

JOM 23701

Synthesis and reactivity towards tetrafluoroboric acid of a new family of heterobimetallic polyhydride complexes

$[(\text{CO})(\text{PPh}_3)_2\text{HRe}(\mu\text{-H})_3\text{RhL}_2]$ ($\text{L} = \text{PPh}_3$, 1,2,5-triphenylphosphole, or $\text{P}(\text{OMe})_3$)

Zhongli He, Denis Neibecker and René Mathieu

Laboratoire de Chimie de Coordination du CNRS, UPR 8241, liée par conventions à l'Université Paul Sabatier et à l'Institut National Polytechnique, 205 Route de Narbonne, 31077 Toulouse Cédex (France)

(Received January 14, 1993)

Abstract

The reaction of $\text{K}[\text{ReH}_6(\text{PPh}_3)_2]$ with $[\text{RhCl}(\text{CO})\text{L}_2]$ [$\text{L} = \text{PPh}_3$, 1,2,5-triphenylphosphole (TPP), or $\text{P}(\text{OMe})_3$] leads to the new electronically unsaturated heterobimetallic polyhydride complexes $[(\text{CO})(\text{PPh}_3)_2\text{HRe}(\mu\text{-H})_3\text{RhL}_2]$ in moderate-to-good yields. The structures of these complexes have been established on the basis of spectroscopic data, especially ^1H and ^{31}P NMR. The bridging hydride ligands are fluxional but there is either a slow or nonexistent exchange between terminal and bridging hydrides. For $\text{L} = \text{PPh}_3$ or TPP, protonation with tetrafluoroboric acid affords quantitatively the cationic complexes $[(\text{CO})(\text{PPh}_3)_2\text{HRe}(\mu\text{-H})_3\text{RhHL}_2]^+$, isolated as the BF_4^- or the BPh_4^- salts.

1. Introduction

Bimetallic polyhydride complexes have recently attracted interest because the presence of terminal and bridging hydride ligands should favour reactivity [1]. Except for the expected conventional reactivity of hydride ligands towards unsaturated organic molecules, polyhydrides usually generate unsaturated complexes as they often quite easily eliminate molecular hydrogen under photolysis [2] or protonation [3].

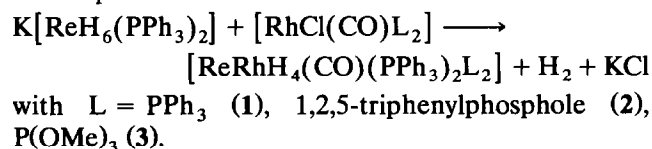
For these reasons we have tried to build new heterobimetallic polyhydride complexes retaining as the building block the complex $[\text{ReH}_7(\text{PPh}_3)_2]$ which can be easily deprotonated to $[\text{ReH}_6(\text{PPh}_3)_2]^-$ [1b]. In a search for a bimetallic complex containing rhenium and a metal able easily to activate hydrogen, we have recently observed that $[\text{ReH}_6(\text{PPh}_3)_2]^-$ reacts with $[\text{RuHCl}(\text{CO})(\text{PPh}_3)_3]$ leading to the new unsaturated complex $[(\text{CO})(\text{PPh}_3)_2\text{HRe}(\mu\text{-H})_3\text{RuH}(\text{PPh}_3)_2]$ in good yield [4,5].

In order to extend this family of bimetallic complexes, we have studied the reaction of $[\text{ReH}_6(\text{PPh}_3)_2]^-$ with some halidocarbonyl complexes $[\text{RhCl}(\text{CO})\text{L}_2]$ with $\text{L} = \text{PPh}_3$, 1,2,5-triphenylphosphole (TPP), or

$\text{P}(\text{OMe})_3$. The reactivity of the resulting new heterobimetallic complexes towards tetrafluoroboric acid has also been examined.

