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Ruthenium(II) complexes containing 4-ferrocenylphenylisocyanide ligands. Crystal structure of *trans*, *trans*, *trans*-[$RuCl_2(POMe–P)_2(FcC_6H_4NC)_2$] (POMe = $PPh_2C_6H_4OCH_3$; Fc = ferrocenyl)

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Abstract

The ruthenium complexes *trans*, *trans*, *trans*-[$RuCl_2(POMe–P)_2(FcC_6H_4NC)_2$] (**1**) (POMe = $PPh_2C_6H_4OCH_3$; Fc = ferrocenyl) and *trans*, *trans*, *trans*-[$RuCl_2(PC2OMe–P)_2(FcC_6H_4NC)_2$] (**2**) (PC2OMe = $PPh_2CH_2CH_2OCH_3$) have been prepared by reaction of FcC_6H_4NC with [$RuCl_2(POMe–P,O)_2$] (**5**) and [$RuCl_2(PC2OMe–P,O)_2$] (**6**), respectively. The mixed carbonyl–isocyanide complexes *trans*, *trans*, *trans*-[$RuCl_2(POMe–P)_2(CO)(FcC_6H_4NC)$] (**3**) and *trans*, *trans*, *trans*-[$RuCl_2(PC2OMe–P)_2(CO)(FcC_6H_4NC)$] (**4**) were prepared by sequential reaction of **5** and **6** with carbon monoxide and FcC_6H_4NC, respectively. These complexes have all been characterized by ^{1}H-NMR, ^{31}P-NMR and infrared spectroscopies as well as microanalysis. The solid-state structure of **1** was determined by X-ray crystallography. All three pairs of ligands are *trans* in this complex, and the ferrocenyl groups are tilted with respect to the phenyl ring plane by 28°. Cyclic voltammetry of these complexes reveals essentially no electronic interaction occurs between the ferrocenyl groups and the Ru.

Keywords: Isocyanide; Ruthenium; Ferrocene; Cyclic voltammetry

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1. Introduction

Transition metal-containing polymers in which the metals are linked by π-conjugated bridges are an important class of materials [1]. The contribution of the metal to the electronic properties of such polymers may result in novel functions and applications. These materials are related to π-conjugated organic polymers [2], which become conductive upon doping, and so the question of which structural elements are prerequisites for similar properties in metal-bearing polymers is an important one. One approach to this issue is to build and study model complexes which can be used to probe charge delocalization across metal and organic bridges, thus allowing the identification of units which facilitate or impede charge transport.

We and others have previously investigated the charge delocalization over metal groups which span two ferrocenyl moieties [3–8]. In these studies, the ferrocenyl groups serve as redox probes, and are selected because of their stability in dual redox states as well as their synthetic manipulability. For instance, in a series of ruthenium bis(ferrocenylacetylide) complexes delocalization of charge between the two ferrocenyl groups is observed both by cyclic voltammetry and near-IR spectroscopy [5].

In this paper, we report the preparation and characterization of complexes in which a ruthenium bis(phenylisocyanide) bridge spans two ferrocenyl groups (**1** and **2**), as well as two carbonyl derivatives **3** and **4**. Cyclic voltammetry of **1**–**4** is used to probe possible charge delocalization between metal centers.

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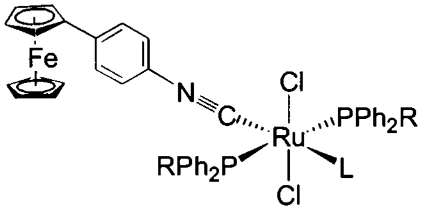


