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An electron-transfer induced migratory insertion reaction originating from a 19-electron cobaltacyclic anion

Andre Morneau, Bernadette T. Donovan-Merkert [1], William E. Geiger *

Department of Chemistry, University of Vermont, Burlington, VT 05405, USA

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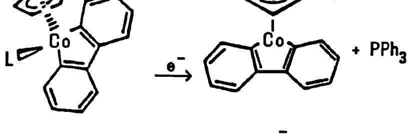
Abstract

The cyclopentadienyl cobaltafluorenyl carbonyl complex $Cp(CO)CoC_{12}H_8$ (**3**) is reduced in THF in a one-electron irreversible process. The reduction products include the fluorenone radical anion, $\mathbf{Fl}^-$, which is proposed to arise through migratory insertion of the CO ligand into the metallacyclic Co–C bond of the 19-electron complex $\mathbf{3}^-$. Electrochemical and IR analyses show that bulk electrolysis under N_2 gives 1/3 equiv. each of $\mathbf{Fl}^-$ and $CpCo(CO)_2$ and 2/3 equiv. of a 17-electron anion $[CpCoC_{12}H_8]^-$ ($\mathbf{2}^-$) Under CO the yield of $\mathbf{Fl}^-$ is quantitative. The insertion product is also formed when the reduction of the analogous phosphine complex $Cp(PPh_3)CoC_{12}H_8$ is performed under CO. An equilibrium between 17- and 19-electron compounds is postulated to account for the synthetic and voltammetric observations. When the cobaltacycle $Cp(PPh_3)CoC_4Ph_4$ (**5**) is reduced under carbon monoxide, the CO-insertion product is the π-cyclopentadienone complex $CpCo(\eta^4\text{-}C_4Ph_4O)$ (**8**). © 2000 Elsevier Science S.A. All rights reserved.

Keywords: Electron transfer; Migratory insertion; Cobalt complexes; Fluorenyl complexes; Cyclopentadienyl complexes

1. Introduction

One-electron reduction of cobaltafluorenyl compounds of the type **1**, $L = PPh_3$, may be used to generate a coordinatively-unsaturated Co(II) center [1,2] owing to rapid cleavage of the Co–P bond in the electron-transfer step (Eq. 1 when $L = PPh_3$):



1, L = PPh₃ 2⁻
3, L = CO (1)

The lifetime of the putative anion $\mathbf{1}^-$ is extremely short. The reduction of **1** is electrochemically irreversible at CV scan rates up to 5000 V s^{-1}, placing an upper limit of about 40 μs on the half-life of $\mathbf{1}^-$ and raising the possibility that cleavage of the Co–P bond is concerted with electron transfer [1]. In an attempt to find a longer-lived (i.e. detectable) 19-electron species we turned to the carbonyl derivative **3** (L = CO), reasoning that a π-acid ligand might stabilize the hypervalent anion. This paper reports the reduction of **3**, which is still found to be fully irreversible, supporting the idea that the LUMO of cobaltacycles of this type has a strong σ* contribution from the Co–L fragment.

A surprising cathodic product was found which distinguishes the reduction of **3** from that of its phosphine or phosphite analogues, namely the fluorenone anion, $\mathbf{Fl}^-$. Formation of this compound suggests that insertion of the carbonyl ligand into the Co–C metallacyclic bond is kinetically competitive with cleavage of the Co–CO bond and is relevant to the phenomenon of accelerated insertion reactions of odd-electron organometallics [3–11] as well as to the more general topic of the reactions of 19-electron systems [12]. We now expand on our earlier communication [1b] to provide details of this electrochemically-induced insertion reaction and show, by contrast, that the reduction of a similar cobaltacyclopentadiene complex **5** under

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* Corresponding author. Tel.: +1-802-656 0268; fax: +1-802-656 8705.

[1] Present address: Department of Chemistry, University of North Carolina at Charlotte, Charlotte, NC 28223, USA.

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CO leads to formation of a metal π-complex in which the carbonyl group has been incorporated into the π-ligand.

2. Experimental

Manipulations were performed under an atmosphere of N_2, unless noted otherwise, using standard Schlenk and dry-box techniques.