2. Results and discussion**2.1. Synthesis of new heterobimetallic rhenium–rhodium polyhydride complexes**

$\text{K}[\text{ReH}_6(\text{PPh}_3)_2]$ reacts at room temperature in THF solution with $[\text{RhCl}(\text{CO})\text{L}_2]$ to give new heterobimetallic complexes



The yield is good for 1 and 2 (63%) but somewhat low for 3 (36%). Moreover, the last complex is quite unstable and has been characterized only by spectroscopic data.

These complexes have 30 valence electrons and are isoelectronic with the previously reported $[(\text{CO})(\text{PPh}_3)_2\text{HRe}(\mu\text{-H})_3\text{RuH}(\text{PPh}_3)_2]$ [4,5].

As we have not been able to obtain good crystals for X-ray structure determination, the structures of these complexes have been inferred from spectroscopic data and especially from ^1H NMR spectroscopy with selec-

Correspondence to: Dr. R. Mathieu.

tive phosphorus decoupling. These data are collected in Table 1.

In the IR spectra, the position of the carbonyl absorption is quite similar for all three complexes and is characterized by a significant shift towards lower wave numbers as compared to the starting complexes. This is consistent with a migration of the CO molecule from rhodium to rhenium, a phenomenon already observed in the synthesis of $[(\text{CO})(\text{PPh}_3)_2\text{HRe}(\mu\text{-H})_3\text{RuH}(\text{PPh}_3)_2]$. It is also consistent with the weak influence of the electronic properties of ligands bound to rhodium on the position of the IR absorption of the carbonyl group.

The NMR data for the three complexes are very similar, suggesting similar structures.

In the ^1H NMR spectra at room temperature, two signals are observed in the hydride region: a broad signal ($\text{H}^1 + \text{H}^2 + \text{H}^4$) and a triplet (H^3) in the intensity ratio of 3/1. The triplet is due to coupling with the two phosphorus atoms bound to rhenium and, as in the Re–Ru complex, is characteristic of a terminal hydride on rhenium.

In the $^{31}\text{P}\{^1\text{H}\}$ NMR spectra at room temperature, two broad doublets of doublets are observed for the ligands P^1 and P^2 bound to rhodium and one doublet due to a small coupling with rhodium is apparent for phosphines P^3 and P^4 bound to rhenium. This weak coupling confirms the dinuclear nature of the complexes.

On lowering the temperature to 193 K we observe in the ^1H NMR spectra of each complex the splitting of the broad resonance into two resonances, in the ratio 2/1. The resonance of intensity 1 (H^4) appears at higher field and is a doublet of doublets due to coupling with rhodium and with one phosphorus bound to rhodium. The resonance of intensity 2 ($\text{H}^1 + \text{H}^2$) is an apparent doublet due to coupling with rhodium. These data are consistent with three exchanging bridging hydrides. It should be pointed out that the chemical shift of the resonance of intensity 2 is around 0 ppm for complexes **1** and **2**. This shift is quite unusual for hydrides bridging these metals, although it has recently been reported that for the $[\text{Re}_2(\mu\text{-H})(\mu\text{-Cl})(\mu\text{-Ph}_2\text{-PCH}_2\text{CH}_2\text{PPh}_2)\text{Cl}_2(\text{CO})_2]$ the hydride resonance is at 12.75 ppm [6].

The resonance of the hydride (H^3) bound to rhenium is only slightly affected by lowering the temperature and, as in the Re–Ru complex already mentioned [7], there is at the most a slow exchange between this hydride and the three bridging hydrides H^1 , H^2 and H^4 .

Lowering the temperature to 193 K does not affect the $^{31}\text{P}\{^1\text{H}\}$ NMR spectra of complexes **1**, **2** and **3** greatly, except for sharpening of the resonances of P^1 and P^2 bound to rhodium.