1 R = o-C_6H_4OMe L = FcC_6H_4NC
2 R = CH_2CH_2OMe L = FcC_6H_4NC
3 R = o-C_6H_4OMe L = CO
4 R = CH_2CH_2OMe L = CO

Fc = ferrocenyl

2. Experimental

2.1. General

$[RuCl_2(POMe$–$P,O)_2]$ (**5**) (POMe = $PPh_2C_6H_4OCH_3$) [9], $[RuCl_2(PC2OMe$–$P,O)_2]$ (**6**) (PC2OMe = $PPh_2CH_2CH_2OCH_3$) [10], and 4-FcC_6H_4NC (Fc = ferrocenyl) [11,12] were all prepared using literature procedures. All other reagents were purchased from either Strem Chemicals or Aldrich and used as received. IR spectra were obtained on a Bomem MB-series spectrometer in CH_2Cl_2 solution or CsI pellets. ^1H- and $^{31}P\{^1H\}$-NMR experiments were performed on a Bruker CPX-200 spectrometer. Spectra were referenced to residual solvent (^1H) or external 85% H_3PO_4 (^{31}P). Samples for elemental analysis were kept under vacuum for several days in order to completely remove trace solvents and water. Electrochemical measurements were conducted on a Pine AFCBP1 bipotentiostat using a Pt disc working electrode, Pt coil wire counter electrode and a silver wire reference electrode. An internal reference (decamethylferrocene) was added to correct the measured potentials with respect to saturated calomel electrode (SCE). The supporting electrolyte was 0.1 M $[(n\text{-}Bu)_4N]PF_6$, which was purified by triple recrystallization from EtOH and dried at 90 °C under vacuum for 3 days. Methylene chloride used in cyclic voltammetry was dried by refluxing over CaH_2.

2.2. *trans*, *trans*, *trans*-$[RuCl_2(POMe$–$P)_2(FcC_6H_4NC)_2]$ (1)

A solution of **5** (105 mg, 0.14 mmol) and FcC_6H_4NC (80 mg, 0.28 mmol) in dry CH_2Cl_2 (50 ml) was stirred at 25 °C under nitrogen for 2.5 h. The ruby-red solution was then concentrated to ca. 5 ml and hexanes added until the solution became cloudy. The product slowly crystallized from this solution at 25 °C yielding dark orange microcrystals. Yield: 121 mg (65%). ^1H-NMR (200 MHz, $CDCl_3$): $\delta = 8.14$ (m, o-PC_6H_5, 4H), 7.28 (m, 6H, m,p-PC_6H_5 and $PC_6H_4OCH_3$), 7.21 (d, $J_{HH} =$ 8.54 Hz, 2H, FcC_6H_4NC), 6.85 (m, 2H, $PC_6H_4OCH_3$), 6.41 (d, $J_{HH} = 8.54$ Hz, 2H, FcC_6H_4NC), 4.58 (t, $J_{HH} =$ 1.79 Hz, 2H, C_5H_4), 4.33 (t, $J_{HH} = 1.79$ Hz, 2H, C_5H_4), 4.01 (s, 5H, Cp), 3.51 (s, 3H, OCH_3). $^{31}P\{^1H\}$-NMR (81.015 MHz, $CDCl_3$): $\delta =$ 3D 29.72 (s). Anal. Found: C, 65.10; H, 4.66; N 2.01. Calc. for $C_{72}H_{60}Cl_2N_2O_2P_2Fe_2Ru$: C, 64.96; H, 4.51; N, 2.11%.

2.3. *trans*, *trans*, *trans*-$[RuCl_2(PC2OMe$–$P)_2(FcC_6H_4NC)_2]$ (2)

A solution of **6** (94 mg, 0.14 mmol) and FcC_6H_4NC (80 mg, 0.28 mmol) in dry CH_2Cl_2 (15 ml) was stirred at 25 °C under nitrogen for 2.5 h. The red solution was concentrated to ca. 1 ml and hexanes added until the solution became cloudy. The product slowly crystallized from this solution at 25 °C to yield brick red microcrystals. Yield: 108 mg (63%). ^1H-NMR (200 MHz, $CDCl_3$): $\delta = 7.86$ (m, 4H, o-PC_6H_5), 7.28 (m, 8H, m,p-PC_6H_5 and FcC_6H_4NC), 6.65 (d, $J_{HH} = 8.50$ Hz, 2H, FcC_6H_4NC), 4.60 (t, $J_{HH} = 1.80$ Hz, 2H, C_5H_4), 4.35 (t, $J_{HH} = 1.80$ Hz, 2H, C_5H_4), 4.01 (s, 5H, Cp), 3.56 (m, 2H, PCH_2), 3.13 (s, 3H, OCH_3), 3.08 (m, 2H, CH_2OCH_3). $^{31}P\{^1H\}$-NMR (81.015 MHz, $CDCl_3$): $\delta =$ 15.03 (s). Anal. Found: C, 61.87; H, 4.97; N, 2.16. Calc. for $C_{64}H_{60}Cl_2N_2O_2P_2Fe_2Ru$: C, 62.24; H, 4.86; N, 2.27%.

