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Synthesis and properties of $\eta^5\text{-C}_5\text{R}_5\text{Fe}(\text{CO})_2(\text{CH}_2)_3\text{Si}(\text{OCH}_3)_3$ ($\text{R} = \text{H}$ or Me). An example of electrooxidative-induced carbonyl insertion

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Abstract

Propyltrimethoxysilane compounds of the type $\eta^5\text{-C}_5\text{R}_5\text{Fe}(\text{CO})_2(\text{CH}_2)_3\text{Si}(\text{OCH}_3)_3$ ($\text{R} = \text{H}$ (**3**); $\text{R} = \text{Me}$ (**4**)), can be conveniently prepared by reaction between the anionic complexes $\text{M}^+[\eta^5\text{-C}_5\text{R}_5\text{Fe}(\text{CO})_2]^-$ ($\text{M}^+ = \text{K}^+$ or Na^+ , $\text{R} = \text{H}$ (**1**); $\text{M}^+ = \text{Na}^+$; $\text{R} = \text{Me}$ (**2**)) and $\text{Cl}(\text{CH}_2)_3\text{Si}(\text{OCH}_3)_3$ in THF. Triphenylphosphine reacts with complex **3** in refluxing acetonitrile, to give the carbonyl insertion product $\eta^5\text{-C}_5\text{H}_5(\text{CO})(\text{PPh}_3)\text{FeC}(\text{O})(\text{CH}_2)_3\text{Si}(\text{OCH}_3)_3$ (**5**). Complex **4** behaves in a very different way in carbon monoxide insertion induced by triphenylphosphine. The oxidations of complexes **3** and **4** in acetonitrile have been studied by a combination of electrochemical and spectroscopic techniques. The results indicate migratory insertion oxidation of complexes **3** and **4**, which is very rapid at room temperature and seems to be accompanied by nucleophilic attack by the solvent. If the reactions are carried out in the presence of triphenylphosphine, the carbon monoxide-insertion reaction is induced by this phosphorus ligand only in the case of complex **3**.

Introduction

We previously reported the synthesis and characterization of some organometallic complexes containing alkoxy-silane- and siloxane-functionalized cyclopentadienyl ligands [1,2]. We have now turned to the prepared new complexes in which the alkoxy-silane or siloxane groups are directly bonded to a metal atom, in order to compare the stabilities and reactivities of these two types of compounds. The

hydrolyzable alkoxy-silane-substituted organometallic complexes should also be suitable moieties for attachment to inorganic or organic support materials for organometallic catalysis [3–6].

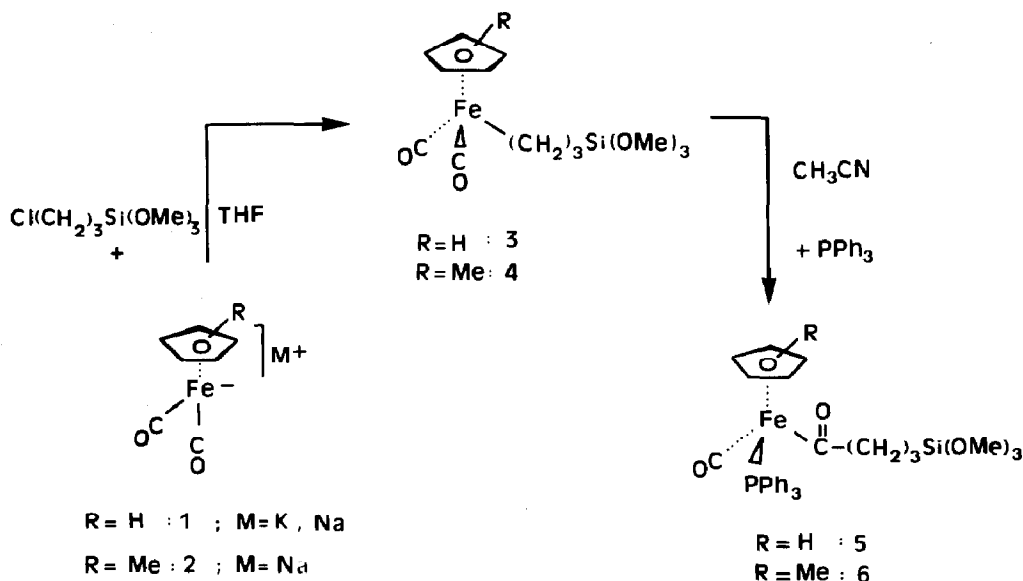
The importance of carbonyl alkyl-organoiron complexes in mechanistic and synthetic organometallic chemistry is well known, and CO insertion into metal–carbon(alkyl) bonds is recognised as an important process in catalytic chemistry [7]. Transition metal acyl complexes are the principal intermediates in a large number of catalytic processes, including hydroformylation of olefins and carbonylation of methanol or organic halides.