2.1. Chemicals

$Cp(CO)CoC_{12}H_8$ (**3**) was prepared by the literature method [13] and stored under N_2 at 253 K. The tetraphenylcyclobutadiene complex $CpCo(\eta^4\text{-}C_4Ph_4H_2)$ (**7**) was prepared by reductive electrolysis [14] of the metallacycle $Cp(PPh_3)CoC_4Ph_4$ (**5**) and purified by column chromatography on neutral alumina using hexanes as the eluent. Carbon monoxide (CP grade, Linde Specialty Chemicals) was passed through traps of soda-lime followed by activated silica gel. Electrochemical solvents and supporting electrolytes were prepared as described [2]. Fluoreneone (Aldrich) was recrystallized from absolute ethanol and vacuum dried. $CpCo(CO)_2$ (**4**) was used as received from Strem.

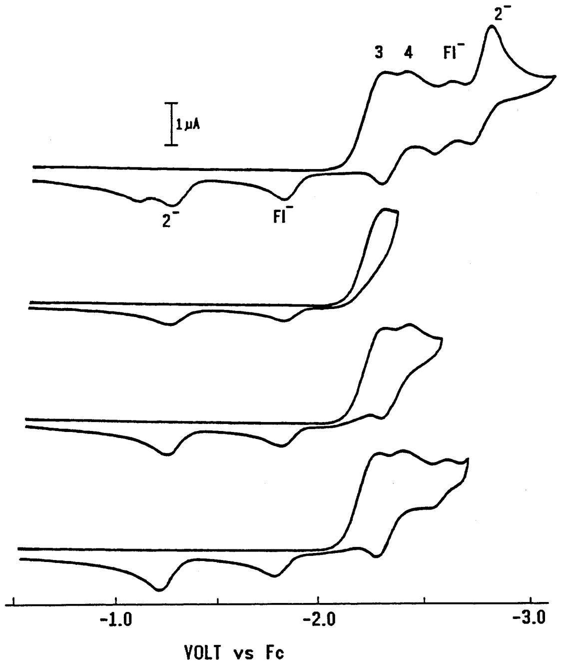


Fig. 1. Cyclic voltammetric scans of 0.5 mM **3** in THF–$[NBu_4][PF_6]$ at the Pt electrode, $T = 273$ K, scan rate 0.2 V s^{-1}, showing the effects of clipping the negative-going scans at different potentials. The legends on the top CV give the species assigned to the individual waves.

2.2. Electrochemical procedures

Voltammetry and electrolyses were performed using procedures largely described earlier [2]. Mercury-coated Pt working electrodes were prepared by the method of Ramaley et al. [15]. Steady-state voltammograms (SSVs) were recorded at Hg-coated electrodes rotated at 1800 rpm. All potentials are reported against the ferrocene/ferrocenium reference couple. The experimental reference electrode was the aqueous saturated calomel electrode (SCE); potentials in THF may be converted from the ferrocene scale to the SCE scale by addition of 0.56 V.

2.3. Infrared analytical procedures

The electrolytic yields of compounds **3**, **4**, and fluorenone (**Fl**) were analyzed by quantitative IR spectroscopy using a Mattson Polaris IR spectrometer operating at 2 cm^{-1} resolution. The analyte solutions were tested in a sealed IR cell having a path length calibrated as 0.48 mm (Spectra-Tech; Barnes Analytical Division). Analytical working curves prepared from standard solutions of each compound in THF–0.1 M $[NBu_4][PF_6]$ were linear within the concentration range of 1×10^{-4} to 8×10^{-3} M (correlation coefficients > 0.999). The analytical wavelengths were 2024 cm^{-1} for **3**, 2021 or 1957 cm^{-1} for **4**, and 1720 cm^{-1} for **Fl**. When mixtures of **3** and **4** were present, the band at 1957 cm^{-1} was used to analyze for **4**. Then spectral subtraction of the 1957 cm^{-1} feature was used to rid the spectrum of the influence of **4**, leaving the 2024 cm^{-1} band for the analysis of **3**.