2.4. *trans*, *trans*, *trans*-$[RuCl_2(POMe$–$P)_2(CO)(FcC_6H_4NC)]$ (3)

Carbon monoxide was bubbled through a solution of **5** (40 mg, 0.053 mmol) in CH_2Cl_2 (15 ml) for 40 min at 25 °C. The mixture was then filtered through Celite and nitrogen was bubbled through the solution for 2 h. FcC_6H_4NC (15 mg, 0.053 mmol) was then added and the resulting solution stirred at 25 °C for 1 h. The mixture was concentrated to 1–2 ml and hexanes added to precipitate **3** as brick red microcrystals. Yield: 31 mg (55%). ^1H-NMR (200 MHz, $CDCl_3$): $\delta = 8.00$ (m, 8H, o-PC_6H_5), 7.31 (m, 16H, m,p-PC_6H_5, $PC_6H_4OCH_3$), 7.22 (d, $J_{HH} = 8.50$ Hz, 2H, FcC_6H_4NC), 6.87 (m, 4H, $PC_6H_4OCH_3$), 6.35 (d, $J_{HH} = 8.50$ Hz, 2H, FcC_6H_4NC), 4.58 (t, $J_{HH} = 1.80$ Hz, 2H, C_5H_4), 4.35 (t, $J_{HH} = 1.80$ Hz, 2H, C_5H_4), 4.00 (s, 5H, Cp), 3.57 (s, 6H, OCH_3). $^{31}P\{^1H\}$-NMR (81.015 MHz, $CDCl_3$): $\delta = 26.21$ (s). Anal. Found: C, 62.63; H, 4.24; N, 1.13. Calc. for $C_{56}H_{47}Cl_2NO_3P_2FeRu$: C, 62.74; H, 4.39; N, 1.31%.

2.5. *trans*, *trans*, *trans*-$[RuCl_2(PC2OMe$–$P)_2(CO)(FcC_6H_4NC)]$ (4)

Carbon monoxide was bubbled through a solution of **6** (54.3 mg, 0.082 mmol) in dry CH_2Cl_2 (15 ml) for 5 min at 0 °C. Nitrogen was then bubbled through the solution for 30 min at 0 °C. The mixture was then

Table 1
Crystallographic and structural refinement data for **1**·6CH_2Cl_2

Empirical formula	$C_{78}H_{72}Cl_{14}Fe_2N_2O_2P_2Ru$
Formula weight	1840.39
Temperature (K)	203(2)
Wavelength (Å)	0.71073
Crystal system	Monoclinic
Space group	*C*2/*c*
Unit cell dimensions	
a (Å)	34.631(4)
b (Å)	12.007(9)
c (Å)	22.278(6)
α (°)	90
β (°)	117.976(7)
γ (°)	90
V (Å^3)	8181(6)
Z	4
D_{calc} (Mg m^{-3})	1.494
Absorption coefficient (mm^{-1})	1.075
F(000)	3736
Crystal size (mm)	0.10 × 0.10 × 0.10
Theta range for data collection (°)	1.33–22.50
Limiting indices	$-37 \leq h \leq 32$, $0 \leq k \leq 12$, $0 \leq l \leq 23$
Reflections collected/unique	9996/5338 [$R_{int} = 0.0943$]
Completeness to $2\theta = 22.50$	99.8%
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	2385/0/391
Goodness-of-fit on F^2	1.022
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0690$, $wR_2 = 0.1925$
R indices (all data)	$R_1 = 0.0962$, $wR_2 = 0.2060$
Largest different peak and hole (e Å^3)	1.054 and −0.993

