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Polyhedron 23 (2004) 1567–1572

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Synthesis and characterization of cyclopentadienylruthenium(II) complexes containing *N*,*N*′-donor Schiff base ligands: crystal and molecular structure of $[(\eta^5\text{-}C_5H_5)Ru(C_5H_4N\text{-}2\text{-}CH{=}N\text{–}C_6H_4\text{–}p\text{-}OCH_3)(PPh_3)]PF_6$ ☆

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Received 27 February 2004; accepted 15 March 2004
Available online 20 April 2004

Abstract

Complexes of the formulae $[(\eta^5\text{-}C_5H_5)Ru(PPh_3)(C_5H_5N\text{-}2\text{-}CH{=}N\text{–}C_6H_4\text{–}p\text{-}X)]^+$ [X = H (**2a**), CH_3 (**2b**), OCH_3 (**2c**), Cl (**2d**), NO_2 (**2e**)] and $[(\eta^5\text{-}C_5H_5)Ru(PPh_3)(C_5H_5N\text{-}2\text{-}CH{=}N\text{–}C_6H_{11})]^+$ (**3**) were prepared by reacting *para*-substituted *N*-(pyrid-2-ylmethylene)-phenylamines (**2-PP**) and *N*-(pyrid-2-ylmethylene)cyclohexylamine (**2-PC**) with $[(\eta^5\text{-}C_5H_5)Ru(PPh_3)_2Cl]$ (**1**) in methanol. These complexes have been isolated as hexa-fluorophosphate salts. Representative complex **2c** has been established by single crystal X-ray diffraction analysis. Complex **2c** crystallizes in the orthorhombic space group P_{bcn}, with $a = 21.1560\ (11)$ Å, $b = 18.3972\ (9)$ Å and $c = 17.5130\ (9)$ Å, $V = 6816.3\ (6)$ Å^3 and $z = 8$. All these complexes were characterized by FT-IR, ^{1}H NMR and $^{31}P\{^1H\}$ NMR spectroscopy.

Keywords: Cyclopentadienyl; Schiff bases; Pyridine-2-carboxaldehyde; Ruthenium

1. Introduction

As a part of our continuing investigation of the complexes of η^5-cyclopentadienyl-ruthenium(II) [1], η^5-indenylruthenium(II) [2], η^5-cyclopentadienylosmium(II) [3] and η^6-areneruthenium(II) [4] with a variety of nitrogen-based ligands, we here report the syntheses of complexes of the cyclopentadienylruthenium(II) system with some chelating Schiff base ligands. Considerable interest has arisen in the chemistry of ruthenium (II) complexes having Schiff bases [5] or chelating nitrogen heterocyclic ligands [6] because of their photochemical and catalytic activities [7]. Steric factors play an important role compared to electronic factors in the structure and chemistry of these complexes. We had earlier reported the reaction of η^5-indenylruthenium(II) [8] and η^6-arene-ruthenium(II) [9] with Schiff base ligands. During the reactions involving the analogous pentamethylcyclopentadienyl or indenyl systems, the products did not show evidence of binding of these organic ligands, simple coordination compounds being formed instead. Although these groups can provide more electron density to the metal for binding, their large bulk compared to the cyclopentadienyl ligand prevents effective binding. This observation indicates steric factors playing a major role compared to electronic factors. However, relatively fewer reports are available in the literature for the analogous cyclopentadienylruthenium(II) Schiff base complexes [10]. We report here the synthesis and characterization of new cationic cyclopentadienylruthenium(II) complexes with *N*,*N*′-donor Schiff base ligands, viz., various

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☆ Supplementary data associated with this article can be found, in the online version, at doi: 10.1016/j.poly.2004.03.007.

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doi:10.1016/j.poly.2004.03.007

para-substituted *N*-(pyrid-2-ylmethylene) phenylamines (**2-PP**) and *N*-(pyrid-2-ylmethylene)cyclohexylamine (**2-PC**). The molecular structure of the complex [(η^5-C_5H_5)Ru(C_5H_4N-2-CH=N–C_6H_4–*p*-OCH_3)(PPh_3)]PF_6 as solved by X-ray crystallography is reported here as well.