In the light of these considerations we decided to study the reactions of 3-chloropropyltrimethoxysilane with the anionic complex $M^+ [\eta^5-C_5H_5Fe(CO)_2]^-$ ($M^+ = K^+$ or Na^+), and its permethylated analogue $Na^+ [\eta^5-C_5Me_5Fe(CO)_2]^-$. The complexes $\eta^5-C_5R_5Fe(CO)_2(CH_2)_3Si(OCH_3)_3$ obtained show very different reactivities towards carbon monoxide insertion induced by triphenylphosphine in polar solvents. We present here details of the preparation, characterization, and properties of the new complexes and their electrochemical behaviour.

Results and discussion

Reactions of 3-chloropropyltrimethoxysilane, $(CH_3O)_3Si(CH_2)_3Cl$, with the sodium or potassium salt of the anions $[\eta^5-C_5R_5Fe(CO)_2]^-$ ($R = H$ (1); $R = Me$ (2)) in THF give the complexes $\eta^5-C_5R_5Fe(CO)_2(CH_2)_3Si(OCH_3)_3$, ($R = H$ (3); $R = Me$ (4)) (Scheme 1).

A THF solution of the sodium salt $Na^+ [\eta^5-C_5H_5Fe(CO)_2]^-$ (1), generated from $[\eta^5-C_5H_5Fe(CO)_2]_2$ and a sodium dispersion as the reducing agent [8], can be employed in situ, for the preparation of complex 3. Alternatively, this reaction can be brought about by using the isolated potassium salt $K^+ [\eta^5-C_5H_5Fe(CO)_2]^-$



Scheme 1

Table 1
Analytical and physical data for the complexes

Complex	Colour	Analysis (Found (calcd.) (%))			IR carbonyl bands ^b	
		C	H	Fe	$\nu(\text{CO})$	$\nu(\text{C(O)})$
$[(\eta^5\text{-C}_5\text{H}_5)\text{Fe}(\text{CO})_2(\text{CH}_2)_3\text{Si}(\text{OCH}_3)_3]$ (3)	orange- yellow	45.8 (45.4)	5.5 (5.8)	16.8 (16.3)	1999vs, 1939vs	
$[(\eta^5\text{-C}_5\text{Me}_5)\text{Fe}(\text{CO})_2(\text{CH}_2)_3\text{Si}(\text{OCH}_3)_3]$ (4)	orange- yellow	52.7 (52.3)	7.0 (7.3)	13.9 (13.5)	1977vs, 1917vs	
$[(\eta^5\text{-C}_5\text{H}_5)(\text{CO})(\text{PPh}_3)\text{FeC}(\text{O})(\text{CH}_2)_3\text{Si}(\text{OCH}_3)_3]$ (5)	yellow	61.9 (61.4)	5.1 (5.8)	9.7 (9.2)	1911vs	1608s
$[(\eta^5\text{-C}_5\text{Me}_5)(\text{CO})(\text{PPh}_3)\text{FeC}(\text{O})(\text{CH}_2)_3\text{Si}(\text{OCH}_3)_3]$ (6)	– ^a	–	–	–	1892vs	1585s

^a Not isolated. The presence of this compound was detected by IR spectroscopy. ^b Data in cm^{-1} . Values for acetonitrile solutions.

prepared from potassium benzophenone ketyl [9], which cleanly reduces the cyclopentadienyldicarbonyliron dimer in THF at room temperature.