allowed to warm to 25 °C, and FcC_6H_4NC (23.5 mg, 0.07 mmol) added. The resulting solution was stirred for 1 h. The mixture was concentrated to 1–2 ml and hexanes added to precipitate **4** as an orange powder. Yield: 55 mg (69%). ^{1}H-NMR (200 MHz, $CDCl_3$): $\delta = 7.77$ (m, 8H, *o*-PC_6H_5), 7.30 (m, 12H, *m,p*-PC_6H_5), 7.23 (d, $J_{HH} = 8.42$ Hz, 2H, FcC_6H_4NC), 6.49 (d, $J_{HH} = 8.42$ Hz, 2H, FcC_6H_4NC), 4.61 (t, $J_{HH} = 1.76$ Hz, 2H, C_5H_4), 4.37 (t, $J_{HH} = 1.76$ Hz, 2H, C_5H_4), 4.00 (s, 5H, Cp), 3.51 (m, 4H, PCH_2), 3.17 (s, 6H, OCH_3), 3.01 (m, 4H, CH_2OCH_3). $^{31}P\{^1H\}$-NMR (81.015 MHz, $CDCl_3$): $\delta =$ 3D 13.80 (s). Anal. Found: C, 58.74; H, 4.88; N, 1.32. Calc. for $C_{48}H_{47}Cl_2NO_3P_2FeRu$: C, 59.07; H, 4.82; N, 1.44%.

2.6. Crystallographic study

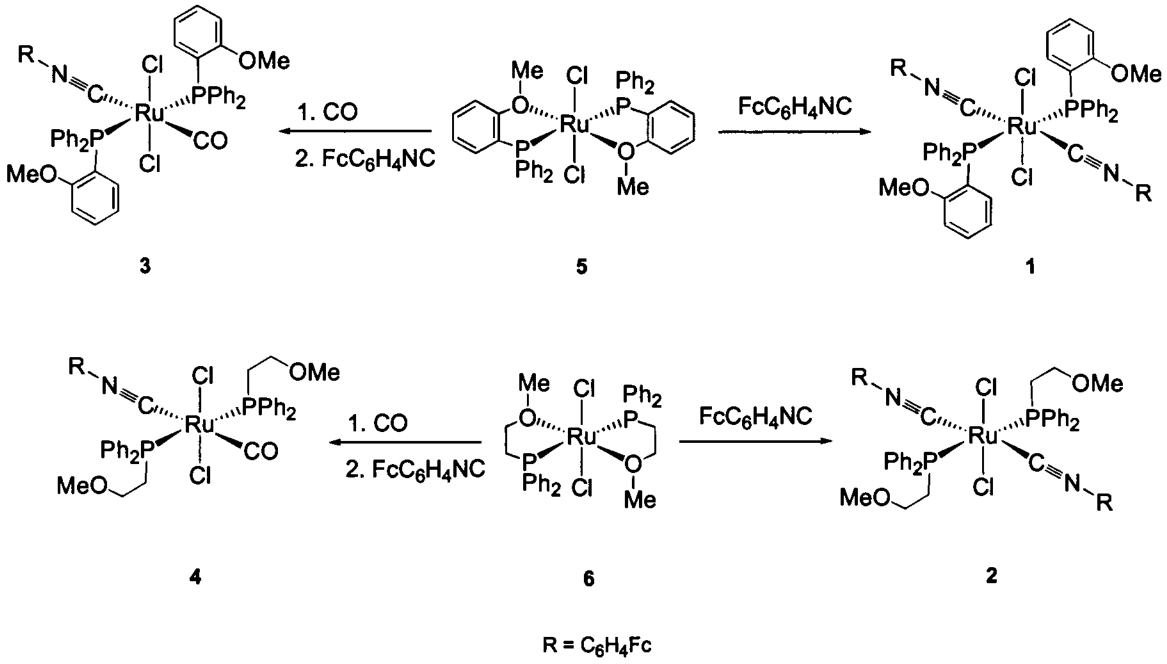
A suitable crystal of **1** which had been crystallized from layered CH_2Cl_2 and hexanes was selected, mounted on a thin glass fiber using paraffin oil and cooled to the data collection temperature. Compound **1** consistently yields small, multiple crystals and this data-set represents the best obtained after several trials. Data were collected on a Bruker AX SMART 1k CCD diffractometer using 0.3° ω-scans at 0, 90, and 180° in ϕ. Unit-cell parameters were determined from 60 data frames collected at different sections of the Ewald sphere. Systematic absences in the diffraction data and unit-cell parameters were consistent with space groups *Cc* and *C*2/*c*. Solution in the centric option yielded chemically reasonable and computationally stable results of refinement. Absorption correction based on redundant data [13] was initially applied but later ignored when the resulting T_{min}/T_{max} ratio was unity. The structure was solved by direct methods, completed with difference Fourier syntheses and refined with full-matrix least-squares procedures based on F^2. The molecule is located at an inversion center. All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were treated as idealized contributions. All scattering factors and anomalous dispersion factors are contained in the SHEXTL 5.10 program library [14]. Crystal data and refinement parameters are summarized in Table 1.