3. Results

3.1. Reduction of 3 under N_2

Cyclic voltammetry scans of the cobaltacycle carbonyl complex **3** demonstrate the elegance of this technique in mapping complex redox processes. The CV scan of Fig. 1 is rich with features, each of which can be assigned to either the reactant **3** or one of its reduction products by matching the observed potentials and chemical reversibilities to those of known compounds or ions. Table 1 and the labels in Fig. 1 summarize these assignments, which show that the reduction of **3** ($E_{pc} = -2.28$ V) produces the 17-electron cobaltacycle $\mathbf{2}^-$ (reduction at -2.80 V and oxidation at -1.26 V), the fluorenone anion $\mathbf{Fl}^-$ (reduction at -2.60 V and oxidation at -1.88 V) and $CpCo(CO)_2$ (**4**, $E_{1/2} = -2.38$ V). The general appearance of the voltammograms did not change over the scan rate range $0.05 < v < 100$ V s^{-1}.

Table 1
Potentials of cobaltacycles and other compounds and ions relevant to this study

Species	No.	Red/Ox	Rev?	Potential (V vs. Fc)	Ref.
$Cp(PPh_3)CoC_{12}H_8$	**1**	redn	no	-2.56 (E_{pc})	[1b] [a]
$[CpCoC_{12}H_8]^-$	**2**$^-$	redn	yes	-2.80 ($E_{1/2}$)	[1a]
		oxdn	no	-1.26 (E_{pa})	[1a]
$Cp(CO)CoC_{12}H_8$	**3**	redn	no	-2.28 (E_{pc})	this work
$CpCo(CO)_2$	**4**	redn	yes	-2.38 ($E_{1/2}$)	[1b] [b]
[fluorenone]$^-$	**Fl**$^-$	redn	yes	-2.60 ($E_{1/2}$)	this work [c]
		oxdn	yes	-1.88 ($E_{1/2}$)	
$Cp(PPh_3)CoC_4Ph_4$	**5**	redn	no	-2.26 (E_{pc} on Hg) -2.42 (E_{pc} on Pt)	[14]
$CpCo(\eta^4\text{-}C_4Ph_4H_2)$	**7**	redn	yes	-2.58 ($E_{1/2}$)	[14]
$CpCo(\eta^4\text{-}C_4Ph_4O)$	**8**	redn $^{0/1-}$	yes	-1.96 ($E_{1/2}$)	
		redn$^{1-/?}$	no	-3.09 (E_{pc})	[17] [a]

[a] Potential vs. SCE converted here to ferrocene scale.

[b] Incorrect potential quoted in Ref. [1b]; $E_{1/2} = -0.82$ V should have been written as -1.82 V. Conversion to ferrocene scale gives value in present table. See also Ref. [24].

[c] Potentials in this table measured on authentic sample in THF, giving slightly different values than reported in the literature (see Ref. [25]) for values in DMF.

Substitution of the π-acid CO in **3** for PPh_3 in **1** ($E_{pc} = -2.56$ V, Table 1) results, as expected, in a positive shift (approximately 280 mV) to a more facile reduction potential, but the 19-electron anion **3**$^-$ is still not detected by voltammetry; instead, rapid cleavage of the Co–CO bond produces the 17-electron metallacycle **2**$^-$ [1,2] and the fluorenone anion. Observation of the latter on the CV time scale suggests that, for the 19-electron system **3**$^-$, two processes occur at competitive rates: (i) loss of CO and (ii) insertion of CO into the Co–C metallacyclic bond.