The preparation of the sodium salt $\text{Na}^+[\eta^5\text{-C}_5\text{Me}_5\text{Fe}(\text{CO})_2]^-$ can be accomplished by reductive cleavage of $[\eta^5\text{-C}_5\text{Me}_5\text{Fe}(\text{CO})_2]_2$, in THF solution with the commercially available sodium dispersion. This method has been used for the analogue cyclopentadienyl $[\eta^5\text{-C}_5\text{H}_5\text{Fe}(\text{CO})_2]_2$ [8], but until now only potassium or sodium salt of the anion $[\eta^5\text{-C}_5\text{Me}_5\text{Fe}(\text{CO})_2]^-$, obtained using a K mirror [10], Na/K alloy [11], or Na/Hg amalgam [12], has been reported. We have found that treatment of $[\eta^5\text{-C}_5\text{Me}_5\text{Fe}(\text{CO})_2]_2$ with an excess of sodium dispersion in refluxing THF for 10 h results complete conversion of the initial permethyl dimer into the anionic complex **2**, as shown by IR monitoring of THF solution. This reaction provides a convenient and easy method of preparing the orange THF solution of **2**, which can be used "in situ" as precursor for the propyltrimethoxysilane complex **4**.

Addition of $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{Cl}$ to a cooled THF solutions of the anionic complex $\text{M}^+[\eta^5\text{-C}_5\text{R}_5\text{Fe}(\text{CO})_2]^-$ ($\text{M} = \text{K}$ or Na for $\text{R} = \text{H}$; $\text{M} = \text{Na}$ for $\text{R} = \text{Me}$) caused an immediate and exothermic reaction, accompanied by a change in the colour of the solution from orange to brown, and separation of NaCl or KCl. After filtration, and column chromatography the products were isolated as orange oils and identified by IR and ^1H NMR spectroscopy as the complexes $\eta^5\text{-C}_5\text{R}_5\text{Fe}(\text{CO})_2\text{-(CH}_2)_3\text{Si}(\text{OCH}_3)_3$, ($\text{R} = \text{H}$ (**3**); $\text{R} = \text{Me}$ (**4**)).

The values of $\nu(\text{CO})$ in Table 1 show a significant decrease on going from complex **3** containing C_5H_5 , to complex **4** containing C_5Me_5 , as consequence of the higher electron donor ability of the C_5Me_5 ligand.

Complex **3** reacted with triphenylphosphine in refluxing acetonitrile, by carbonyl insertion into the bond between the iron atom and the propyltrimethoxysilane group, to give the acyl derivative $(\eta^5\text{-C}_5\text{H}_5)(\text{CO})(\text{PPh}_3)\text{FeC}(\text{O})(\text{CH}_2)_3\text{Si}(\text{OCH}_3)_3$ (**5**), which after column chromatography was obtained as a stable yellow crystalline solid. The IR spectrum of **5** exhibits the expected terminal carbonyl band, and in addition a strong and broad band at 1608 cm^{-1} (see Table 1) attributable to the carbonyl of the acyl group in the $(\text{CH}_2)_3\text{C}(\text{O})\text{Fe}$ entity, these data agree with those found for similar cyclopentadienyliron complexes containing acyl groups [13–15].

