

# Reactivity of 17- and 19-electron organometallic complexes. Formation of bent sandwich 19-electron radical cation complexes of osmium and ruthenium

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## Abstract

Redox behavior of indenyl sandwich complexes of general formula  $(\eta^5\text{-C}_9\text{H}_7)\text{ML}$  ( $\text{M} = \text{Ru}$  and  $\text{L} = \eta^5\text{-C}_9\text{H}_7$  (I),  $\eta^5\text{-C}_5\text{H}_5$  (II),  $\eta^5\text{-C}_5\text{Me}_5$  (III);  $\text{M} = \text{Os}$ ,  $\text{L} = \eta^5\text{-C}_9\text{H}_7$  (IV)) has been studied by means of cyclic voltammetry at the Pt-electrode at  $-85$ – $20^\circ\text{C}$  in THF, MeCN and  $\text{CH}_2\text{Cl}_2$ . The title complexes have been found to undergo reversible one-electron oxidation to corresponding radical cations, whose stability and reactivity depend on the nature of both a metal and the second aromatic  $\pi$ -ligand L and nucleophilic properties of the solvent. Fast reversible interaction of the electrogenerated 17-electron radical cations with nucleophiles yields bent sandwich 19-electron radical cations,  $[(\eta^5\text{-C}_9\text{H}_7)\text{M}(\text{Nu})\text{L}]^{+\cdot}$  ( $\text{Nu} = \text{Cl}^-$ , MeCN, THF), the latter undergo one-electron oxidation to corresponding 18-electron  $[(\eta^5\text{-C}_9\text{H}_7)\text{M}(\text{Nu})\text{L}]^{2+}$  dications.

**Keywords:** Ruthenium; Osmium; Cyclopentadienyl; Indenyl; Electrochemistry

## 1. Introduction

Electrochemical oxidation of bis-cyclopentadienyl complexes and their decamethyl-substituted analogs has been studied more extensively among the sandwich complexes of the iron group metals with  $\eta^5$ -coordinated aromatic ligands [1,2]. Reactivity of the 17-electron radical cations formed as primary products is strongly dependent on the nature of both the metal and  $\pi$ -ligands. Investigations of redox reactions of iron group metal sandwich complexes with  $\eta^5$ -coordinated polycyclic aromatic ligands are few and far between. The potentials of electrochemical oxidation were determined for  $\eta^5$ -indenyl and  $\eta^5$ -fluorenyl complexes of iron [3–5] and ruthenium [6]. Some reactions of the 17-electron radical cation of dibenzoferrrocene generated under oxidation have been reported [4,5]. However, the data available are not sufficient for a comparative analysis of general trends in redox properties of sandwich complexes of the iron group metals with  $\eta^5$ -coordinated

polycyclic ligands. For this purpose, the electrochemical behavior of complexes of general formula  $(\eta^5\text{-C}_9\text{H}_7)\text{ML}$ , where  $\text{M} = \text{Ru}$ ,  $\text{L} = \eta^5\text{-C}_9\text{H}_7$  (I),  $\eta^5\text{-C}_5\text{H}_5$  (II),  $\eta^5\text{-C}_5\text{Me}_5$  (III) and  $\text{M} = \text{Os}$ ,  $\text{L} = \eta^5\text{-C}_9\text{H}_7$  (IV), was studied by means of cyclic voltammetry in aprotic solvents of different nucleophilic properties (THF, MeCN,  $\text{CH}_2\text{Cl}_2$ ).

## 2. Results and discussion

Two anodic one-electron diffusion-controlled peaks ( $i_p/\nu^{1/2} = \text{const.}$ ) are observed on cyclic voltammograms of the indenyl complexes of ruthenium and osmium I–IV at the Pt-electrode in  $\text{CH}_2\text{Cl}_2$  within the temperature range  $-80$ – $20^\circ\text{C}$  (Fig. 1, curve a; Table 1). The first peaks have appropriate cathodic responses ( $\Delta E = E_p^a - E_p^c = 60$  mV at  $20^\circ\text{C}$ ,  $i_p^c/i_p^a = 1$  where  $E_p^a$ ,  $i_p^a$ ,  $E_p^c$ ,  $i_p^c$  are the potentials and heights of the anodic and cathodic peaks respectively), which provide evidence for chemical and electrochemical reversibility [7] of the oxidation of the starting complexes to corresponding 17-electron radical cations.