3. Results

3.1. Synthesis and structure of **1** and **2**

The complexes **1** and **2** were synthesized by reaction of 4-ferrocenylphenylisocyanide (FcC_6H_4NC) with **5** and **6**, respectively (Scheme 1). The oxygen of the hemilabile phosphine–ether ligand is displaced by the isocyanide while the phosphorus remains coordinated to the ruthenium. The structure of **1**·6CH_2Cl_2 was unambiguously determined by X-ray crystallography (Fig. 1). The ruthenium in **1** is in a slightly distorted octahedral environment and lies at an inversion center, with the three pairs of ligands oriented *trans* to each other. Both ferrocenyl groups are tilted with respect to the phenyl ring plane with a dihedral angle of 28° (Fig. 2).

The ^{31}P-NMR spectrum of **2** consists of a singlet at $\delta = 29.7$ and the IR spectrum contains one band in the N≡C stretching region at $\nu_{NC} = 2092$ cm^{-1}. An all-*trans* product was also obtained by others when **5** was reacted with *t*-BuNC [15]. The spectral data obtained for **2** indicates that this complex also has an all-*trans* configuration. Lindner has previously reacted *t*-BuNC with **6** and proposed that the obtained product is [$RuCl_2$(PC2OMe–*P*)$_2$(*t*-BuNC)$_2$] with the phosphine and chloro ligands both in a *cis* orientation, based on the presence of an assymetric (310 cm^{-1}) and symmetric (262 cm^{-1}) Ru–Cl_2 stretch in the infrared region [16]. The far-IR spectra of **1** and **2** both contain a medium strength absorption at ca. 315 cm^{-1} which we assign to a *trans* $RuCl_2$ stretch. In addition there are several weak absorptions in this region (see Table 2) and a band at 310 cm^{-1} which is present in the spectra



Scheme 1.

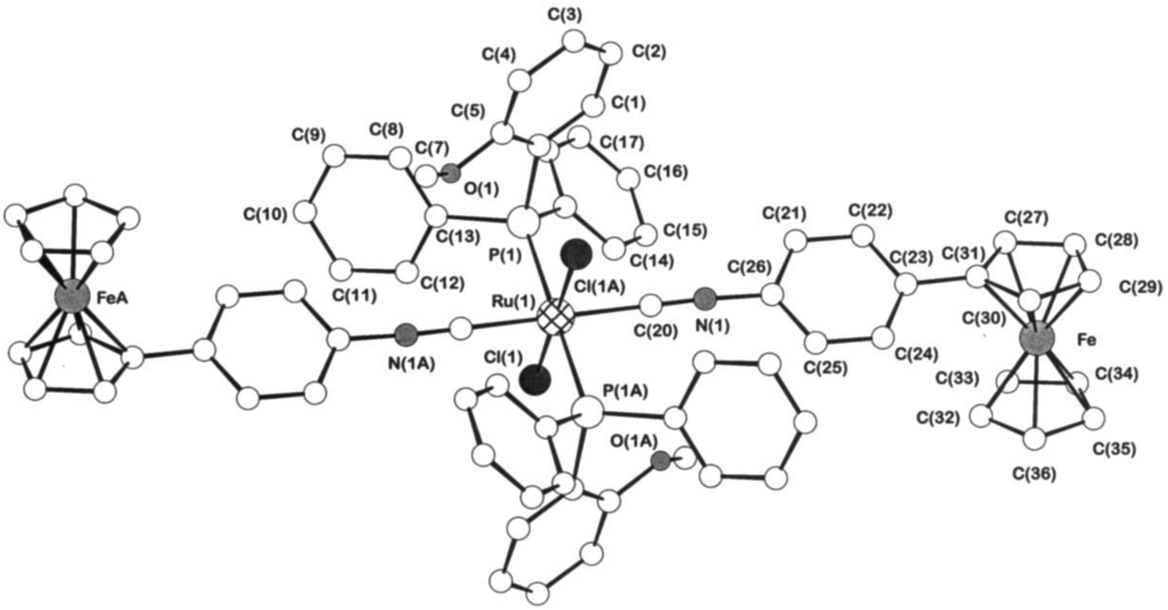


Fig. 1. Chem 3D representation of **1**. Hydrogen atoms are omitted for clarity. Selected bond lengths (Å): Ru–Cl(1) = 2.4249(19), Ru–C(20) = 1.978(8), Ru–P(1) = 2.383(2), C(20)–N(1) = 1.138(8), and N(1)–C(26) = 1.394(7). Selected bond angles (°): Cl(1)–Ru–Cl(1A) = 180.00(8), C(20)–Ru–C(20A) = 180.0(4), P(1)–Ru–P(1A) = 180.00(8), Cl(1)–Ru–P(1) = 87.32(7), Ru–C(20)–N(1) = 178.6(6), and C(20)–N(1)–C(26) = 176.9(7).