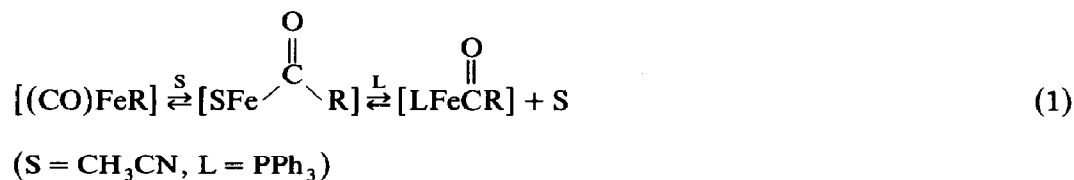
The rate of the reaction was found to be solvent dependent; reaction of **3** with PPh_3 in refluxing THF also gave the insertion product **5**, but about three times as slowly as in acetonitrile. Similar results were observed by Green et al. [15] during kinetic investigations of the reactions of alkyldicarbonylcyclopentadienyliron complexes with some phosphorus ligands. The choice of solvent often has an important influence on the rates of alkyl-CO migratory insertion reactions [16], but the solvents effects are still poorly understood.

Of interest are the different reactivities of complexes **3**, **4** and **5** towards silica surfaces. Complexes **3** and **4** can be readily purified via column chromatography on silica, but attempts to purify the acyl derivative **5** in this way failed because it attached to the column, and it is necessary to use silica pretreated with Me_3SiCl in C_6H_6 or commercially available silanised silica.

This difference in behaviour of the complexes **3** and **4** and the carbonyl insertion product **5** may be due to the different polarities and lengths of the silane chain bonded to the iron atom, since these factors are known to affect the behaviour of metal complexes towards support surfaces [17].

We found that the behaviour of the permethyl complex $\eta^5\text{-C}_5\text{Me}_5\text{Fe}(\text{CO})_2\text{-(CH}_2)_3\text{Si}(\text{OCH}_3)_3$ (**4**) in the carbon monoxide insertion induced by triphenylphosphine in acetonitrile was very different from that previously described for the unsubstituted cyclopentadienyl analogous complex **3**. We attempted to synthesize the acyl derivative of **4** under the conditions used for **3**, but after 30 h of treatment of **4** with PPh_3 in refluxing acetonitrile, only partial carbon monoxide insertion was detected by IR monitoring involving the decrease in $\nu(\text{CO})$ from 1977 to 1917 cm^{-1} for **4**, and the appearance of new medium bands at 1892 and 1585 cm^{-1} attributable to the carbonyl insertion product **6**. Attempts to make pure $(\eta^5\text{-C}_5\text{Me}_5)(\text{CO})(\text{PPh}_3)\text{-FeC}(\text{O})(\text{CH}_2)_3\text{Si}(\text{OCH}_3)_3$ (**6**) even under forcing conditions (long reaction times and use of an excess of triphenylphosphine ligand were unsuccessful, and only a mixture of complexes **4** and **6** was obtained, and the components could not be separated.

The difference in reactivity of complexes **3** and **4**, towards carbon monoxide insertion induced by triphenylphosphine in acetonitrile can be accounted for in terms of the mechanism of the reaction, which can be schematically represented as in eq. 1 [18].



Owing to the presence of the pentamethylcyclopentadienyl ring, the metal centre in **4** is more basic than that in **3**, as is confirmed by the results of the electrochemical studies, and so **4** is less susceptible to the nucleophilic attack, involved in the mechanisms proposed for carbon monoxide insertion into the iron-carbon bonds [19,20]. On the other hand, steric effects probably also operate, because the value of the cone angle of the $\eta^5\text{-C}_5\text{Me}_5$ ligand (θ 142°) [21] is substantially greater than in the unsubstituted cyclopentadienyl ring ($\eta^5\text{-C}_5\text{H}_5$, θ 110°), so that the replacement of the solvent molecule in the second step of reaction 1 by the PPh_3 ligand is inhibited by the steric hindrance which arises when the effective size of the

intermediate acyl species exceeds a critical value, and consequently no CO insertion into Fe–C bond is observed. We attempted the CO insertion reaction with complex **4** with the smaller phosphine, PBu_3 , but no insertion product was observed during several days in refluxing CH_3CN or THF solvents. Similar results were previously obtained for some carbonylpentamethylcyclopentadienylmolybdenum complexes, for which reaction has been observed only with very small phosphine ligands [18a]. An example of CO insertion into alkyl-organoiron complexes containing the pentamethylcyclopentadienyl ligand was recently reported [20]. Thus, whereas $\eta\text{-C}_5\text{H}_5\text{Fe}(\text{CO})_2\text{CH}_3$ undergoes an alkyl-CO migratory insertion reaction when either PPh_3 or CO is used as the incoming ligand, with the related complex $\eta\text{-C}_5\text{Me}_5\text{Fe}(\text{CO})_2\text{CH}_3$ it is necessary to use a raised CO pressure (80 psig of CO) and $\text{CF}_3\text{CH}_2\text{OH}$ as solvent.