2. Experimental

2.1. General remarks

All reactions were carried out in distilled and dried solvents under a dry nitrogen atmosphere. $RuCl_3 \cdot 3H_2O$ was purchased from Arora Matthey Ltd. and used as such. Pyridine-2-carboxaldehyde (Fluka) was purchased and used as received. All liquid amines were reagent grade and distilled prior to use, while solid amines were used as such. Elemental analyses were performed in a Perkin–Elmer-2400 CHN/O analyzer. Infrared spectra were recorded on a Perkin–Elmer-model 983 spectrophotometer with the sample prepared as KBr pellets. Electronic spectra were recorded on a Hitachi-300 spectrophotometer. Conductivity measurements were performed in Wayne Kerr automatic precision bridge B-905. The ^1H NMR spectra were recorded in $CDCl_3$ solvent (with tetramethylsilane as the internal standard) on a Bruker ACF-400 MHz spectrometer, coupling constants *J* being given in hertz (hz). $^{31}P\{^1H\}$ NMR chemical shifts were recorded relative to H_3PO_4 (85%). The ligands C_5H_4N-2-CH=C_6H_4–*p*-X (where X = H, CH_3, OCH_3, Cl, NO_2) [11], and the precursor complex [(η^5-C_5H_5)Ru(PPh_3)$_2$Cl] (**1**) [12] were prepared following literature methods.

2.2. Preparation of [(η^5-C_5H_5)Ru(PPh_3)(C_5H_5N-2-CH=N–C_6H_4–p-X)]PF_6 (2a–e), (X = H (2a), CH_3 (2b), OCH_3 (2c), Cl (2d), NO_2 (2e)

The following general procedure was used for preparation of these five complexes:

The mixture of the starting complex [(η^5-C_5H_5)Ru(PPh_3)$_2$Cl] (0.1 g, 0.110 mmol), the appropriate Schiff base ligand **2-PP** (0.165 mmol) and NH_4PF_6 (0.165 mmol) was refluxed in methanol (40 ml) whereby the orange color suspension gradually changed to a dark brown solution. It was refluxed for 6 h, and the solvent then rotary evaporated. The residue was dissolved in dichloromethane and filtered through a short silica gel column to remove insoluble material. The filtrate was again concentrated to about 2 ml, whereupon addition of excess hexane gave the desired complexes (**2a**–**e**) as dark brown solids. The solid was washed with diethyl ether and dried under vacuum to afford an 80–85% yield of the complexes.

2.3. Preparation of [(η^5-C_5H_5)Ru(PPh_3)(**2-PC**)] PF_6 (3)

This complex was prepared in a similar manner to that of the preparation of (**2a**–**e**), except that the ligand **2-PC** was used instead of **2-PP** to yield the dark brown solid complex in 80% yield.

