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Rhodium(III) pentamethyl cyclopentadienyl complexes incorporating 1-(4-cyanophenyl)-imidazole: role of solvent in ligand substitution reactions

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Abstract

Reaction of the dimeric rhodium complex $[\{(\eta^5\text{-}C_5Me_5)Rh(\mu\text{-}Cl)Cl\}_2]$ with an excess of 1-(4-cyanophenyl)-imidazole in dichloromethane afforded neutral mononuclear complex $[(\eta^5\text{-}C_5Me_5)RhCl_2(CPI)]$ (CPI = 1-(4-cyanophenyl)-imidazole) **1**. The complex **1** reacted with EPh_3 (E = P, As, Sb) and N–N donor bases 2,2′-bipyridine and 1,10-phenanthroline in different solvents to give substitution products wherein, nature of the product was governed by polarity of the solvents employed in the reaction. Resulting complexes have been characterized by elemental analyses, spectral (FAB-MS, IR, ^1H,^{13}C, ^{31}P NMR, UV–Vis, Emission) and electrochemical studies. Coordination of CPI through imidazole nitrogen and the presence of *pendant* nitrile group have been supported by spectral studies.

Keywords: Rhodium; Pentamethyl cyclopentadienyl; 1-(4-cyanophenyl)-imidazole; Piano-stool

1. Introduction

The dimeric chloro bridged complexes $[\{(\eta^5\text{-}C_5Me_5)M(\mu\text{-}Cl)Cl\}_2]$ (M = Rh or Ir) have been the subject of investigation by many research groups as these are very useful starting materials [1]. The complexes undergo rich variety of chemistry via the intermediacy of chloro bridge cleavage reactions leading to formation of a series of interesting neutral and cationic mononuclear complexes [2]. Despite extensive studies on the complex $[\{(\eta^5\text{-}C_5Me_5)M(\mu\text{-}Cl)Cl\}_2]$, reactivity of this complex with 1-(4-cyanophenyl)imidazole (CPI) has yet to be explored. CPI is an interesting molecule which behaves as bridging ligand as well as exhibit *twisted internal charge transfer* [3]. It is expected that the complexes in which, redox sites are bridged by this type of molecule could be a good model to test the possibility of molecular switching. Bridging ability coupled with the *twisted internal charge transfer (TICT)* in CPI, offers interesting perspectives for the study of intra molecular metal to metal charge transfer as well as photo-induced geometrical changes.

Further, it is established that the metal to ligand bond strength has remarkable importance in coordination/organometallic synthetic process [4]. Metal–ligand link can be targeted as strong (metal–phosphine) or weak (metal–ether or amine) bond. The strength of the metal–ligand link depends on the nature of transition metal i.e. its oxidation state and coordination number, but for a single metal targeted complex the polarity of the solvent and availability of the counter ion in the reaction medium becomes determining factor for resulting complexes [5]. For neutral approaching ligands, the polar solvent facilitates the reaction by polarizing the reactant, but for a non-polar solvent such type of

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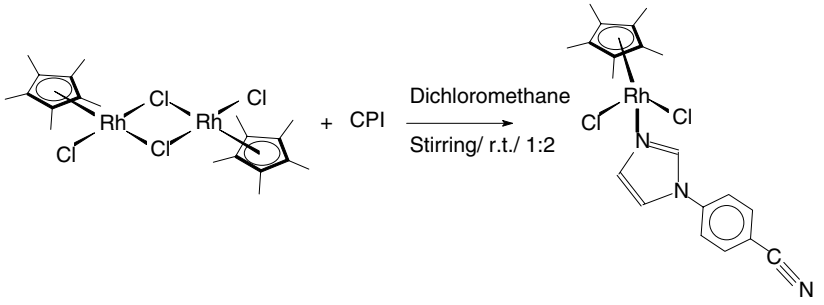
polarization is not possible, leading to entirely different products. Because of our interests in the development of new *metallo-ligands* based on organometallic system, we have reacted the dimeric chloro bridged rhodium complex $[\{(\eta^5\text{-}C_5Me_5)Rh(\mu\text{-}Cl)Cl\}_2]$ with CPI [6]. In this note, we report the synthesis, spectral characterization of new *piano-stool* complex $[\{(\eta^5\text{-}C_5Me_5)RhCl_2(CPI)]$ **1**. We also describe herein, the effect of polarity of the solvents in the ligand substitution reactions of complex **1**.