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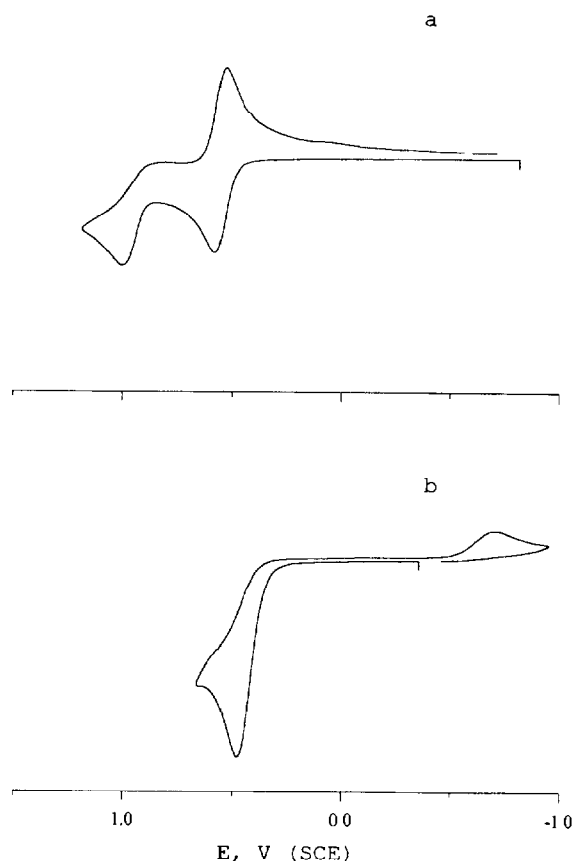


Fig. 1. Cyclic voltammograms of the complex **I** in  $\text{CH}_2\text{Cl}_2$  /0.1 M  $\text{Bu}_4\text{NPF}_6$  at the Pt-electrode, scan rate  $200 \text{ mV s}^{-1}$ ,  $T = 20^\circ\text{C}$ . Curve a, complex **I** alone; curve b, complex **I** in the presence of the five-fold  $\text{Cl}^-$  ion excess ( $\text{Et}_3\text{BzNCl}$ ).

Preparative electrochemical oxidation of complex **I** is accompanied by consumption of 1 F per mol and results in the formation of a dark-green solution of the paramagnetic complex characterized by the ESR spectra ( $g_{\parallel} = 2.622$ ,  $g_{\perp} = 1.990$ ). Reduction of the oxidized solution leads to the regeneration of complex **I** with 90% yield. Oxidation of the 17-electron radical cations at potentials of the second anodic peak is irreversible down to  $-80^\circ\text{C}$ , which is confirmed by the absence of the corresponding cathodic counterpart. This indicates an extremely low stability of 16-electron dications even

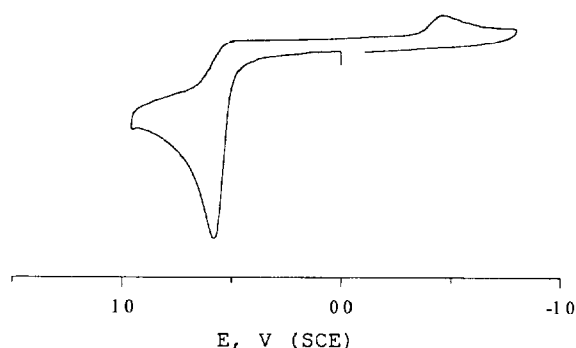
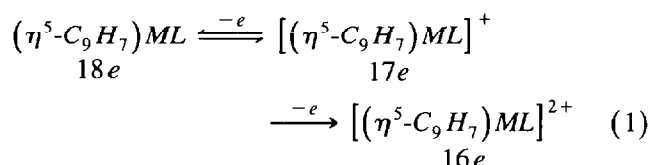


Fig. 2. Cyclic voltammogram of the complex **I** in  $\text{MeCN}$  /0.1 M  $\text{Bu}_4\text{NPF}_6$  at the Pt-electrode, scan rate  $200 \text{ mV s}^{-1}$ ,  $T = 20^\circ\text{C}$ .