of **1**–**4** as well as in other Ru carbonyl complexes and is therefore likely a Ru–C stretch. The similarities between the spectra of **1** and **2** support the assignment of **2** as an all *trans* isomer.

*3.2. Synthesis and structure of **3** and **4***

Reaction of **5** or **6** with carbon monoxide yields an equilibrium mixture of the monocarbonyl and dicarbonyl complexes in both cases. Bubbling nitrogen through this mixture drives the equilibrium to the monocarbonyl complex, to which FcC_6H_4NC is added yielding complexes **3** and **4**, respectively (Scheme 1). The ^{31}P-NMR spectra of **3** and **4** contain only one singlet which indicates that the phosphorus nuclei are equivalent. The IR spectra of these complexes contain

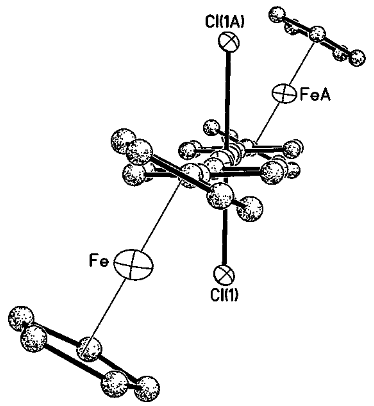


Fig. 2. ORTEP projection of **1** (30% probability ellipsoid shown) along the NC–Ru–CN axis. The phosphine and chloro ligands are omitted for clarity.

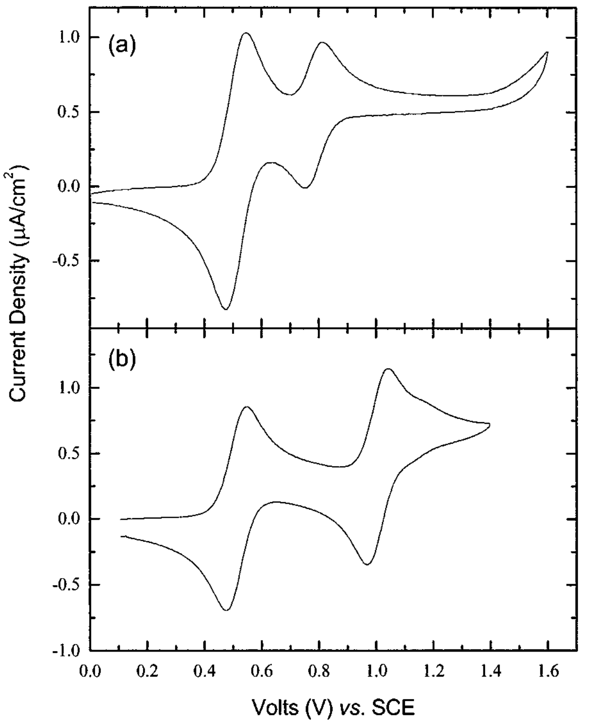


Fig. 3. Cyclic voltammogram of (a) **1** and (b) **3** in CH_2Cl_2 containing 0.1 M[(*n*-Bu)$_4$]PF_6. Scan rate = 50 mV s^{-1}.

two bands between 1900 and 2200 cm^{-1} (Table 1). The higher energy band in each case is assigned to the CO stretch and the lower energy band is assigned to the NC stretch, and these bands appear close to those previously observed for *trans*, *trans*, *trans*-[$RuCl_2$-(PC2OMe)$_2$(CO)(*t*-BuNC)] by Lindner [16]. The far-IR spectra of **3** and **4** contain medium strength absorptions at 327 and 325 cm^{-1}, respectively, indicating the chloro ligands are oriented *trans* in these complexes. The far-IR spectra of *trans*, *trans*, *trans*-[$RuCl_2$(POMe)$_2$-(CO)(*t*-BuNC)] and *trans*, *trans*, *trans*-[$RuCl_2$-(PC2OMe)$_2$(CO)(*t*-BuNC)] have bands at 324 and 320 cm^{-1}, respectively [15,16].