2. Results and discussion

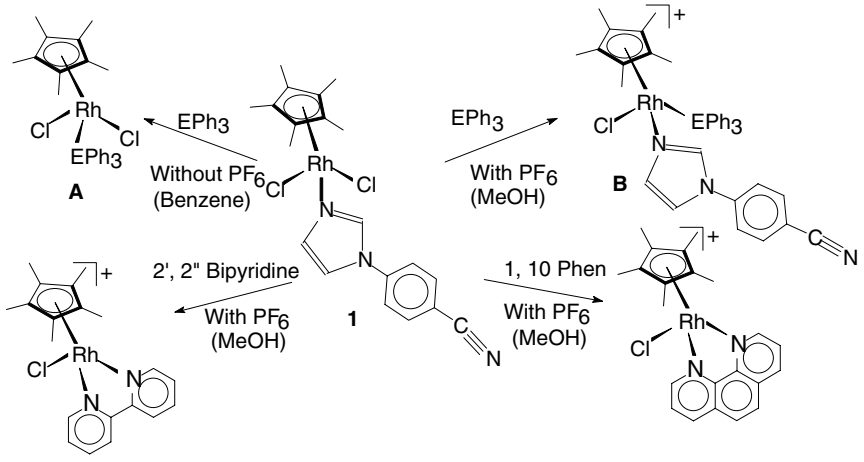
Reactions of the dimeric chloro bridged rhodium complex $[\{(\eta^5\text{-}C_5Me_5)Rh(\mu\text{-}Cl)Cl\}_2]$ with two equivalents of the bridging ligand CPI in dichloromethane under stirring conditions at room temperature gave a neutral complex $[(\eta^5\text{-}C_5Me_5)RhCl_2(CPI)]$ **1** in excellent yield (Scheme 1). This orange complex is air stable and soluble in water and in most of the common organic solvents.

The complex **1**, reacted with various bases viz., EPh_3 (E = P, As and Sb) and N–N donor bases 2,2′-bipyridine and 1,10-phenanthroline under varying reaction conditions. The substitution chemistry depends mainly on the lability and inertness of the metal–ligand link. The strength of the link depends on the nature (acidity or basicity) of different substituents around the metal center along with the nature of the metal center itself. In particular, the strength of a bond is a matter of reaction medium and conditions. Taking these observations in action, one can use polarity of the solvent as a tool to control the metal to ligand bond strength, to yield a set of complexes with tailored charges and substituted ligand.

In the present study, we observed that treatment of the complex **1** with EPh_3 in a polar solvent like methanol afforded cationic mononuclear complexes with the formulation $[(\eta^5\text{-}C_5Me_5)RhCl(CPI)(EPh_3)]^+$ (E = P, **2**; E = As, **3**; E = Sb, **4**). On the other hand, its reaction in a non polar solvent like benzene led in the formation of neutral known complexes $[(\eta^5\text{-}C_5Me_5)RhCl_2(EPh_3)]$ (E = P, As and Sb) (Scheme 2) [7]. In the reactions involving EPh_3, substitution of the chloro group, suggested that the metal to CPI bond in the complex **1** is quite strong. This observation is contrary to our findings on the analogous arene ruthenium and η^3:η^3-bis allyl ruthenium complexes incorporating 4-cyanopyridine



Scheme 1.



Scheme 2.