in such a weakly coordinating solvent as  $\text{CH}_2\text{Cl}_2$ . The process of **I–IV** complex oxidation in  $\text{CH}_2\text{Cl}_2$  is presented as



The mechanism of electro-oxidation of complexes **I–IV** changes dramatically when strongly coordinating solvents are used instead of  $\text{CH}_2\text{Cl}_2$ , which does not exhibit nucleophilic properties ( $\text{DN} \approx 0$  [8]) in practice. In the  $\text{MeCN}$  medium all the complexes studied undergo a single two-electron, rather than one-electron, irreversible oxidation (Fig. 2, Table 1). The oxidation is followed by the appearance of cathodic peaks on the reverse part of the cyclic voltammogram; these can be ascribed to the reduction of the products formed. The difference in electrochemical behavior in  $\text{MeCN}$  vs.  $\text{CH}_2\text{Cl}_2$  indicates the solvent participation in the overall electrode reaction. The analogous participation of the solvent was observed at oxidation of  $(\eta^6\text{-C}_6\text{H}_3\text{Me}_3)\text{W}(\text{CO})_3$  [9].

To elucidate the oxidation mechanism of complexes **I–IV** in the presence of a nucleophile we undertook cyclic voltammetry studies of the title complexes in the THF media. Although THF is a stronger nucleophile ( $\text{DN} = 20.0$  [8]) compared with  $\text{MeCN}$  ( $\text{DN} = 14.4$  [8]),

Table 1

Oxidation potentials  $E^0$  V (SCE) of the Ru and Os sandwich complexes  $\text{L}_1\text{ML}_2$  with  $\eta^5$ -indenyl and  $\eta^5$ -cyclopentadienyl ligands at the Pt-electrode,  $T = 20^\circ\text{C}$  in various solvents

$\text{L}_1$	M	$\text{L}_2$	$\text{CH}_2\text{Cl}_2$		THF	MeCN
			0/+	+ /2 + <sup>a</sup>	0/2 + <sup>b</sup>	0/2 + <sup>c</sup>
$\text{C}_9\text{H}_7$	Ru	$\text{C}_9\text{H}_7$	0.55	1.00	0.61	0.58
$\text{C}_9\text{H}_7$	Ru	$\text{C}_5\text{H}_5$	0.77	1.11	0.69	0.77
$\text{C}_9\text{H}_7$	Ru	$\text{C}_5\text{Me}_5$	0.53	0.95	0.60	0.55
$\text{C}_5\text{H}_5$	Ru	$\text{C}_5\text{H}_5$	0.99 <sup>d</sup>		0.93 <sup>c,e</sup>	
$\text{C}_5\text{Me}_5$	Ru	$\text{C}_5\text{Me}_5$	0.53	1.15	0.63 <sup>f</sup>	
$\text{C}_9\text{H}_7$	Os	$\text{C}_9\text{H}_7$	0.43	1.13	0.47 <sup>f</sup>	0.47

<sup>a</sup> Irreversible one-electron oxidation, peak potential; <sup>b</sup> reversible two-electron oxidation; <sup>c</sup> irreversible two-electron oxidation, peak potential;

<sup>d</sup> Ref. [16], using  $E^0(\text{Cp}_2\text{Fe}/\text{Cp}_2\text{Fe}^+) = 0.43 \text{ V (SCE)}$ ; <sup>e</sup> Ref. [17]; <sup>f</sup> reversible one-electron oxidation.