From all these spectroscopic data, we propose the structure shown in Fig. 1 for these dinuclear complexes. In this structure the geometry around rhodium

TABLE 1. Spectroscopic data for the complexes $[(\text{CO})(\text{PPh}_3)_2\text{HRe}(\mu\text{-H})_3\text{RhL}_2]$ [**1**, L = PPh_3 ; **2**, L = TPP; **3**, L = $\text{P}(\text{OMe})_3$]

Complex	IR $\nu(\text{CO})$ (cm^{-1})	$^1\text{H}^{\text{b,c}}$ NMR δ (ppm); J (Hz)	$^{31}\text{P}\{^1\text{H}\}^{\text{b}}$ NMR δ (ppm); J (Hz)	T (K)
1	1847	$\text{H}^3 - 3.94$ (t); $J(\text{P-H}) = 44.0$	$\text{P}^1 55.8$ (d)	296
		$\text{H}^1 + \text{H}^2 + \text{H}^4 - 4.10$ (b)	$J(\text{P}^1\text{-Rh}) = 154.0$; $J(\text{P}^1\text{-P}^2) = 37.6$ $\text{P}^2 44.7$ (dd)	
		$\text{H}^1 + \text{H}^2 - 0.72$ (d); $J(\text{Rh-H}) = 20.0$ $\text{H}^3 - 4.02$ (t); $J(\text{H}^3\text{-P}^3) = J(\text{H}^3\text{-P}^4) = 44.7$ $\text{H}^4 - 10.54$ (dd); $J(\text{H}^4\text{-Rh}) = 26.0$; $J(\text{P-H}) = 62.0$	$J(\text{P}^2\text{-Rh}) = 190.8$; $J(\text{P}^2\text{-P}^1) = 37.6$ $\text{P}^3 + \text{P}^4 42.7$ (d); $J(\text{Rh-P}) = 7.1$	193
2	1850	$\text{H}^1 + \text{H}^2 + \text{H}^4 - 3.10$ (b); $J(\text{Rh-H}) = 27.0$	$\text{P}^1 52.5$ (dd)	300
		$\text{H}^3 - 4.53$ (t); $J(\text{P-H}) = 49.1$	$J(\text{P}^1\text{-Rh}) = 146.0$; $J(\text{P}^1\text{-P}^2) = 39.5$ $\text{P}^2 46.3$ (dd)	
		$\text{H}^1 + \text{H}^2 0.11$ (dd); $J(\text{Rh-H}) = 25.7$; $J(\text{P-H}) = 25.0$ $\text{H}^3 - 4.75$ (t); $J(\text{H}^3\text{-P}^3) = J(\text{H}^3\text{-P}^4) = 48.5$ $\text{H}^4 - 9.78$ (dd); $J(\text{H}^4\text{-Rh}) = 27.5$; $J(\text{P-H}) = 64.7$	$J(\text{P}^2\text{-Rh}) = 193.0$; $J(\text{P}^2\text{-P}^1) = 39.5$ $\text{P}^3 + \text{P}^4 40.6$ (d); $J(\text{Rh-P}) = 6.6$	183
3	1850	$\text{H}^1 + \text{H}^2 - 2.78$ (d); $J(\text{Rh-H}) = 20.0$	$\text{P}^1 145.6$ (dd)	193
		$\text{H}^3 - 4.14$ (t); $J(\text{H}^3\text{-P}^3) = J(\text{H}^3\text{-P}^4) = 45.1$	$J(\text{P}^1\text{-Rh}) = 226.7$; $J(\text{P}^1\text{-P}^2) = 70.0$ $\text{P}^2 142.8$ (dd)	
		$\text{H}^4 - 10.20$ (dd); $J(\text{H}^4\text{-Rh}) = 20.0$; $J(\text{P-H}) = 80.6$	$J(\text{P}^2\text{-Rh}) = 275.0$; $J(\text{P}^2\text{-P}^1) = 70.0$ $\text{P}^3 + \text{P}^4 43.3$ (d); $J(\text{Rh-P}) = 6.0$	

^a in CH_2Cl_2 ; ^b in CD_2Cl_2 ; ^c hydride region.