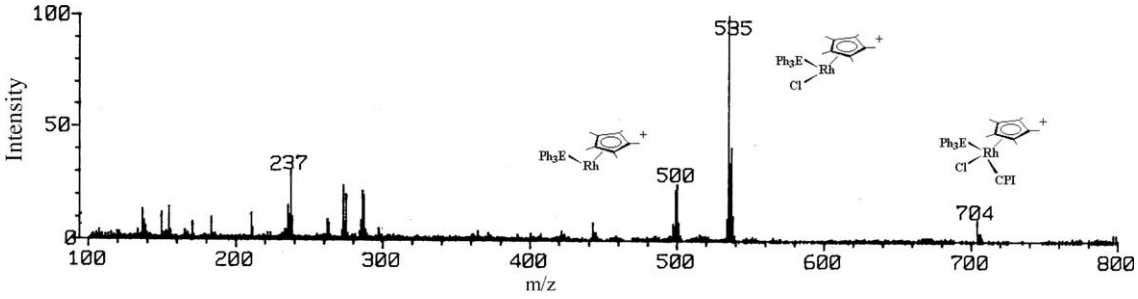


Fig. 1. FAB Mass Spectra of the complex **2** with fragmentation pattern.

wherein, the respective base displaces metal bound 4-cyanopyridine [6a,b]. Clearly, polarization of the Rh–Cl bond in this case is responsible for the formation of cationic complexes. Since, polarization of the Rh–Cl bond is not possible in benzene therefore, it leads to substitution of the coordinated CPI by the respective bases.

In the reactions involving N–N donor bases 2,2′-bipyridine and 1,10-phenanthroline, we could only isolate the complexes $[(\eta^5\text{-}C_5Me_5)RhCl(bipy)]^+$ and $[(\eta^5\text{-}C_5Me_5)RhCl(phen)]^+$ already reported in the literature [9]. Clearly, in these reactions one of the chloro groups and coordinated CPI is being displaced to form $[(\eta^5\text{-}C_5Me_5)Rh(N\text{–}N)Cl]^+$. Steric stability of the cationic complexes $[(\eta^5\text{-}C_5Me_5)Rh(N\text{–}N)Cl]^+$ over the complexes $[(\eta^5\text{-}C_5Me_5)Rh(CPI)(N\text{–}N)]^{2+}$ favored the formation of former species.

Information about the composition of the complexes and structure and bonding has been derived from analytical spectral studies. Analytical data of the complexes conformed well to their respective formulations. More information about composition of the complexes was also obtained from FAB MS. Resulting data is recorded in the experimental section and representative spectrum of the cationic complex **2** is shown in Fig. 1. The position of different peaks and overall fragmentation pattern in the FAB MS of the respective complexes is consistent with their formulations.

The IR spectra of the mononuclear complex **1** and the cationic complexes **2**, **3** and **4** displayed sharp and intense bands around 2226 cm^{-1} corresponding to ν(C≡N). As the position of the ν(C≡N) remained unaltered, it suggested linkage of CPI through its imidazole nitrogen [3]. It was further supported from the blue shifts in the position of ν(C=C) and ν(C–N).

The ^{1}H NMR spectral data of the complexes along with their assignments are recorded in the experimental section. In the ^{1}H NMR spectra of the complex **1–4**, C_5Me_5 protons displayed down field shift as compared to that in the precursor complex. Downfield shift in the position of the C_5Me_5 protons might result from the change in electron density on the metal center due to linkage of CPI through its imidazole nitrogen. The conjugative and electron withdrawing abilities of the –cyano group pulls electron density away from the imidazole nucleus towards itself, leading to a decrease of electron density on the metal center which in turn, may pull more electron density away from the η^5-C_5Me_5, leading to deshielding of η^5-C_5Me_5 protons. The ^{1}H NMR spectral data of neutral complexes of general formulation $[(\eta^5\text{-}C_5Me_5)RhCl_2(EPh_3)]$ are consistent with those already reported in the literature [7,8]. The position and integrated intensity of various resonance supported well the presence of ligand CPI and formulation of the respective complexes. The nitrile carbon of the CPI in the complexes resonated at ~113 ppm, while the other carbons of CPI resonated in the range 123.01–151.90 ppm [9].