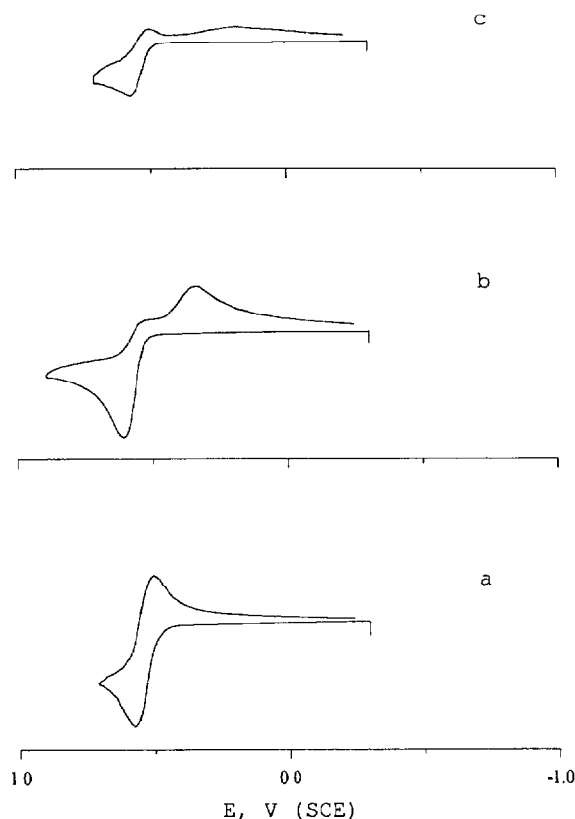
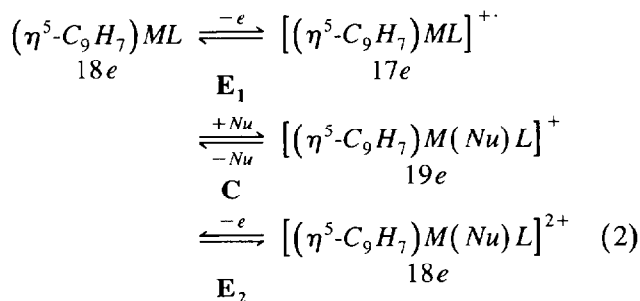


Fig. 3. Cyclic voltammograms of the complex **I** in THF/0.05 M Bu<sub>4</sub>NPF<sub>6</sub> at the Pt-electrode, scan rate 200 mV s<sup>-1</sup> at the various temperatures. Curve a at 20°C, curve b at -35°C, curve c at -90°C. Peak heights are normalized to the ferrocene oxidation peak at the same conditions.

the latter is known to be more reactive towards electrophiles. The reason for the higher reactivity of MeCN could be the smaller size of an acetonitrile molecule.

Two-electron diffusion-controlled oxidation peaks were observed in the cyclic voltammograms of complexes **I–III** at the Pt-electrode in THF, like those in MeCN. However, in contrast to the process in MeCN, two-electron oxidation of the above complexes in THF appeared to be reversible at room temperature, which was confirmed by the corresponding cathodic responses on their cyclic voltammograms ( $\Delta E = 70\text{--}80$  mV,  $i_p^c/i_p^a = 1$  at 20°C) (Fig. 3, curve a; Table 1). The temperature decrease led to a diminishing of the  $i_p^c/i_p^a$  ratio and at  $-20\text{--}-30^\circ\text{C}$  irreversible anodic two-electron peaks were observed in the cyclic voltammograms. Furthermore, the temperature decrease resulted in the appearance of broad cathodic responses on the reverse part of the cyclic voltammograms, like those in MeCN (Fig. 3, curve b). At lower temperatures this peak was not observed in practice, whereas the height of the anodic peak decreased down to that of the one-electron and the oxidation process became reversible. Such a conclusion could be made, since the corresponding cathodic response ( $\Delta E = 40$  mV,  $-70^\circ\text{C}$ ) was observed in the voltammograms (Fig. 3, curve c).

The data obtained indicate that one-electron oxidation of complexes **I–III** in THF is accompanied by a fast reversible reaction of an electroactive intermediate formation, which oxidizes more readily than the starting complex (an E<sub>1</sub>CE<sub>2</sub> process,  $E_1^0 \geq E_2^0$ ). Reversibility of the two-electron process of oxidation at room temperature indicates the fast equilibrium chemical step. Comparison of cyclic voltammograms of complexes **I–III** in CH<sub>2</sub>Cl<sub>2</sub> and THF showed the chemical step to be a reversible reaction of electrogenerated 17-electron radical cations with the solvent (Nu = THF), which yielded 19-electron radical cations,  $[(\eta^5\text{-C}_9\text{H}_7)\text{M}(\text{L})(\text{Nu})]^+$ , oxidizing to corresponding 18-electron dication at less positive potentials according to



The temperature decrease down to  $-20\text{--}-30^\circ\text{C}$  led to a sharp drop in the rate of the backward step of the reversible C-stage. The cathodic response on the cyclic voltammograms disappeared, however, the oxidation process remained two-electron (Fig. 3, curve b). With further decrease in the temperature, the rate of the forward reaction of the C-stage diminished to result in reversible one-electron oxidation of complexes **I–III** in THF at  $T < -70^\circ\text{C}$ , like that in CH<sub>2</sub>Cl<sub>2</sub> at room temperature (Fig. 3, curve c).