Electrochemical studies

The oxidative electrochemistry of the synthesized compounds was investigated in acetonitrile with $[\text{N-n-Bu}_4][\text{PF}_6]$ as supported electrolyte. Electrochemical parameters of the complexes are summarized in Table 2. The room-temperature cyclic voltammograms of complexes **3** and **4** show an anodic irreversible wave *A* at scan rates as high as 5 Vs^{-1} . Following this anodic wave, a reversible couple *B/B'*, not present in the initial scan, appears (Fig. 1) with a peak separation ΔE_p of 78 mV, and a ratio of cathodic to anodic currents i_{p_a}/i_{p_c} of 1 for **3** and < 1 for **4**. At either lower temperatures (-30°C), and/or higher sweep rates the process becomes reversible, the *A/A'* being $i_{p_c}/i_{p_a} = 1$ and ΔE ca. 76 mV.

These electrochemical processes are diffusion controlled, with the anodic current function $i_p/v^{1/2}$ independent of the scan rate (*v*) over the range $0.1\text{--}1 \text{ Vs}^{-1}$. Potentiostatic coulometry and comparisons of limiting currents obtained from rotating disk electrode (RDE) voltammograms for **3** and **4** with those for ferrocene indicate an one-electron exchange in the first oxidation step *A*. As shown in Table 2, the greater donor ability of the pentamethylcyclopentadienyl ring results in a shift of the anodic peaks to less positive potentials.

When the complexes were electrochemically oxidized (at $E_p > E_{p_A}$) in situ in the cavity of an EPR spectrometer, a single signal is observed, with a *g* value of 2.03 for **3** and 2.00 for **4**, values typical of those for Fe^{III} complexes [22].

After exhaustive bulk electrolysis, the IR spectra of the solutions show only one terminal carbonyl absorption (at 2005 cm^{-1} for **3** and 1980 cm^{-1} for **4**) and a

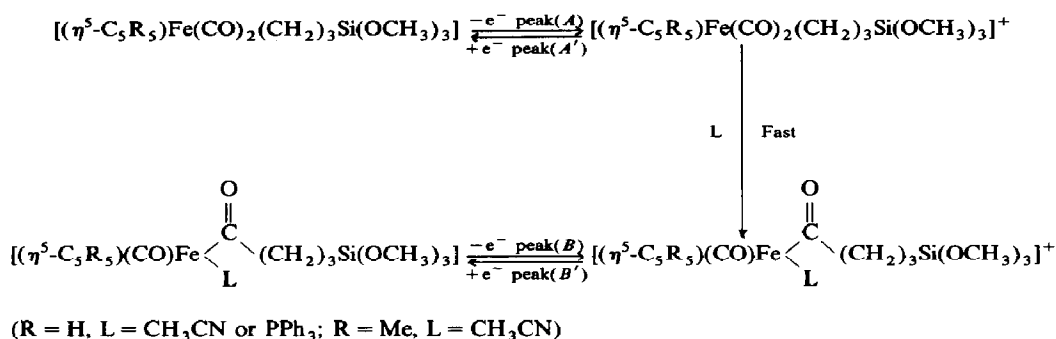
Table 2

Cyclic voltammetric ^a data for the complexes at 200 mV s^{-1}

Compound	E_p (A)	<i>B/B'</i>			<i>A/A'</i> ^b		
		$E_{1/2}$	ΔE_p	i_c/i_a	$E_{1/2}$	ΔE_p	i_c/i_a
3	+0.51	-0.33	78	1	+0.49	76	1
4	+0.25	-0.49	78	0.7	+0.22	78	0.97
5		-0.13	76	1			