The absorption spectral data of the complexes **1–4** is recorded in the experimental section along with the other selected analytical and spectral data of the complexes (Fig. 2). The electronic spectra shows a band at 250–260 nm corresponding to intra-ligand π–π* transition and another band of lower energy in the region 373–403 nm assignable to MLCT transition [10]. The MLCT maxima of the complexes shifted towards lower energy side with an increase in the σ donor ability of the ligand. It is well known that imidazole is a poor accepter in comparison to the benzonitrile, which leads to a weak π-back bonding with the metal center, in turn shift in the

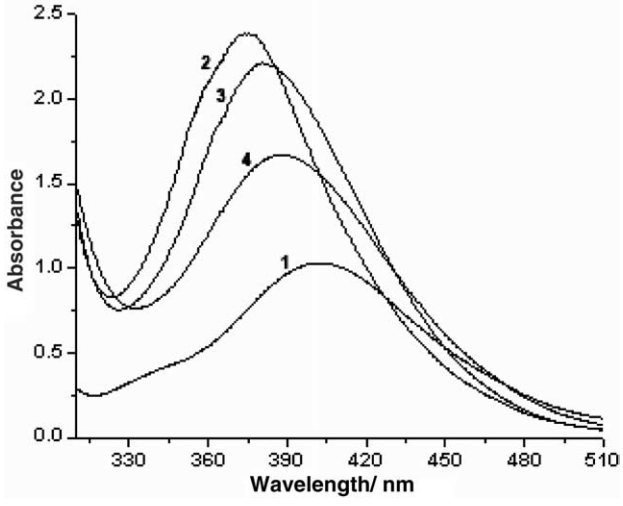


Fig. 2. Electronic absorption spectra of the complexes **1–4**.

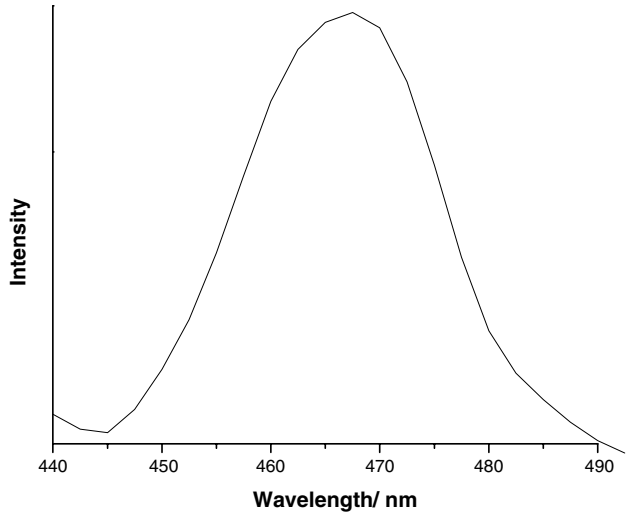


Fig. 3. Emission spectra of the complex **1** in CH_2Cl_2 at *rt*.

position the MLCT to the higher energy side. The latter band can be assigned to the Rh-imidazole moiety. All the substituted products (**2–4**) of the complex **1** showed higher energy MLCT maxima (**1** < **4** < **3** < **2**) than the precursor complex **1**.

The fluorescence spectrum (Fig. 3) of the complex $[(\eta^5\text{-}C_5Me_5)RhCl_2(CPI)]$ **1** shows emission maxima at 467 nm upon excitation at 403 nm with a quantum yield (Φ) of 0.014. λ_{max}^{abs} were taken as excitation λ_{max} for recording the emission spectra. Emission spectrum was obtained in dichloromethane at *rt*. Upon excitation at 403 nm, corresponding to metal to cyano phenyl imidazole (CPI) charge transfer, emission is intense and could not be attributed to the free ligand. The emission maxima and quantum yields for complexes **2**, **3** and **4** are 438 nm (0.018), 442 nm (0.021) and 449 nm (0.023), respectively. The position and shape of the emission band indicated that it is due to MLCT states.