The breadth ($E_p - E_{p/2}$) of the cathodic peak for **3** is 85 mV, suggesting an electrochemically irreversible process with a transfer coefficient, α, of about 0.56. The one-electron stoichiometry was diagnosed by the similarity of the CV peak height of **3** to that of **1**, which is known to be reduced by a one-electron process [2]. Bulk electrolysis at the foot of the reduction wave of **3** ($E_{appl} = -2.20$ V, coulometry average 1.1 F/eq) confirmed this. A balanced reaction involving single electron stoichiometry and the chemical species detected by voltammetry (Eq. (2)) suggests theoretical mole fraction (mf) yields of 0.33 for both **Fl**$^-$ and $CpCo(CO)_2$ and 0.66 for **2**$^-$:

$$3\mathbf{3} + 3e^- \rightarrow \mathbf{Fl}^- + CpCo(CO)_2\ (\mathbf{4}) + 2\mathbf{2}^- \quad (2)$$

Actual electrolytic yields were tested by a combination of coulometry, voltammetry and IR analysis. The yield of **Fl**$^-$ was obtained after first converting the radical anion back to its neutral form, **Fl**. A potential of $E_{appl} = -1.55$ V is benign to all the catholyte reaction products except for **Fl**$^-$, which is oxidized back to **Fl**. After such a 'back-electrolysis' a sample of the solution was withdrawn for quantitative IR analysis of both **Fl** (1720 cm^{-1}) and $CpCo(CO)_2$ (2024 and 1957 cm^{-1}). Prior to the back-electrolysis the solution had an intense band at ca. 1549 cm^{-1} which can be attributed to the carbonyl stretching band of the radical anion **Fl**$^-$. The decrease of approximately 171 cm^{-1} in the CO frequency in going from **Fl** to **Fl**$^-$ is consistent with the results of Clark and Evans on benzoquinones and their reduction products. These authors reported an average of -155 cm^{-1} shift for this band in a series of quinones reduced to their radical anions [16]. The steady-state plateau current of the **Fl** reduction wave ($E_{1/2} = -1.88$ V) after back-electrolysis served as an independent measure of the yield of fluorenone.

The yield of **2**$^-$ was obtained by coulometry. After the initial back-electrolysis at $E_{appl} = -1.55$ V, the solution consists of **2**$^-$, **Fl** and **4**, only the first of which is oxidizable at $E_{appl} = -1.2$ V. A second back-electrolysis at the latter potential gave a coulometric count that was related to the concentration of **2** by Faraday's law. The results are given in Table 2. The yields of all three products are acceptably close to those expected on the basis of Eq. (2). The total mf of cobalt-containing species (**4** and **2**$^-$) was measured as 0.80 to 0.84; some $[Co(CO)_4]^-$ (detected by IR, 1887 cm^{-1}) accounts for the 'missing' cobalt. The overall reduction mechanism is shown in the Scheme 1.

3.2. Reduction of 3 under CO

The behavior of this system under a carbon monoxide atmosphere was investigated. The 17 e^-/19 e^- equilibrium of Eq. (3), well established for analogues involving PR_3 in place of CO [2], plays the key role in allowing

$$\mathbf{2}^- + CO \rightleftharpoons \mathbf{3}^- \quad (3)$$

completion of the insertion reaction. Even though this equilibrium undoubtedly lies far in favor of the 17-electron **2**$^-$, a very rapid insertion of CO into the Co–C metallacyclic bond in 19-electron **3**$^-$

Table 2
Product yields in bulk electrochemical reduction of **3** or **5** in THF–0.1 M $[NBu_4][PF_6]$ expressed as mole fractions of reactant

Reactant	Analyte	Analytical method		
		Coulometry [a]	SSV [b]	IR
(i) **3** under N_2	**Fl**$^-$		0.33	0.29
	$CpCo(CO)_2$ (**4**)		0.29	0.25
	2$^-$	0.55		
(ii) **3** under CO	**Fl**$^-$		1.00	1.00
	4		0.57	0.60
	2$^-$	~0		
(iii) **5** under CO	**Fl**$^-$		0.76	0.50

[a] Coulometry of bulk oxidation ($E_{appl} = -1.3$ V) of **2**$^-$ produced in reductive electrolyses of **3**.

[b] From plateau height of steady-state voltammogram obtained with rotating mercury drop electrode.