^a At a glassy carbon electrode in acetonitrile. Potentials are relative to the $[\text{Fe}(\eta^5\text{-C}_5\text{H}_5)_2]^+ / [\text{Fe}(\eta^5\text{-C}_5\text{H}_5)_2]$ couple. ^b At -30°C .

strong acyl absorption at 1695 cm^{-1} for **3** and 1675 cm^{-1} for **4**, indicating an acyl(carbonyl) rather than a dicarbonyl structure. This suggests the formation of radical cations $[(\eta^5\text{-C}_5\text{R}_5)(\text{CO})(\text{CH}_3\text{CN})\text{FeC}(\text{O})(\text{CH}_2)_3\text{Si}(\text{OCH}_3)_3]^+$ (see electrochemical scheme 2).



Scheme 2

Addition of PPh₃ to acetonitrile solutions of **3** changes the CV curves (Fig. 1). The reversible system *B/B'* shifts to more positive potentials, the peak potentials now being identical to the oxidation and reduction peak potentials observed in the CV curves of the acetonitrile solutions of the chemically supplied acyl complex **5** (Table 2, Fig. 1). Furthermore the species formed in the first oxidation step (*A*) shows ESR signals ($g = 2.180$) which are in accord with the formation of the cation $[5]^+$.

These results indicate that migratory insertion occurs on oxidation of compounds **3** and **4** (Scheme 2). This reaction is rapid at room temperature and is probably accompanied by incorporation of a nucleophile, and shows an extremely large acceleration of the CO insertion upon one-electron oxidation. A similar electro-oxidatively-induced insertion has been proposed in the electrochemical oxidation of $(\eta\text{-Cp})\text{Fe}(\text{CO})_2(\text{CH}_3)$ ($\eta\text{-Cp} = \text{C}_5\text{H}_5, \text{MeC}_5\text{H}_4$) [23,24].

In the case of the pentamethylcyclopentadienyl complex **4** a peak current ratio $i_{p_a}/i_{p_c} < 1$ was observed for the pair *B/B'*, suggesting that the complex $(\eta^5\text{-C}_5\text{Me}_5)(\text{CO})(\text{CH}_3\text{CN})\text{FeC}(\text{O})(\text{CH}_2)_3\text{Si}(\text{OCH}_3)_3$ formed is somewhat unstable in the cyclic voltammetry time scale. Furthermore addition of PPh₃ to acetonitrile solutions of **4** cause no change in the initial cyclic voltammograms, which is in accord with the chemical experiments.

The dependence on the nature of the entering ligand suggests that the migratory insertion may be assisted by a nucleophile, as observed for other complexes [25,26]. Thus it is possible to define a mechanism that does not involve the 15-electron species $[(\eta^5\text{-C}_5\text{H}_5)(\text{CO})\text{FeC}(\text{O})(\text{CH}_2)_3\text{Si}(\text{OCH}_3)_3]^+$ which would be generated in the absence of participation by a nucleophile [24]. An alternative way of avoiding a 15-electron species would involve dihapto coordination of the acyl ligand [27].

Experimental

All reactions and manipulations were carried out under oxygen-free nitrogen by Schlenk techniques. Tetrahydrofuran (THF) was distilled from sodium benzophe-