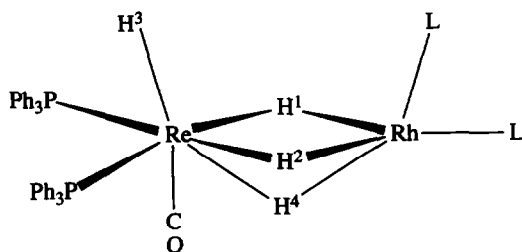


Fig. 1. Proposed structure for the complexes **1** ($L = PPh_3$), **2** ($L = TPP$) and **3** ($L = P(OMe)_3$).

is a distorted trigonal bipyramid, and around rhenium a capped octahedron. This arrangement is consistent with the magnetic equivalence of the two phosphorus atoms bound to rhenium and of two of the bridging hydrides. The third bridging hydride is *trans* to one phosphorus bound to rhodium, consistent with the observation of a P–H coupling constant.

The fluxionality of this molecule can be accounted for by a rotation of the three bridging hydride ligands relative to the fixed $ReH(CO)(PPh_3)_2$ and RhL_2 moieties.

2.2. Reaction of the heterobimetallic polyhydrides **1** and **2** with tetrafluoroboric acid

We have already reported that protonation of $[(CO)(PPh_3)_2HRe(\mu-H)_3RuH(PPh_3)_2]$ forms a cationic complex containing molecular hydrogen $[(CO)(PPh_3)_2HRe(\mu-H)_3Ru(H_2)(PPh_3)_2]^+$ [4]. Although we were aware that rhodium does not markedly stabilize the molecular hydrogen ligand [8], we have also carried out the reaction of **1** and **2** with tetrafluoroboric acid.

Two new complex salts **4** ($L = PPh_3$) and **5** ($L = TPP$) (the latter after BPh_4^- for BF_4^- exchange) have been obtained from this reaction, which can be reversed by adding a slight excess of triethylamine. Spectroscopic data for these complexes are presented in Table 2.

TABLE 2. Spectroscopic data for the complexes $[(CO)(PPh_3)_2HRe(\mu-H)_3RhHL_2][BF_4]$ (**4**, $L = PPh_3$; **5**, $L = TPP$)

Complex	IR $\nu(CO)$ (cm^{-1})	1H δ (ppm); J (Hz)	^{31}P NMR δ (ppm); J (Hz)	T (K)
4	1888	$H^1 - 0.57(b)$	P^1 48.8(dddd) $J(P^1-P^2) = 20.3$; $J(P^1-P^3) = 16.7$; $J(P^1-P^4) = 4.8$; $J(P^1-Rh) = 106.6$	294
		$H^2 - 3.93(t)$; $J(P-H) = 49.0$		
5	1882	$H^3 - 4.40(b)$	P^2 42.9(ddt) $J(P^2-P^1) = 20.3$; $J(P^2-P^3) \approx J(P^2-P^4) < 5$; $J(P^2-Rh) = 105.5$ P^3 39.4(m) $J(P^3-P^1) = 18.0$; $J(P^3-P^2) \approx J(P^3-P^4) \approx 4.8$; $J(P^3-Rh) = 22.0$ P^4 38.6(m) $J(P^4-P^1) \approx J(P^4-P^2) \approx J(P^4-P^3) < 5$; $J(P^4-Rh) = 18.7$	193
		$H^4 - 6.60(b)$		
		$H^5 - 16.10(dt)$; $J(Rh-H) = 17.5$; $J(P-H) = 22.3$		
		$H^1 - 0.66(b)$; $J(H^1-Rh) = 24.1$		
		$H^2 - 4.09(t)$; $J(H^2-P^3) = J(H^2-P^4) = 47.0$		
		$H^3 - 4.30(dd)$; $J(H^3-Rh) = 29.0$; $J(H^3-P) = 76.0$		
		$H^4 - 6.45(dd)$; $J(H^4-Rh) = 30.5$; $J(H^4-P) = 103.0$		
		$H^5 - 15.76(b)$; $J(H^5-Rh) = 14.4$		
		H^1 0.65(b)		
		$H^2 - 3.98(t)$; $J(P-H) = 51.5$		
$H^3 - 4.20(b)$				
$H^4 - 6.80(bd)$; $J(P-H) = 111.0$				
$H^5 - 17.73(dt)$; $J(Rh-H) = 14.0$; $J(P-H) = 23.4$				
H^1 0.55(b); $J(H^1-Rh) = 23.4$; $J(H^1-P^3) = 20.0$				
$H^2 - 4.08(t)$; $J(H^2-P^3) = J(H^2-P^4) = 50.6$				
$H^3 - 4.16(dd)$; $J(H^3-Rh) = 38.7$; $J(H^3-P) = 63.0$				
$H^4 - 6.83(dd)$; $J(H^4-Rh) = 31.7$; $J = 98.0$; $J(H^4-P^1)$ or $J(H^4-P^2)$				
$H^5 - 17.72(dt)$; $J(H^5-Rh) = 15.0$; $J(H^5-P^1) = J(H^5-P^2) = 23.0$				