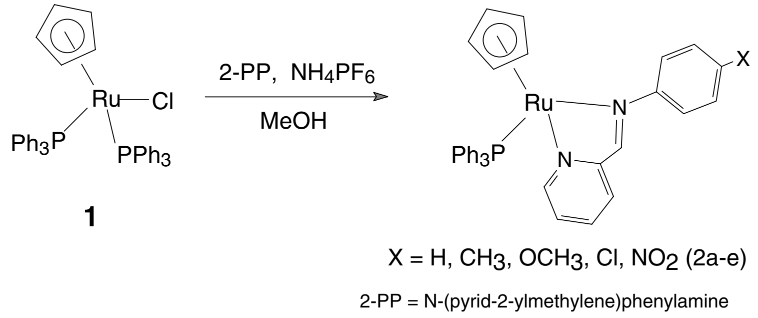
3. Structure analysis and refinement

X-ray quality crystals of the complex **2c** were grown by slow diffusion of hexane into a dichloromethane solution. A brown crystal of complex **2c** was mounted on a Bruker Apex CCD diffractometer in a full reciprocal sphere equipped with CCD detector and used for data collection. X-ray intensity data were collected with graphite mono-chromated Mo Kα radiation at 293(2) K, with 0.3° scans in ω scan mode and 10-s exposure time per frame. The intensity data were corrected for Lorentz and polarization effects through the SAINT program [13]. A summary of the crystal data, data collection parameters and convergence results are compiled in Table 3. An empirical absorption correction was made by modeling a transmission surface by spherical harmonics employing equivalent reflections with $I > 3\sigma(I)$ (program SADABS) [14]. The structure was solved by direct methods [15]. All the non-hydrogen atoms were refined anisotropically using the full-matrix least-squares technique on F^2 using the SHELXL 97 software [16]. All the hydrogen atom positions were found from difference Fourier synthesis after four cycles of anisotropic refinement and as "riding" model. Fig. 1 is the ORTEP [17] representation of the molecules with 50% probability thermal ellipsoids displayed. Refinement coverage at a final value of $R_1 = 0.0462$ (for observed data *F*) and at value of $wR_2 = 0.1192$, respectively (for unique data F^2).

4. Results and discussion

The synthetic reaction of the cyclopentadienyl complex **1** with excess of *N,N'*-donor Schiff base ligands, viz., **2-PP** and **2-PC**, in methanol under refluxing condition resulted in the formation of brown colored and air-stable cationic complexes of the type **2** (Scheme 1) by dissociation of one of the triphenylphosphines and chloride ligand. Here, we would like to point out that when similar reactions were carried out with complexes bearing the substituted analogue Cp* and the indenyl group instead, the resulting complexes had no organic groups bound to the metal.

These complexes are highly soluble in polar solvents such as chloroform, dichloromethane, etc., but insoluble in non-polar solvents such as hexane, pentane, etc.



Scheme 1.

These complexes were characterized by using analytical, IR, ^{1}H and ^{31}P NMR spectroscopic techniques. The data, as presented in Table 1, support the formation of these complexes. The X-ray structure of the representative complex **2c** was determined to confirm the structure of the complex. The IR spectra of these complexes show a strong band in the range of 1602–1584 cm^{-1} due to the $\nu_{\mathrm{C=N}}$ group of the Schiff base ligand. In addition, the IR spectra contain strong bands due to the phenyl groups of triphenylphosphine and a strong band at 840 cm^{-1} due to the $\nu_{\mathrm{P-F}}$ of the PF_6 group. Complex **2e** shows the characteristic bands for ν_{NO2} (asymmetric stretch at 1523 cm^{-1} and symmetric stretch 1346 cm^{-1}).

In the ^{1}H NMR spectra of these complexes, the cyclopentadienyl ring protons display a sharp singlet at 4.7 ppm. The presence of η-Cp, PPh_3 and **2-PP** or **2-PC** in a 1:1:1 ratio is inferred from the areas obtained upon peak integration of the ^{1}H NMR spectra. The resonance of the *ortho* proton of the pyridine ring of the ligand is observed as a doublet in the range 9.28–8.45 ppm in these complexes. The multiplet observed in the range 8.13–6.76 ppm is due to the phenyl protons of the triphenylphosphine moiety, the amine group and the *N*-heterocyclic ring of the ligands (**2-PP** or **2-PC**). The ^{31}P NMR spectra of the complexes **2** and **3** exhibit a single sharp resonance for triphenylphosphine around 47 ppm, whereas the starting complex [(η^5-Cp)Ru(PPh_3)$_2$Cl] (**1**) exhibits it at 42 ppm for both triphenylphosphines indicating the cationic nature of these complexes. The ionic nature of these complexes was confirmed by carrying out conductivity measurements (Table 2).

The electronic spectra and conductivity data of these complexes are shown in Table 2. The electronic spectra of these complexes in dichloromethane exhibit bands in the range of 428–461 nm. This low-energy absorption is assigned to a Ru dπ to ligand π* metal-to-ligand charge transfer (MLCT) transition. The molar conductivity of the complexes in acetonitrile solvent ranges from 123.0 to 150.0 S cm^2 mol^{-1}, suggesting that these complexes are ionically dissociated in the ratio of 1:1 [18].