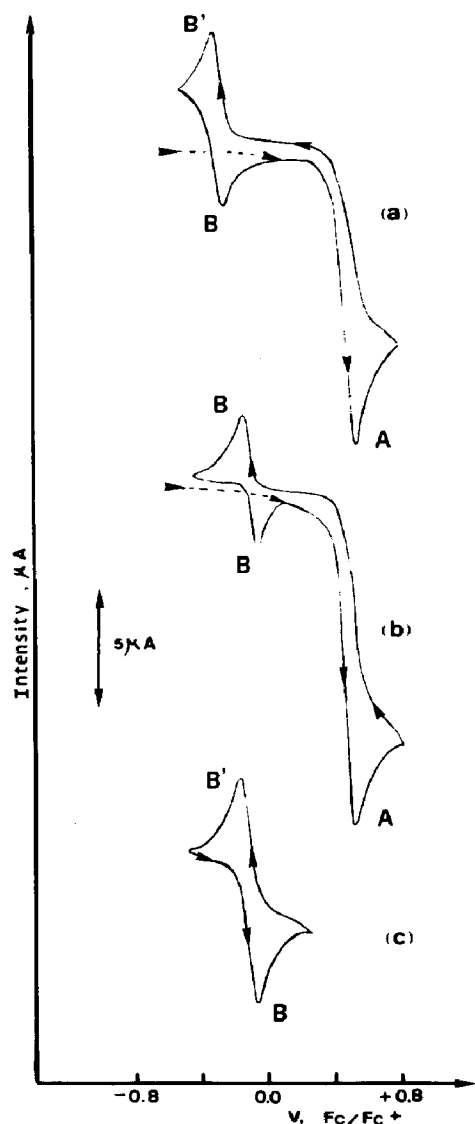


Fig. 1. Cyclic voltammograms, in CH_3CN solutions, at a glassy carbon electrode for: (a) $(\eta^5\text{-C}_5\text{H}_5)\text{Fe}(\text{CO})_2(\text{CH}_2)_3\text{Si}(\text{OCH}_3)_3$ (**3**), scan rate 0.2 Vs^{-1} ; (b) $(\eta^5\text{-C}_5\text{H}_5)\text{Fe}(\text{CO})_2(\text{CH}_2)_3\text{Si}(\text{OCH}_3)_3$ (**3**), in the presence of PPh_3 , scan rate 0.2 Vs^{-1} ; (c) $(\eta^5\text{-C}_5\text{H}_5)(\text{CO})(\text{PPh}_3)\text{FeC}(\text{O})(\text{CH}_2)_3\text{Si}(\text{OCH}_3)_3$, (**5**), scan rate 0.2 Vs^{-1} .

none ketyl under nitrogen before use. Acetonitrile was refluxed in phosphorus pentoxide and distilled from calcium hydride before use. Infrared spectra were recorded in the range $4000\text{--}200 \text{ cm}^{-1}$ on a Nicolet 5DX, FT-IR spectrophotometer with Nujol mull and liquid films between CsI windows. The solution spectra were examined in a 0.1 mm cell with KBr windows. The proton NMR spectra were recorded on a Bruker WH-200-5Y (200 MHz, Fourier-Transform mode) spectrometer. Chemical shifts (δ) are reported in ppm, relative to internal SiMe_4 .

3-Chloropropyltrimethoxysilane provided by the Dow-Corning Corporation and purified by distillation in vacuo, b.p. 81°C at 0.5 mmHg.

For column chromatography, either neutral alumina (Fluka) or silica (Merck), both pretreated at 250 °C and at 10⁻³ mmHg, was used for compounds **3** and **4**. For complex **5** silica (Merck) pretreated with Me₃SiCl in C₆H₆ [28] or commercially-available silanised silica (Merck) can be used.

The electrochemical apparatus and techniques have been described previously [29]. All potentials are relative to the [Fe(η⁵-C₅H₅)₂]⁺ [Fe(η⁵-C₅H₅)₂]⁻ couple.

Preparation of complexes M[(η⁵-C₅R₅)Fe(CO)₂]

(a) *R = H (1)*. The sodium salt, Na[(η⁵-C₅H₅)Fe(CO)₂] was prepared by treatment of [(η⁵-C₅H₅)Fe(CO)₂]₂ with an excess of a sodium dispersion in refluxing THF for 6 h, as previously described [8]. This THF solution can be used in situ for the preparation of the derivative **3**.

The potassium salt was synthesized by use of potassium benzophenone ketyl in THF solution, at room temperature. After filtration and washing, pure K[(η⁵-C₅H₅)Fe(CO)₂] was obtained.