^a in CH_2Cl_2 ; ^b in CD_2Cl_2 ; ^c hydride region.

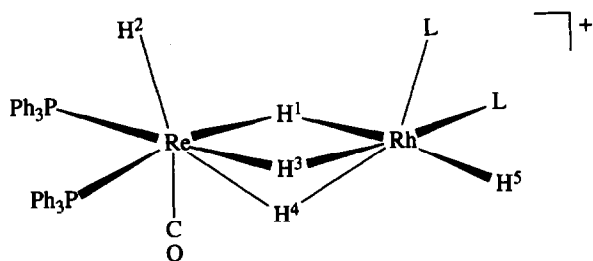


Fig. 2. Proposed structure for the complexes 4 ($L = PPh_3$) and 5 ($L = TPP$).

Protonation induces a shift of the $\nu(\text{CO})$ absorption towards higher frequencies consistent with cationic structure.

The NMR data for these complexes are very similar, suggesting similar structures. At room temperature we observe five signals of the same intensity in the hydride region of the ^1H NMR spectra: one triplet characteristic of a terminal hydride (H^2) on rhenium, a doublet of triplets characteristic of a terminal hydride (H^5) on rhodium and three broad resonances for H^1 , H^3 , and H^4 .

On lowering the temperature to 193 K, these three broad hydride resonances change to a doublet (H^1) due to coupling with rhodium, and two doublets of doublets (H^3 and H^4) due to coupling with rhodium and with one phosphorus bound to rhodium. The values of $J(\text{P}-\text{H})$ are consistent with a *trans* position of the two ligands [1c], the hydrides bridging the two metals. The two resonances of the terminal hydrides (H^2 and H^5) are slightly affected by lowering the temperature, a broadening of the resonance of the terminal hydride on rhodium being observed for complex 4.

At 193 K, we also observe four multiplets in the $^{31}\text{P}\{^1\text{H}\}$ NMR spectra, the phosphorus atoms bound to rhenium, P^3 and P^4 , showing a small coupling with rhodium.

From these observations we conclude that protonation of 1 and 2 has induced the formation of a Rh-H terminal bond and not the expected molecular hydrogen ligand. Moreover, in these cationic complexes, the exchange of the bridging hydride is noticeably slowed down as three separate broad signals are observed at room temperature.

Accordingly we propose the structure shown in Fig. 2 for these two complexes, the geometry around rhodium being a distorted octahedron.