3.3. Cyclic voltammetry

The cyclic voltammograms (CVs) of both **1** (Fig. 3a) and **2** contain two reversible oxidation waves (Table 2). The lower potential wave for both complexes is approximately twice the intensity of the second wave, and is consequently assigned to the simultaneous oxidation of the two ferrocenyl groups. The second wave is then assigned to the reversible Ru$^{II/III}$ oxidation. The CVs of **3** (Fig. 3b) and **4** also contain two reversible waves of equal area, and the lower potential wave is again assigned to the oxidation of the ferrocenyl group. The higher potential wave corresponds to the Ru$^{II/III}$ oxidation, shifted positive relative to the corresponding wave in **1** and **2** due to the presence of the electron withdrawing carbonyl group.

4. Discussion and conclusions

The electrochemical data for these complexes may be used to assess the degree of interaction between the ferrocenyl groups. In complexes **1** and **2**, the simultaneous oxidation of the two ferrocenyl groups indicates that interaction between these groups over the rigid Ru bis(phenylisocyanide) bridge is small. This is in contrast with the relatively large interaction observed for bis-

Table 2
Infrared and cyclic voltammetric data for **1**–**4**

Complex	Selected IR [a] and FIR [b] bands [c] (cm^{-1})	$E_{1/2}$(Fe) [d] (V)	$E_{1/2}$(Ru) [d] (V)
1	2098 (s, CN), 317 (m, $RuCl_2$)	0.51	0.73
2	2092 (s, CN), 315 (m, $RuCl_2$)	0.51	0.90
3	2001 (s, CO), 2166 (s, CN), 327 (m, $RuCl_2$)	0.53	1.03
4	1983 (s, CO), 2178 (s, CN), 325 (m, $RuCl_2$)	0.54	1.19

[a] In CH_2Cl_2.

[b] In CsI, additional far-IR bands (cm^{-1}): **1**, 310 (m), 291 (w); **2**, 310 (sh), 297 (w); **3**, 310 (m); and **4**, 312 (m) (s, strong; m, medium; w, weak; sh, shoulder).

[c] ±5 cm^{-1}.

[d] ±0.01 V.

(ferrocenylacetylide) complexes of Ru, in which the difference in the oxidation potentials of the ferrocenyl centers is between 90 and 220 mV [5], however in these complexes the bridge between the metal centers is shorter. A second indication that the electronic interaction across the phenylisocyanide bridge is small, comes from the observation that the ferrocenyl oxidation potential of the ligand is invariant to the change in the ligand *trans* to it on the Ru. The ferrocenyl oxidation potential is essentially the same when the *trans* ligand is a donor (RNC in **1** and **2**) or an acceptor (CO in **3** and **4**). This same change results in a ca. 0.3 V increase in the oxidation potential of the Ru, and a substantial shift in the CN stretching frequency of the coordinated ferrocenylphenylisocyanide.

It is possible that the lack of interaction between metal centers is a consequence of poor coupling between the substituted phenyl ring and the NC bond. This has been previously observed by Cotton who showed that in a series of metal complexes containing *para*-substituted phenylisocyanides, the substituents on the phenyl group had almost no effect on the NC frequency either in the free isocyanides or in the ligands [17]. Furthermore, in the solid-state the phenyl ring plane is tilted with respect to the ferrocenyl group by 28° (Fig. 2), suggesting that π-conjugation between these rings is also not optimal. On the other hand, an electrochemical study of the chromium complex [(FcNC)Cr$(CO)_5$] in which the ferrocenyl group in the ligand is directly attached to the isocyanide group, showed no shift in the ferrocenyl oxidation potential relative to FcNC [11]. This suggests that in complexes where the isocyanide is a σ-donor, with no back-bonding from the metal, there is effectively no conjugation between the metal and ligand preventing electronic interaction between the ferrocenyl group and the metal center. The greater degree of interaction observed for the acetylide complexes may therefore be due to the back-bonding which occurs in many cases, thus allowing dπ–pπ conjugation.

5. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC no. 153354. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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