Cyclic voltammogram (Fig. 4) of the complex **1** in acetonitrile exhibits an irreversible oxidation peak at 1.30 V corresponding to Rh^{IV}/Rh^{III} oxidation of rhodium metal center bound to the imidazole and two irreversible reduction peaks at −1.0 and −1.8 V can be assigned to the stepwise reduction of the CPI ligand [3].

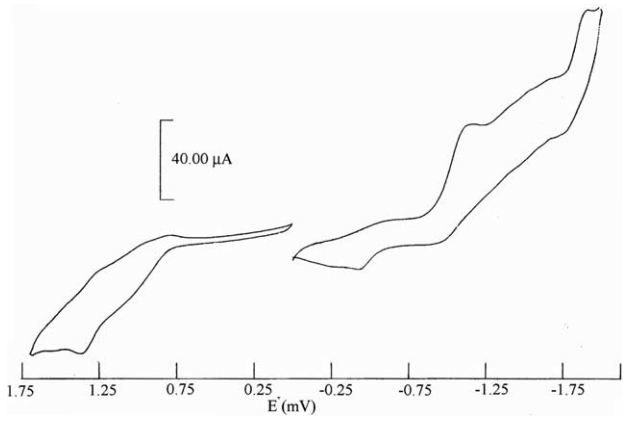


Fig. 4. Cyclic voltammogram of the complex 1.

In this work with the help of analytical and spectral data we have shown that linkage of CPI in the complexes **1–4** is taking place through its imidazole nitrogen and the nitrile group is present as a pendant donor group. Poor quality of crystals of the complex **1** and its derivatives has restricted us to provide any structural support at this stage. Due to presence of *pendant* nitrile group, the complexes **1–4** have the potential to behave as *metallo-ligands* and could be employed in the synthesis of homo/hetero binuclear mixed valence systems having interesting properties. The results reported herein also demonstrated the role of solvent to control substitution chemistry about the metal center and resulting into a complex with a choice of substituted ligand.

3. Experimental

All the synthetic manipulations were performed under oxygen free nitrogen atmosphere. The solvents were dried and distilled before use following the standard procedures. Pentamethylcyclopentadiene, triphenylphosphine, triphenylarsine, 2,2′-bipyridine, 1,10-phenanthroline, hydrated rhodium(III) chloride, ammonium hexafluorophosphate (all Aldrich) were used as received. The ligand 1-(4-cyanophenyl)-imidazole [3] and the precursor complex $[\{(\eta^5\text{-}C_5Me_5)Rh(\mu\text{-}Cl)Cl\}_2]$ were prepared and purified following the literature procedures [11]. Elemental analyses, spectral and electrochemical data of the complexes were obtained as described elsewhere [12].