Table 1
Analytical[a], FT IR, ^{1}H NMR[b] and ^{31}P NMR of the complexes **2a**–**e** and **3**

Complex	Analyses %			IR (KBr Pellets, cm^{-1})	^{1}H NMR δ [multiplicity, nH, J (Hz)]	^{31}P NMR (δ)
	C	H	N			
2a	55.6 (56.0)	4.0 (4.3)	3.7 (3.4)	1587($\nu_{\mathrm{C=N}}$) 839($\nu_{\mathrm{P-F}}$)	9.28 (d, 1H, 5.48); 8.26 (d, 1H); 7.69–6.96 (m, 23H); 4.66 (s, 5H, Cp).	46.91
2b	56.1 (56.6)	4.1 (4.3)	3.6 (2.9)	1602($\nu_{\mathrm{C=N}}$) 840($\nu_{\mathrm{P-F}}$)	9.26 (d, 1H, 2.96); 8.23 (d, 1H); 7.65–6.91 (m, 22H); 4.67 (s, 5H, Cp); 2.36 (s, 3H).	47.09
2c	55.0 (55.5)	4.1 (4.5)	3.6 (3.2)	1598($\nu_{\mathrm{C=N}}$) 837($\nu_{\mathrm{P-F}}$)	9.20 (d, 1H, 5.56); 8.23 (d, 1H); 7.69–6.76 (m, 22H); 4.68 (s, 5H, Cp); 3.84 (s, 3H)	47.25
2d	53.2 (53.8)	3.7 (3.2)	3.5 (2.9)	1586($\nu_{\mathrm{C=N}}$) 837($\nu_{\mathrm{P-F}}$)	9.21 (d, 1H, 5.44); 8.31 (d, 1H); 7.73–6.96 (m, 22H); 4.68 (s, 5H, Cp)	46.24
2e	52.5 (52.4)	3.6 (3.9)	5.3 (5.7)	1589($\nu_{\mathrm{C=N}}$) 840($\nu_{\mathrm{P-F}}$)	8.45 (d, 1H, 2.96); 8.11 (d, 1H); 7.83–6.93 (m, 22H); 4.69 (s, 5H, Cp)	46.70
3	55.3 (55.1)	4.6 (4.2)	3.7 (3.9)	1589($\nu_{\mathrm{C=N}}$) 839($\nu_{\mathrm{P-F}}$)	9.06 (d, 1H, 7.04); 8.23 (d, 1H); 8.14–7.04 (m, 18H); 4.65 (s, 5H, Cp); 1.12–2.10 (m, 10H).	47.35

[a] Calculated values are in parentheses.

[b] In $CDCl_3$; s singlet; d, doublet; m, multiplet; $J_{(\mathrm{H-H})}$ in Hz.

Table 2
UV–Vis and conductivity data of the complexes at room temperature

S. no.	Complexes	λ_{max} (nm)	Conductivity, Λ_m (S cm^2 mol^{-1})
2a	$[(\eta^5\text{-}C_5H_5)Ru(PPh_3)(C_5H_4N\text{-}2\text{-}CH{=}N\text{–}C_6H_4\text{–}p\text{-}H)]PF_6$	454	150.00
2b	$[(\eta^5\text{-}C_5H_5)Ru(PPh_3)(C_5H_4N\text{-}2\text{-}CH{=}N\text{–}C_6H_4\text{–}p\text{-}CH_3)]PF_6$	452	126.02
2c	$[(\eta^5\text{-}C_5H_5)Ru(PPh_3)(C_5H_4N\text{-}2\text{-}CH{=}N\text{–}C_6H_4\text{–}p\text{-}CH_3)]PF_6$	454	143.90
2d	$[(\eta^5\text{-}C_5H_5)Ru(PPh_3)(C_5H_4N\text{-}2\text{-}CH{=}N\text{–}C_6H_4\text{–}p\text{-}Cl)]PF_6$	458	137.91
2e	$[(\eta^5\text{-}C_5H_5)Ru(PPh_3)(C_5H_4N\text{-}2\text{-}CH{=}N\text{–}C_6H_4\text{–}p\text{-}NO_2)]PF_6$	461	136.79
3	$[(\eta^5\text{-}C_5H_5)Ru(PPh_3)(C_5H_4N\text{-}2\text{-}CH{=}N\text{–}C_6H_{11})]PF_6$	428	123.80