3. Conclusion

The reaction of $\text{K}[\text{ReH}_6(\text{PPh}_3)_2]$ towards some halidocarbonyl complexes $[\text{RhCl}(\text{CO})\text{L}_2]$ has led to a new family of electronically unsaturated heterobimetallic

polyhydride complexes $[(\text{CO})(\text{PPh}_3)_2\text{HRe}(\mu\text{-H})_3\text{RhL}_2]$ isoelectronic with the recently synthesized $[(\text{CO})(\text{PPh}_3)_2\text{HRe}(\mu\text{-H})_3\text{RuH}(\text{PPh}_3)_2]$. The action of tetrafluoroboric acid on these complexes leads to the formation of $[(\text{CO})(\text{PPh}_3)_2\text{HRe}(\mu\text{-H})_3\text{RhHL}_2][\text{BF}_4]$ which do not contain a molecular hydrogen ligand, which is not very surprising considering the small number of known dihydrogen rhodium complexes.

4. Experimental section

All reactions were performed under dinitrogen using standard Schlenk techniques. IR spectra were recorded on a Perkin-Elmer 225 spectrometer. NMR spectra were recorded on Bruker AC 80 (^{31}P), AC 200 (^1H) and WM 250 (^1H , ^{31}P) instruments. Variable temperature and selective decoupling experiments were carried out on a WM 250 machine. Elemental analyses (C and H) were performed in our laboratory and by the Service Central de Microanalyse du CNRS for the other elements. TPP [9], $[\text{ReH}_7(\text{PPh}_3)_2]$ [10], $[\text{RhCl}(\text{CO})\text{L}_2]$ [$L = \text{PPh}_3$ or $\text{P}(\text{OMe})_3$] [11], and $[\text{RhCl}(\text{CO})(\text{TPP})_2]$ [12] were prepared according to published procedures.

4.1. Synthesis of $[(\text{CO})(\text{PPh}_3)_2\text{HRe}(\mu\text{-H})_3\text{RhL}_2]$ ($L = \text{PPh}_3$, TPP, or $\text{P}(\text{OMe})_3$)

$[\text{ReH}_7(\text{PPh}_3)_2]$ (0.3 g, 0.42 mmol) was dissolved in THF (30 ml) and stirred with an excess of KH. After 1 h the solution was filtered to remove the unreacted KH. To the filtrate was added a stoichiometric amount of $[\text{RhCl}(\text{CO})\text{L}_2]$ and the mixture was stirred for 1 h. The solution turned brown and was evaporated to dryness under vacuum. The residue was dissolved in toluene and the solution was filtered through Celite to remove KCl. The complexes were isolated by crystallization from toluene/hexane.

$[(\text{CO})(\text{PPh}_3)_2\text{HRe}(\mu\text{-H})_3\text{Rh}(\text{PPh}_3)_2]$ (1); brown powder (63% yield). Anal. Calcd. for $\text{C}_{73}\text{H}_{64}\text{OP}_4\text{ReRh}$: C, 63.99; H, 4.71; P, 9.04; Re, 13.59; Rh, 7.51. Found: C, 62.85; H, 4.89; P, 8.76; Re, 12.17; Rh, 7.54%.

$[(\text{CO})(\text{PPh}_3)_2\text{HRe}(\mu\text{-H})_3\text{Rh}(\text{TPP})_2]$ (2); green powder (62% yield). Anal. Calcd. for $\text{C}_{81}\text{H}_{68}\text{OP}_4\text{ReRh}$: C, 66.16; H, 4.66; P, 8.43; Re, 12.66; Rh, 6.99. Found: C, 65.77; H, 4.97; P, 8.32; Re, 11.79; Rh, 7.05%.

$[(\text{CO})(\text{PPh}_3)_2\text{HRe}(\mu\text{-H})_3\text{Rh}(\text{P}(\text{OMe})_3)_2]$ (3), brown powder (36% yield). This compound was not stable enough to obtain a reliable analysis.