The difference in the behavior of complexes **I–III** in THF and CH<sub>2</sub>Cl<sub>2</sub> is due to the difference in their nucleophilic properties. It is evident that THF, which exhibits nucleophilic properties, interacts more readily with the electrogenerated 17-electron radical cations **I**<sup>•+</sup>–**III**<sup>•+</sup> than CH<sub>2</sub>Cl<sub>2</sub>, the reaction proceeding reversibly due to relatively weak nucleophilic properties of THF. Interaction of radical cations with stronger nucleophiles should shift the C-stage equilibrium to the 19-electron adduct and result in irreversible two-electron oxidation of the complexes, like that observed in MeCN (see above). An analogous situation takes place when the Cl<sup>-</sup> anion (Et<sub>3</sub>BzNCl) is added to the solution of the complexes in CH<sub>2</sub>Cl<sub>2</sub>. In the presence of Cl<sup>-</sup> ions, the anodic peak of complex **I** oxidation becomes an irreversible two-electron one and the cathodic peak ( $E_p^c = -0.65$  V) arises on the reverse part of the cyclic voltammogram. It can probably be attributed to the reduction of the 18-electron complex formed,  $[(\eta^5\text{-C}_9\text{H}_7)_2\text{RuCl}]^+$  (Fig. 1, curve b), similarly to that observed at the oxidation of  $(\eta^5\text{-C}_5\text{Me}_5)_2\text{Ru}$  in the presence of Cl<sup>-</sup> ions [10].

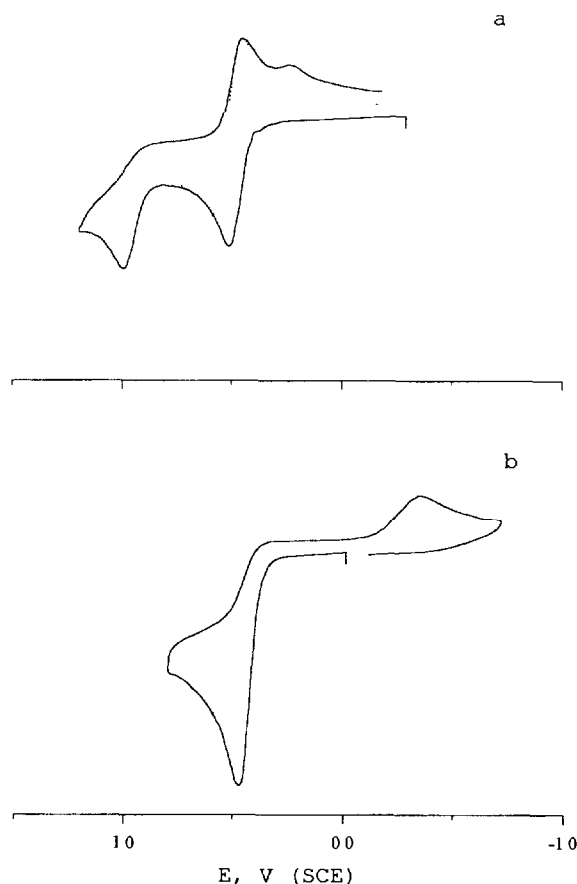


Fig. 4. Cyclic voltammograms of the complex **IV** in THF/0.1 M  $\text{Bu}_4\text{NPF}_6$  (curve a) and in MeCN/0.1 M  $\text{Bu}_4\text{NPF}_6$  (curve b) at the Pt-electrode, scan rate  $200 \text{ mV s}^{-1}$ ,  $T = 20^\circ\text{C}$ .