Table 3
Summary of structure determination of complex $[(\eta^5\text{-}C_5H_5)Ru(C_5H_4N\text{-}2\text{-}CH{=}N\text{–}C_6H_4\text{–}p\text{-}OCH_3)]PF_6$ (**2c**)

Formula	$C_{36}H_{32}F_6N_2OP2Ru$
Formula weight	785.65
Crystal system	orthorhombic
Space group	*Pbcn*
Unit cell dimensions	
a (Å)	21.1560(11)
b (Å)	18.3972(9)
c (Å)	17.5130(3)
V (Å^3)	6816.3(6)
Z	8
Crystal size (mm)	$0.3 \times 0.3 \times 0.6$
D_{calc} (g cm^{-3})	1.531
F(0 0 0)	3184
θ range for data collection (°)	1.87–28.30
Index ranges	$-28 \leqslant h27$, $-23 \leqslant k \leqslant 24$, $-23 \leqslant l \leqslant 23$
Reflections collected	57 254
Independent reflections	8285 [$R_{int} = 0.0354$]
Absorption coefficient (mm^{-1})	0.619
Absorption correction	empirical (SADABS)
Refinement method	full-matrix least-squares on F^2
Data/restraints/parameters	8285/0/356
Goodness-of-fit on F^2	1.006
Final *R* indices [$I > 2\sigma(I)$]	$R_1 = 0.0462$, $wR_2 = 0.1192$
R indices (all data)	$R_1 = 0.0594$, $wR_2 = 0.1286$
Largest difference peak and hole (e Å^{-3})	0.896 and −0.530

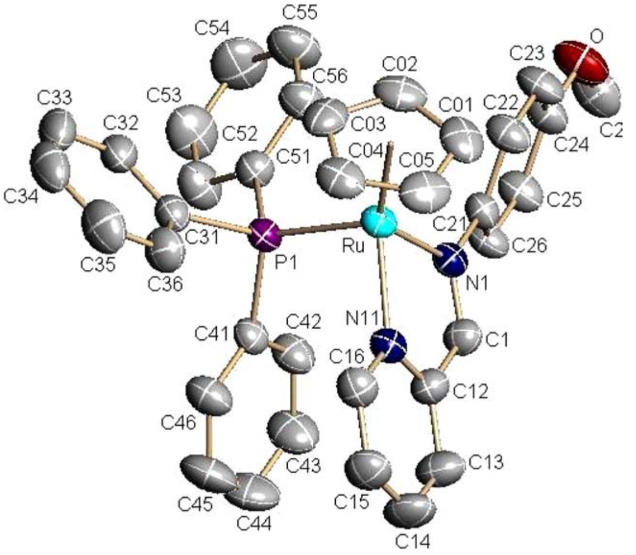


Fig. 1. ORTEP drawing of compound **2c** with 50% probability thermal ellipsoids. Hydrogen atoms and PF_6 omitted for clarity.

5. Crystal structure of 2c

The structure of complex **2c** is shown in Fig. 1. Representative bond length and bond angle values are listed in Table 4. The ruthenium atom is coordinated to two nitrogen atoms of the Schiff base ligand, one triphenylphosphine ligand and a pentahapto-bound cyclopentadienyl ligand. The geometry around the metal atom can be regarded as distorted octahedral if the η^5-cyclopentadienyl group is assumed to occupy three facial coordinated positions.