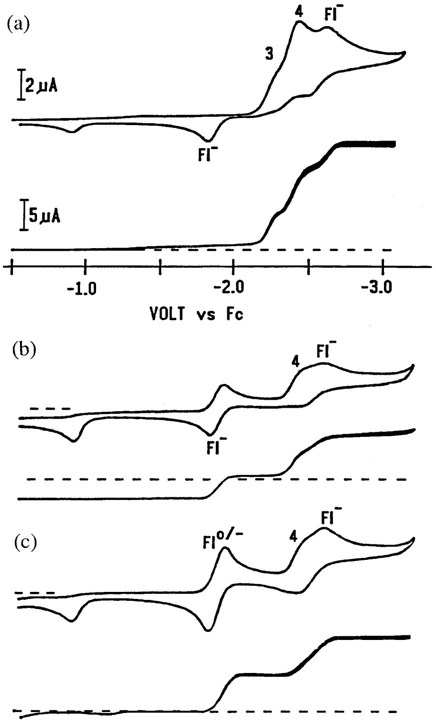
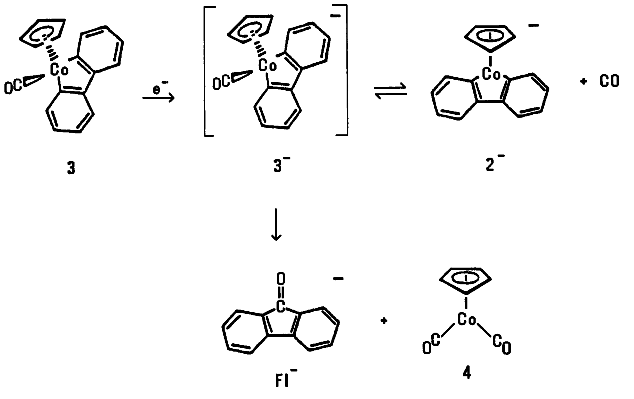


Fig. 2. CV (scan rate 0.2 V s^{-1}) and SSV curves (0.005 V s^{-1}) for a bulk electrolysis experiment involving 1.3 mM **3** in THF–$[NBu_4][PF_6]$ at room temperature under CO. (a) before electrolysis (b) after exhaustive electrolysis at $E_{appl} = -2.30$ V and (c) after back-electrolysis at $E_{appl} = -1.55$ V.

can give efficient formation of the insertion product **Fl**$^-$. This expectation is supported by the CV scan of Fig. 2(a) in which the peak for **Fl**$^-$ is enhanced compared to the behavior under N_2 and that of **2**$^-$ is absent, suggesting complete reaction of the latter over the CV time scale in the presence of excess CO.

Bulk reduction at $E_{appl} = -2.30$ V confirmed this conclusion, since a high yield of fluorenone anion is obtained according to both voltammetric and IR analysis (Table 2). The electrolysis products and their oxidation states can be followed by a combination of CV and SSV scans. After bulk reduction of **3** under CO (Fig. 2(b)) the only electroactive products are $CpCo(CO)_2$ (**4**) and **Fl**$^-$ and/or **Fl**. The steady-state (SSV) scan of Fig. 2(b) shows that about 80% of the fluorenone is present in its anionic form. When the electrolyzed solution of Fig. 2(b) is oxidized at $E_{appl} = -1.55$ V, **Fl**$^-$ is oxidized to neutral **Fl** and the reversible wave at $E_{1/2} = -1.88$ V becomes cathodic (Fig. 2(c)). Concerning the cobalt-containing products, a somewhat lower than expected yield of **4** (0.57 mf) is explained by an increase in $[Co(CO)_4]^-$ owing to partial electrolytic reduction of $CpCo(CO)_2$ at this applied potential. The anodic wave at approximately -0.9 V most likely arises from the oxidation of $[Co(CO)_4]^-$.

The amount of insertion product could also be increased if CO was added after electrolysis of **3** under N_2. For example, when CO was admitted at open circuit to a solution containing **2**$^-$, the 17-electron anion reacted over about a 10 min period to give an approximate 50% increase in the **Fl**$^{0/-}$ wave concomitant with loss of the wave for **2**$^-$.



Scheme 1.