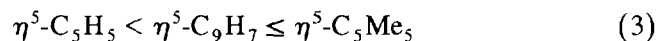
In contrast to the oxidation of complexes **I–III**, that of **IV** in THF is a reversible one-electron process even at room temperature, i.e. the radical cation  $\text{IV}^{+\cdot}$  is more inert towards interaction with THF, though its reaction with strong nucleophiles, MeCN or  $\text{Cl}^-$ , is similar to that of radical cation complexes  $\text{I}^{+\cdot}$ – $\text{III}^{+\cdot}$  (Fig. 4). Preparative one-electron oxidation of **IV** in THF (either electrochemical or chemical by  $\text{AgBF}_4$ ) yields a moderately stable brown solution, whose ESR spectrum consists of only one signal with  $g = 2.003$ .

Two-electron chemical oxidation of ruthenocene and osmocene in the presence of strong nucleophiles is known to lead to the formation of cationic complexes  $[(\eta^5\text{-C}_5\text{H}_5)_2\text{MX}]^{n+}$  ( $\text{M} = \text{Ru, Os}$ ,  $\text{X} = \text{Cl, Br, I}$ ,  $n = 1$ ;  $\text{M} = \text{Os}$ ,  $\text{X} = \text{MeCN}$ ,  $n = 2$ ) [11–13] of a bent sandwich structure [14]. Analogously one can suppose that electrogenerated indenyl complexes of  $\text{Ru}^{\text{IV}}$  and  $\text{Os}^{\text{IV}}$  and their corresponding 19-electron radical cations are also of a bent sandwich structure.

Therefore the interaction of 17-electron radical cations of  $\eta^5$ -indenyl complexes of Ru and Os with nucleophiles resulting in the formation of bent sandwich 19-electron radical cations (or radicals in the case of anionic nucleophiles, e.g.  $\text{Nu} = \text{Cl}^-$ ) facilitates much

further radical cation oxidation to the dications (or cations) stabilized by the electron pair of the nucleophile.

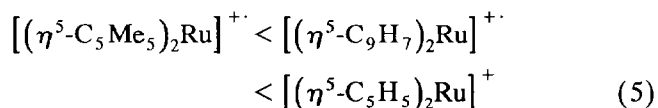
The comparison of potentials of one-electron oxidation of complexes **I–III** in  $\text{CH}_2\text{Cl}_2$  (see Table 1) indicates that the  $\pi$ -ligand electron donor ability increases in the following order:



as shown previously [6]. Hence the reactivity of corresponding 17-electron radical cation complexes  $\text{I}^{+\cdot}$ – $\text{III}^{+\cdot}$  towards nucleophiles increases in the following sequence:



The same may be declared for the 17-electron radical cations of homoligand sandwich complexes:



The latter sequence is in accordance with the electrochemical behavior of corresponding complexes. Thus ruthenocene,  $\text{Cp}_2\text{Ru}$ , is known to undergo reversible one-electron oxidation in the  $\text{AlCl}_3/\text{N-butylpyridinium chloride}$  melt [15] and/or in  $\text{CH}_2\text{Cl}_2$  with extremely weakly coordinating electrolyte  $\text{Bu}_4\text{N}^+\text{TFPB}^-$  ( $\text{TFPB}^- = \text{tetrakis[bis(3,5-trifluoromethyl)phenyl]-borate}$ ) [16], whereas in THF and MeCN media electrochemical oxidation proceeds via an irreversible two-electron step according to the ECE scheme [1,17]. Apparently, formation of 19-electron intermediate is the key step of the two-electron oxidation of ruthenocene  $\text{Cp}_2\text{Ru}$ . However, a permethylated analog of ruthenocene,  $[(\eta^5\text{-C}_5\text{Me}_5)_2\text{Ru}]$ , oxidizes reversibly to the radical cation even in MeCN [10]. Finally, as shown in the present work, the number of electrons involved into the electrochemical oxidation of bis(indenyl)ruthenium complex changes from one to two, if  $\text{CH}_2\text{Cl}_2$  is replaced by strongly coordinating solvents, e.g. THF or MeCN.

### 3. Experimental section

$^1\text{H}$  NMR spectra were acquired on a Bruker WP 200 SY spectrometer and ESR spectra were recorded on a Varian 12 spectrometer.