The $[(\eta^5\text{-}C_5H_5)Ru(PPh_3)(C_5H_5N\text{-}2\text{-}CH{=}N\text{–}C_6H_4\text{–}p\text{-}OCH_3)]PF_6$ complex crystallizes in the orthorhombic space group P_{bcn}. The cyclopentadienyl group is clearly bonded in a pentahapto fashion to the metal. Three of the Ru–C bond lengths, viz., those involving the C(03), C(04) and C(05), are shorter (2.191, 2.182 and 2.196 Å) than the other two bonds involving the C(01) and C(02) carbon atoms (2.214 and 2.211 Å). The average Ru–C distance is 2.199 Å, whereas the distance between the ruthenium atom and the centroid of the ring is 1.837 Å at the axis $x = 0.0947$, $y = 0.0534$ and $z = 0.4796$. The Ru–P bond length is 2.3100 Å, which is within the usual range of Ru–P bond distances (2.20–2.43 Å) [19]. The ruthenium atom is directly coordinated to two nitrogen atoms of the Schiff base ligand with an average distance of 2.090 Å. The bite angle of the chelating ligand is 76.42(8°), not very different from that in other related complexes [20]. The geometry of the complex is octahedral (of the piano-stool type) with the cyclopentadienyl moiety occupying three coordination sites. This is evident in the nearly 90° values for the bond angle N(1)–Ru–P(1) between the non-cyclopentadienyl ligands (90.39(6)°) and for the bond angle N(11)–Ru–P(1) (92.67(6)°) at the metal centre. The orientation of the phenyl group of the amine is similar to the observed mutually perpendicular orientation found for similar types of ligand in arene ruthenium complexes [9].

Table 4
Selected bond lengths (Å) and bond angles (°) for [CpRu(PPh$_3$)(C$_5$H$_5$N-2-CH=N–C$_6$H$_4$–*p*-OCH$_3$)]PF$_6$ (**2c**)

Bond lengths					
Ru(1)–N(11)	2.0801(13)	Ru(1)–N(1)	2.110(2)	Ru(1)–P(1)	2.3100(8)
Ru(1)–C(01)	2.214(2)	Ru(1)–C(02)	2.211(2)	Ru(1)–C(03)	2.191(2)
Ru(1)–C(04)	2.182(2)	Ru(1)–C(05)	2.196(2)	P(1)–C(51)	1.8357(12)
P(1)–C(31)	1.8570(12)	P(1)–C(41)	1.8363(12)	N(1)–C(1)	1.296(4)
N(1)–C(21)	1.438(3)	N(11)–C(12)	1.3900	N(11)–C(16)	1.3900
Ru–Cp[a]	1.837				
Bond angles					
N(11)–Ru–N(1)	76.42(8)	N(11)–Ru–P(1)	92.67(6)		
N(1)–Ru–P(1)	90.39(6)				

[a] Ruthenium to centroid of Cp.

6. Conclusion

We have observed interesting phenomena in the case of the cyclopentadienyl complex **1**, which distinguish it from the analogous pentamethylcyclopentadienyl or indenyl ruthenium systems. Unlike the case of the complex **1**, the reactions of Schiff bases with the [(η^5-Cp*)Ru(PPh$_3$)$_2$Cl] and [(η^5-ind.)Ru(PPh$_3$)$_2$Cl] systems yield complexes without any organic group. We suggest that in order to obtain complexes bound to organic ligands for these cases, one has to start from acetonitrile complexes. The reactions of [(η^5-Cp)Ru(PPh$_3$)$_2$Cl] with Schiff base ligands, viz, *para*-substituted *N*-(pyrid-2-yl methylene)phenylamines and *N*-(pyrid-2-ylmethylene)-cyclohexylamine, in methanol yielded complexes **2a**–**e** of the type [[(η^5-Cp)Ru (PPh$_3$)(**2-PP**)]X and [(η^5-Cp)Ru (PPh$_3$)(**2-PC**)]X]. The molecular structure of the representative complex **2c** has been characterized by single crystal X-ray diffraction analysis.

7. Supplementary material

Crystallographic data for the structural analysis have been deposited at the Cambridge Crystallographic Data Centre (CCDC), CCDC No. 231959 for complex **2c**. 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; website: www: http://www.ccdc.cam.ac.uk).

Acknowledgements

We thank Professor R.H. Duncan Lyngdoh for his help in preparing the manuscript. K.M.R. and P.G. thank the Sophisticated Instruments Facility (SIF), Indian Institute of Science, Bangalore, for providing the NMR facility.