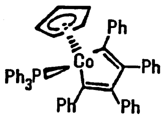
3.3. Reduction of 1 under CO

The triphenylphosphine complex **1** was investigated under CO. In sharp contrast to its behavior under N_2 (Fig. 3(a)) [2], reduction under CO gave no evidence for the unsaturated 17-electron metallacycle **2**$^-$ in either CV scans or bulk electrolyses. In fact, the only CV feature negative of -2 V is a broad wave with E_{pc} approximately -2.86 V (Fig. 3(b)). The probable electrolysis products **3**, **4** and **Fl**$^-$ all have reduction processes near to or more positive than this value, so the cathodic wave of Fig. 3(b) is undoubtedly a composite of these processes. Noteworthy, however, is the absence of a wave (for oxidation of **2**$^-$) at $E_{pa} = -1.26$ V, implying that CO has reacted with any **2**$^-$ produced in the reduction. After bulk reduction and back-electrolysis the yield of **Fl** was about 0.76 (mf). No $CpCo(CO)_2$ was formed in the electrolysis because the applied potential was sufficiently negative to reduce **4** to $[Co(CO)_4]^-$ (the reduction of **1** requires a more negative potential compared to **3**).

The above experiments are all consistent with the 17-electron/19-electron equilibrium of Eq. (3) again being operative, as previously demonstrated with phosphines [1,2].

3.4. Reduction of 5 under CO

The cobaltacycle **5** has been shown [14] to reduce initially by the same mechanism as observed for **3**, namely rapid loss of the two-electron ligand (PPh_3 for **5**) following one-electron transfer to give a 17-electron metallacyclic anion ($\mathbf{6}^-$, Fig. 4(a), dotted line observed under N_2). The mechanisms depart at this point because **6** rearranges (with gain of protons and another electron) to a π-complex containing an η^4-tetraphenylbutadiene ligand **7** [14].



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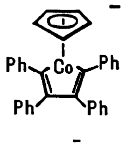


6⁻

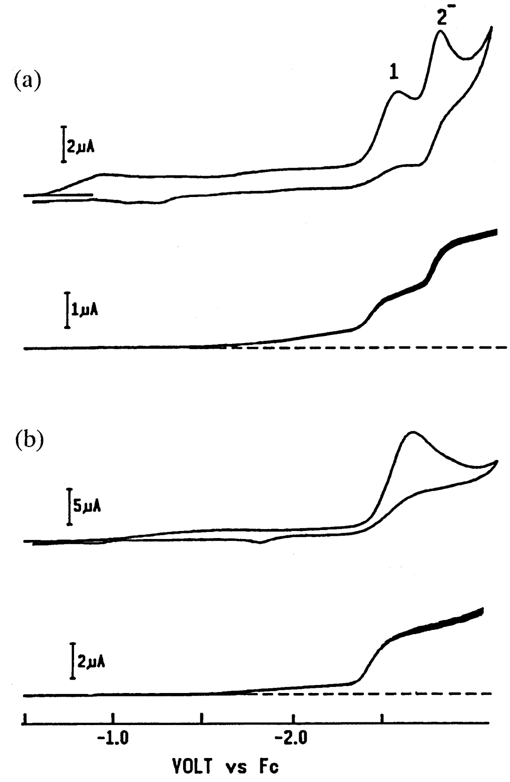


Fig. 3. CV (scan rate 0.2 V s^{-1}) and SSV curves (0.005 V s^{-1}) of 1.5 mM **1** at room temperature. (a) under N_2 and (b) under CO.

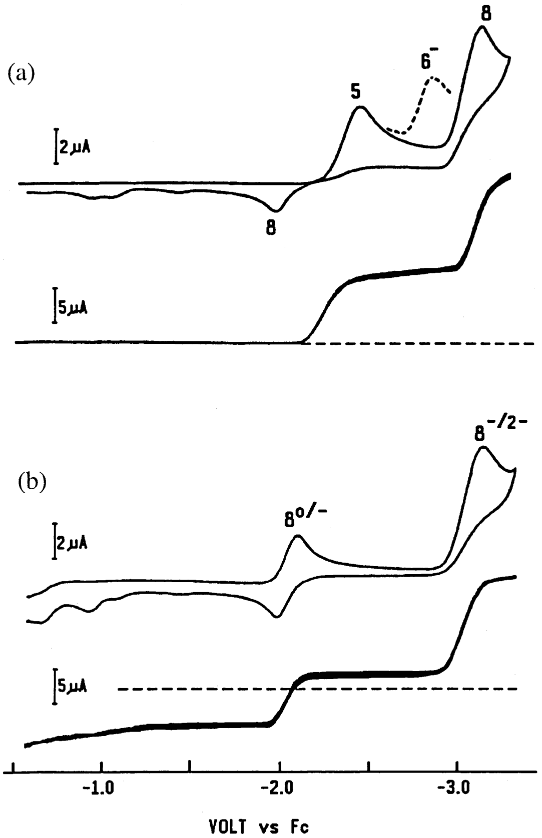
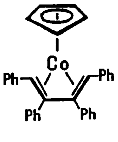