(b) *R = Me (2)*. A mixture of [(η⁵-C₅Me₅)Fe(CO)₂]₂ (1.0 g, 2.0 mmol) and Na dispersion (0.36 g, 6.0 mmol) was kept under vacuum for 30 min then oxygen-free N₂ was introduced and degassed THF (100 cm³) was added. The mixture was refluxed for 10 h, after which time monitoring by IR spectroscopy indicated that none of the starting dimer remained. This orange mixture, which contained the anion Na⁺[(η⁵-C₅Me₅)Fe(CO)₂]⁻, was used in situ for the subsequent reactions described below.

Preparation of (η⁵-C₅R₅)Fe(CO)₂(CH₂)₃Si(OCH₃)₃

(a) *R = H (3)*. A THF solution of (CH₃O)₃Si(CH₂)₃Cl (1.33 g, 6.75 mmol), was added dropwise to a stirred degassed solution (30 cm³), of K[(η⁵-C₅H₅)Fe(CO)₂] (1.0 g, 4.6 mmol) in THF and cooled to -10 °C. The reaction was monitored by IR spectroscopy, the initial ν(CO) bands of **1** at 1868, 1792 and 1772 cm⁻¹, disappeared to give way to new strong bands at 2000 and 1942 cm⁻¹. After 1 h the mixture was allowed to warm to room temperature then stirred for 12 h. The brown-yellow solution was filtered and evaporated and the residue was dissolved in n-pentane and chromatographed on a neutral alumina (or silica) (3 × 30 cm). Elution with n-pentane followed gave **3** as a yellow-orange oil. Yield 70%. δ(H) (CDCl₃): 0.7–0.9 (2 H, m, CH₂Si); 1.85 (4 H, m, (CH₂)₂); 3.55 (9 H, s, (OCH₃)₃); 4.72 (5 H, s, C₅H₅).

(b) *R = Me (4)*. A similar procedure was used with Na[(η⁵-C₅Me₅)Fe(CO)₂] (1.1 g, 4.05 mmol) (in 50 cm³ of THF), and (CH₃O)₃Si(CH₂)₃Cl (1.2 g, 6.1 mmol). After 12 h, the initial bands of the sodium salt **2** at 1805 and 1870 cm⁻¹, had completely disappeared, and been replaced by two new strong bands at 1981 and 1922 cm⁻¹. After work-up as for **3**, complex **4** was isolated as red-orange oil. Yield 65%. δ(H) (CDCl₃): 0.7–0.9 (2H, m, CH₂Si), 1.73 (15 H, s, C₅(CH₃)₅); 1.90 (4 H, m, (CH₂)₂); 3.57 (Si(OCH₃)).

Preparation of (η⁵-C₅H₅)(CO)(PPh₃)FeC(O)(CH₂)₃Si(OCH₃)₃ (5)

A mixture of **3** (1 g, 2.9 mmol) and triphenylphosphine (0.76 g, 3.0 mmol), in 50 ml of CH₃CN under nitrogen was heated under reflux for 20 h. The progress of the reaction was monitored by IR spectroscopy. The initial bands of complex **3** at 1999 and 1939 cm⁻¹ gradually disappeared, and were replaced by a strong single terminal

carbonyl band at 1912 cm^{-1} , and a strong and broad band at 1608 cm^{-1} attributable to the acyl group.

When the reaction was complete, the solvent was removed, and the residue was dissolved in ether and chromatographed on silanised silica gel. Elution with ether gave a yellow band, the solution from which was concentrated to 20 ml and cooled to give a yellow crystalline solid, which was filtered off. Yield 45%. $\delta(\text{H})$ (CDCl_3): 0.85 (2 H, m, CH_2Si), 1.87 (4 H, m, $(\text{CH}_2)_2$), 3.55 (9 H, m, $\text{Si}(\text{OCH}_3)_3$), 4.75 (5 H, s, C_5H_5), 7.48 (15 H, m br, C_6H_5).

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