References

[1] (a) M.R. Kollipara, C.R.K. Rao, P.S. Zacharias, Polyhedron 16 (1997) 2369;
(b) R. Lalrempuia, P. Govindaswamy, Y.A. Mozharivskyj, M.R. Kollipara, Polyhedron 23 (2004) 1069;
(c) M.R. Kollipara, L. Mishra, U.C. Agarwala, Polyhedron 6 (1987) 1383.

[2] M.R. Kollipara, E.K. Rymmai, Polyhedron 22 (2003) 307.

[3] (a) R. Lalrempuia, P.J. Carroll, M.R. Kollipara, Polyhedron 22 (2003) 1391;
(b) R. Lalrempuia, Hemant Yennawar, Y.A. Mozharivskyj, M.R. Kollipara, J. Organomet. Chem. 689 (2004) 539.

[4] (a) R. Lalrempuia, M.R. Kollipara, Polyhedron 22 (2003) 3155;
(b) M.R. Kollipara, P. Sarkhel, S. Chakraborty, R. Lalrempuia, J. Coord. Chem. 56 (2003) 1085;
(c) Lalrempuia, P.J. Carroll, M.R. Kollipara, J. Coord. Chem. 56 (2003) 1499.

[5] S.A. Serron, C.M. Haar, S.P. Nolan, Organometallics 16 (1997) 5120.

[6] (a) C.W. Chang, G.H. Lee, Organometallics 22 (2003) 3107;
(b) M.R. Kollipara, E.K. Rymmai, Polyhedron 22 (2003) 307.

[7] (a) C.S. Hauser, C. Slugove, K. Mereiter, R. Schmid, K. Kirchner, L. Xiao, W. Weissensteiner, J. Chem. Soc., Dalton Trans. (2001) 2989;
(b) U. Karllson, G.Z. Wang, J.-E. Backvall, J. Org. Chem. 59 (1994) 1196;
(c) C. Ruppin, P.H. Dixneuf, S. Lewlier, Tetrahedron Lett. 20 (1998) 5365;
(d) B. Seiller, C. Bruneau, P.H. Dixneuf, J. Chem. Soc., Chem. Commun. 493 (1994);
(e) V. Rotleng, R. Bertani, M. Pfeffer, C. Sirlin, J. Hirschinges, Inorg. Chem. 40 (2001) 5117;
(f) C.A. Merlic, M.E. Pauly, J. Am. Chem. Soc. 18 (1996) 11319.

[8] S.S. Keisham, Y.A. Mozharivskyj, P.J. Carroll, M.R. Kollipara, J. Organomet. Chem. 689 (2004) 1249.

[9] R. Lalrempuia, M.R. Kollipara, P.J. Carroll, Polyhedron 22 (2003) 605.

[10] S.K. Mandal, A.R. Chakravarty, Polyhedron 10 (1991) 2483.

[11] (a) R.N. Dominey, B. Hauser, J. Habbard, J. Dunham, Inorg. Chem. 30 (1991) 4754;
(b) E.V. Dose, L.J. Wilson, Inorg. Chem. 17 (1978) 2660.

[12] M.I. Bruce, N.J. Windsor, Aust. J. Chem. 30 (1977) 1601.

[13] D. Becke, J. Chem. Phys. 98 (1993) 5648.

[14] XRD Single Crystal Software; Bruker Analytical X-ray systems, Madison, WI, 2002.

[15] G.M. Sheldrick, SHELXS 86, Program for Crystal Structure Solution, University of Göttingen, Göttingen, Germany, 1986.

[16] M. Sheldrick, SHELXS 97, Program for Crystal Structure Refinement, University of Göttingen, Göttingen, Germany, 1997.

[17] C.K. Johnson, ORTEP II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations, ORNL-5138, 1976.
[18] J. Geary, Coord. Chem. Rev. 7 (1971) 81.
[19] L.J. Guggenberger, Inorg. Chem. 12 (1973) 1317.
[20] S.K. Mandal, A.R. Chakravarty, Polyhedron 11 (1992) 823.