4.2. Synthesis of $[(\text{CO})(\text{PPh}_3)_2\text{HRe}(\mu\text{-H})_3\text{RhHL}_2][\text{BF}_4]$ ($L = \text{PPh}_3$ or TPP)

To a solution of 1 or 2 (0.1 g) in dichloromethane (10 ml) was added one equivalent of $\text{HBF}_4 \cdot \text{OEt}_2$. The solution immediately turned red, and after 10 min at room temperature it was evaporated to dryness under

vacuum. Recrystallization from dichloromethane/diethyl ether gave complexes **4** and **5**.

$[(\text{CO})(\text{PPh}_3)_2\text{HRe}(\mu\text{-H})_3\text{RhH}(\text{PPh}_3)_2][\text{BF}_4]$ (**4**); orange powder (90% yield). Anal. Calcd. for $\text{C}_{73}\text{H}_{65}\text{BF}_4\text{OP}_4\text{ReRh}$: C, 60.13; H, 4.49; F, 5.21; P, 8.50; Re, 12.77; Rh, 7.06. Found: C, 60.02; H, 4.63; F, 5.14; P, 8.21; Re, 11.97; Rh, 7.33%.

$[(\text{CO})(\text{PPh}_3)_2\text{HRe}(\mu\text{-H})_3\text{RhH}(\text{TPP})_2][\text{BPh}_4]$ (**5**); orange powder (90% yield) after exchange of the anion with NaBPh_4 in acetone solution. Anal. Calcd. for $\text{C}_{105}\text{H}_{89}\text{BOP}_4\text{ReRh}$: C, 70.43; H, 5.01; P, 6.92; Re, 10.40; Rh, 5.75. Found: C, 68.83; H, 5.18; P, 6.78; Re, 9.73; Rh, 5.28%.

Acknowledgment

The authors wish to thank the CNRS for financial support. Thanks are also due to Dr. J. Lacey for his help in editing the English of this paper.

References

- 1 See for instance (a) L.M. Venanzi, *Coord. Chem. Rev.*, **43** (1982) 251; (b) D. Alvarez Jr., E.G. Lundquist, J.W. Ziller, W.J. Evans

- and K.G. Caulton, *J. Am. Chem. Soc.*, **111** (1989) 8392. (c) J.T. Poulton, K. Foltling and K.G. Caulton, *Organometallics*, **11** (1992) 1364; (d) C.P. Casey and G.T. Whiteker, *Inorg. Chem.*, **29** (1990) 876; (e) K.E. Meyer, P.E. Fanwick and R.A. Walton, *Inorg. Chem.*, **31** (1992) 4486 and references therein.
- 2 G.L. Geoffroy and R. Pierantozzi, *J. Am. Chem. Soc.*, **98** (1976) 8054.
- 3 G.G. Hlatky and R.H. Crabtree, *Coord. Chem. Rev.*, **65** (1985) 1.
- 4 M. Cazanoue, Z. He, D. Neibecker and R. Mathieu, *J. Chem. Soc., Chem. Commun.*, (1991) 307.
- 5 Z. He, D. Neibecker, N. Lukan and R. Mathieu, *Organometallics*, **11** (1992) 817.
- 6 S.J. Chen and K.R. Dunbar, *Inorg. Chem.*, **29** (1990) 529.
- 7 Z. He, S. Nefedov, D. Neibecker, N. Lukan and R. Mathieu, *Organometallics*, in press.
- 8 P.G. Jessop and R.H. Morris, *Coord. Chem. Rev.*, **121** (1992) 155.
- 9 B. Lukas, R.M.G. Roberts, J. Silver and A.S. Wells, *J. Organomet. Chem.*, **256** (1983) 103.
- 10 D. Baudry, M. Ephritikhine and H. Felkin, *J. Organomet. Chem.*, **224** (1982) 363.
- 11 D. Evans, J.A. Osborn and G. Wilkinson, *Inorg. Synth.*, **11** (1968) 99.
- 12 D. Neibecker and R. Réau, *J. Mol. Catal.*, **57** (1989) 153.