Electrochemical measurements were carried out on a PAR Model 173 potentiostat, a PAR Model 179 digital coulometer, and a PAR Model 175 function generator. A home-made three-electrode low-temperature electrochemical cell was used, equipped with a Pt-disk (1 mm diameter) working electrode, Ag/AgCl reference electrode, and Pt-foil counter electrode. The working electrode was polished with diamond paste (down to  $1 \mu\text{m}$

in diameter) and carefully washed before use. The reference electrode was separated from the main solution by a salt bridge having a graphite rod sealed into the glass tip and filled with a supporting electrolyte solution to prevent contamination of the solution with traces of water and chlorine ions. The reference electrode was kept at room temperature during low-temperature measurements. All potentials are given vs. SCE and referred to the  $\text{Cp}_2\text{Fe}/\text{Cp}_2\text{Fe}^+$  or  $\text{Cp}_2^*\text{Fe}/\text{Cp}_2^*\text{Fe}^+$  couples, whose potentials are +0.43, +0.44, +0.42 and –0.10, 0.00, –0.08 V (SCE) in  $\text{CH}_2\text{Cl}_2$ , THF and MeCN respectively. Electrolysis at controlled potentials was performed on a P-5827M potentiostat and Radelkis OH-404 digital coulometer in a two-compartment cylindrical cell equipped with a fritted glass diaphragm. A Pt-gauze working electrode of area  $35\text{ cm}^2$  was used.

### 3.1. Materials and procedures

All the procedures were carried out under argon. Dichloromethane was purified as described in Ref. [18] and stored under argon in the dark at  $0^\circ\text{C}$ . Tetrahydrofuran was distilled from sodium-benzophenone immediately prior to its use. Acetonitrile was purified by the procedure in Ref. [19], stored over calcium hydride and distilled immediately prior to use.  $\text{Bu}_4\text{NPF}_6$  (Fluka) was used as a supporting electrolyte and dried by melting in vacuo immediately prior to solvent distillation. The purified solvent ( $\text{CH}_2\text{Cl}_2$ , MeCN or THF) was distilled under argon into a special burette with the supporting electrolyte salt and transferred into an electrochemical air-proof cell, preliminarily dried in vacuo and filled with argon. This procedure prevents the solution from contamination by traces of air and/or water. For low-temperature measurements the cell was thermostatted with ethanol, bath cooled with liquid nitrogen in a Dewar vessel.

### 3.2. Synthesis

Complex **I** was synthesized according to Ref. [20]. Complexes **II** and **III** were synthesized in the reaction of indenyllithium with either  $[(\eta^5 - \text{C}_5\text{R}_5)\text{Ru}(\text{MeCN})_3]\text{PF}_6$  ( $\text{R} = \text{H}, \text{Me}$ ) or  $[(\eta^5 - \text{C}_5\text{Me}_5)\text{RuCl}_2]_x$  according to Ref. [21].

#### 3.2.1. Preparation of $(\eta^5 - \text{C}_9\text{H}_7)_2\text{Os}$

3 g of Zn dust were added in portions to the mixture of 2.2 g (2.48 mmol)  $(\text{Bu}_4\text{N})_2\text{OsCl}_6$  and 6 ml of indene in 100 ml MeOH/EtOH (1:1). The reaction mixture was stirred for 0.5 h at room temperature, 2 h at  $45^\circ\text{C}$ , and then refluxed for 4 h. After cooling the solvent was evaporated and the residue extracted with a petroleum

ether/benzene (1:1) mixture. The extract was evaporated and a mixture of crystals and an oil-like product obtained. The residue was washed with a cold mixture of  $\text{Me}_2(\text{CO})/\text{EtOH}$  (1:1), the crystals being filtered off, dissolved in  $\text{CH}_2\text{Cl}_2$  and precipitated by ether. After recrystallization from the MeOH/ $\text{Me}_2\text{CO}$  (4:1) mixture, 86 mg (11% yield) of complex **IV** (golden-brown crystals) were obtained. Anal. Found: C, 51.51; H, 3.42; Os, 45.58.  $\text{C}_{18}\text{H}_{14}\text{Os}$  Calc.: C, 51.41; H, 3.36; Os, 45.23%.  $^1\text{H}$  NMR ( $(\text{CD}_3)_2\text{CO}$ ): 4.84 (t,  $1\text{H}_b$ , 1.8 Hz); 5.15 (d,  $2\text{H}_a$ , 2.2 Hz); 6.42–6.47 (t,  $2\text{H}_{\alpha(\beta)}$ ); 6.57–6.62 (t,  $2\text{H}_{\beta(\alpha)}$ ).

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