(\text{O})$ ) was refluxed for 2 h. Evaporation of the solvent and crystallization at  $-20^\circ\text{C}$  in methanol afforded:

**5a** (0.38 g, 35% yield) as dark brown crystals. Anal. Found: C, 56.64; H, 3.20.  $\text{C}_{41}\text{H}_{27}\text{O}_9\text{PFe}_3$  calc.: C, 57.07; H, 3.13%.

**5b** (0.43 g, 42% yield) as dark red crystals. Anal. Found: C, 54.75; H, 3.40.  $\text{C}_{38}\text{H}_{29}\text{O}_9\text{PFe}_3$  calcd.: C, 55.07; H, 3.50%.

**5c** (0.47 g, 45% yield) as dark red crystals. Anal. Found: C, 51.40; H, 3.0.  $\text{C}_{37}\text{H}_{25}\text{O}_{11}\text{PFe}_3$  calcd.: C, 52.60; H, 3.0%.

**5d** (0.25 g, 25% yield) as dark brown crystals. Anal. Found: C, 52.65; H, 3.08.  $\text{C}_{37}\text{H}_{25}\text{O}_{10}\text{PFe}_3$  calcd.: C, 53.62; H, 3.01%.

### Preparation of **7a-7d**

A solution of the appropriate complexes **5a-5d** (0.5 g) were dissolved in dichloromethane ( $10\text{ cm}^3$ ) was placed in a stainless steel autoclave pressurized to 20 atm of

carbon monoxide, and stirred for 24 h. After depressurization the solution was evaporated to dryness. Crystallization from a small amount of methanol at  $-20^{\circ}\text{C}$  gave nearly quantitative yields of:

**7a** as brown crystals. Anal. Found: C, 57.03; H, 2.97.  $\text{C}_{24}\text{H}_{27}\text{O}_{10}\text{PFe}_3$  calcd.: C, 56.63; H, 3.03%.

**7b** as brown crystals. Anal. Found: C, 54.47; H, 3.05.  $\text{C}_{39}\text{H}_{29}\text{O}_{10}\text{PFe}_3$  calcd.: C, 54.67; H, 3.38%.

**7c** as brown crystals. Anal. Found: C, 52.12; H, 2.81.  $\text{C}_{38}\text{H}_{25}\text{O}_{12}\text{PFe}_3$  calcd.: C, 52.29; H, 2.86%.

**7d** as brown crystals. Anal. Found: C, 52.18; H, 2.95.  $\text{C}_{38}\text{H}_{25}\text{O}_{11}\text{PFe}_3$  calcd.: C, 53.27; H, 2.92%.

#### *Preparation of 8a and 8b*

A solution of **5a** or **5b** (0.5 g) was refluxed in 2-methoxyethanol for 18 h, then cooled, filtered, and evaporated to dryness in vacuo. Dissolution of the residue in methanol and cooling to  $-20^{\circ}\text{C}$  afforded:

**8a** (0.25 g, 50% yield) as red crystals. Anal. Found: C, 56.78; H, 3.0.  $\text{C}_{41}\text{H}_{25}\text{O}_9\text{PFe}_3$  calcd.: C, 57.20; H, 2.90%.

**8b** (0.10 g, 25% yield) as dark red crystals. Anal. Found: C, 54.91; H, 3.33.  $\text{C}_{38}\text{H}_{27}\text{O}_9\text{PFe}_3$  calcd.: C, 55.20; H, 3.26%.

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