Fig. 4. CV (scan rate 0.2 V s^{-1}) and SSV curves (0.005 V s^{-1}) of 2 mM **5** under CO at room temperature in THF–$[NBu_4][PF_6]$. (a) before electrolysis and (b) after exhaustive electrolysis at $E_{appl} = -2.55$ V. Dotted line in (a) gives the result under nitrogen.

The reduction of **5** under CO, however, results in an η^4-diolefin complex in which CO inserts into the Co–metallacyclic bond. A CV scan (Fig. 4(a), solid line) shows two major features besides that of the reduction wave of **5** at $E_{pc} = -2.42$ V, namely a reversible couple at $E_{1/2} = -1.96$ V and an irreversible reduction at $E_{pc} = -3.09$ V. These two features, formed almost exclusively in the bulk electrolysis of **5** (Fig. 4(b)), fall at potentials consistent with those observed for CpCo(η^4-tetraphenylcyclopentadienone) (**8**) [17]. The formation of **8** as the major electrolysis product was confirmed by its isolation after bulk electrolysis of **5** under CO (column chromatography under N_2; IR and NMR analysis). A small amount (< 5%) of the free ligand tetraphenylcyclopentadieneone was also found.



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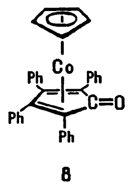

8

Since complex **7** is known to react with CO at elevated temperatures over longer times to produce **8**

[18], it was important to perform a control experiment to assure that **7** was not an intermediate leading to **8** under electrolytic conditions. Accordingly, **7** was reduced electrolytically ($E_{\mathrm{appl}} = -2.7$ V) under CO; the exclusive electrolysis product had an irreversible reduction peak at -2.8 V. Although the product was not identified, it was definitely not the π-tetraphenylcyclopentadienone complex **8**, allowing us to conclude that the reductive route of **5** to **8** does not proceed through **7**.

4. Discussion

The irreversibility of the reduction of **3** shows that one or more rapid reactions either accompany or closely follow formation of the 19-electron system $\mathbf{3}^-$. Consistent with the LUMO of cobaltacycles of the type $Cp(L)CoC_{12}H_8$ being anti-bonding between the metal and the two-electron ligand L [1,2], scission of the Co–CO bond is observed, with formation of the 17-electron anion $\mathbf{2}^-$. Formation of the insertion product $\mathbf{Fl}^-$ was unexpected. The stoichiometry of the reaction is established as that given in Eq. (2), but the mechanism is less certain. Given the rapidity of the formation of the insertion product (some $\mathbf{Fl}^-$ is detected within 10 ms in CV scans) and the firm establishment [2] of the 17-electron/19-electron equilibrium of Eq. (3), it is quite likely that insertion occurs in the metastable system $\mathbf{3}^-$ (Scheme 1). The question of the electronic structure of the radical in the insertion step has been raised in a number of cases involving CO insertions and electron-transfer catalyzed reactions [4–6,19]. In the most probable scenario [19,20] that the Cp ring does not undergo hapticity lowering in $\mathbf{3}^-$, a 19-electron complex appears to be responsible for the greatly accelerated insertion of CO into the Co–C metallacyclic bond in $\mathbf{3}^-$ compared to **3**. This is another example of the enormous rate enhancements sometimes possible in reactions of odd-electron organometallic compounds [21–23].

Acknowledgements

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