3.1. Preparation of complexes

3.1.1. Preparation of $[(\eta^5\text{-}C_5Me_5)RhCl_2(CPI)]$ (1)

To a suspension of $[\{(\eta^5\text{-}C_5Me_5)Rh(\mu\text{-}Cl)Cl\}_2]$ (0.310 g, 0.5 mmol) in 25 ml of dichloromethane, CPI (0.169 g, 1.0 mmol) was added and contents of the flask were stirred at *rt* for 5 h. The resulting yellow-orange solution was filtered through celite to remove any solid impurities. The filtrate was concentrated under vacuum to dryness and washed several times with diethyl ether. The solid mass was extracted with dichloromethane, filtered and was precipitated by addition of petroleum ether (60–80°). The resulting crystalline product was filtered, washed twice with methanol and petroleum ether (60–80°). Yield (0.182 g, 76%) (Found: C, 50.27; H, 4.62; N, 8.69% M 478 $C_{20}Cl_2H_{22}N_3Rh$: C, 50.21; H, 4.60; N, 8.79%). IR (cm^{-1}, nujol): 2221.8. λ_{max}/nm, CH_2Cl_2 (ε/$dm^3\ mol^{-1}\ cm^{-1}$): 403(2060), 260(190325), 238(179768). ^1H NMR (300 MHz; $CDCl_3$, $SiMe_4$, *J* Hz): δ 1.68(s, 15H, $\eta^5\text{-}C_5Me_5$), 8.48(s, 1H), 7.77(d, 1H, 9.0Hz), 7.80(d, 1H, 9.1Hz), 7.47(d, 2H, 6.0Hz), 7.41(d, 2H, 9.0Hz). $^{13}C\{^1H\}$ NMR

($CDCl_3$): 151.40(αC–Imd), 148.60(α′C–Imd), 132.90 (αC–CNPh), 129.40(γC–CNPh), 127.21(βC–CNPh), 124.43(δC–CNPh), 124.30(βC–Imd), 112.12(CN–CNPh), 93.46(CC–Cp*), 9.49(CH_3–Cp*). FAB-MS *m/z* 479(478), $[M]^+$; 443(442), $[M]^+$–Cl; 274(273), $[M]^+$–Cl–CPI; 238(236) $[M]^+$–Cl–CPI–Cl.

3.1.2. Preparation of $[(\eta^5\text{-}C_5Me_5)RhCl(CPI)(PPh_3)]PF_6$ (2)

To a suspension of $[(\eta^5\text{-}C_5Me_5)RhCl_2(CPI)]$ (0.478 g, 1.0 mmol) in 25 ml of methanol, PPh_3 (0.262 g, 1.0 mmol) was added and contents of the flask were heated under reflux for 1 h. The resulting dark red solution was cooled to room temperature and filtered through celite. Ammonium hexafluorophosphate dissolved in 10 ml of methanol was added to the filtrate, whereupon a red crystalline solid separated. It was filtered and washed twice with methanol and diethyl ether. Yield (0.613 g, 72%) (Found: C, 53.61; H, 4.62; N, 5.19% M 849 $C_{38}ClF_6H_{37}N_3P_2Rh$: C, 53.71; H, 4.36; N, 4.95%). IR (cm^{-1}, nujol): 2230.0. λ_{max}/nm, CH_2Cl_2 (ε/$dm^3\ mol^{-1}\ cm^{-1}$): 373(4777), 251(248329), 232(189671). ^1H NMR (300 MHz; $CDCl_3$, $SiMe_4$, *J* Hz): δ 1.47 (s, 15H, η^5-C_5Me_5), 7.21–7.45 (br. m, aromatic protons of PPh_3), 7.98(s, 1H), 7.18(d, 1H, 8.8Hz), 7.77(d, 2H, 8.4Hz), 7.44(d, 2H, 8.5Hz), 7.60(d, 2H, 8.7Hz), ^{31}P NMR (300 MHz): 10.85(s, PPh_3) −149.52 (sep., PF_6). $^{13}C\{^1H\}$ NMR ($CDCl_3$): 151.90(αC–Imd), 148.30 (α′C–Imd), 132–138.67(m, Ph), 131.50(αC–CNPh), 129.01(γC–CNPh), 128.20(βC–CNPh), 125.43(δC–CNPh), 123.01(βC–Imd), 113.11(CN–CNPh), 90.34(CC–Cp*), 8.68(CH_3–Cp*). FAB-MS *m/z* 704(706), $[M]^+$; 535(535), $[M]^+$–CPI; 500(500) $[M]^+$–CPI–Cl; 237(237) $[M]^+$–CPI–Cl–PPh_3.

3.1.3. Preparation of $[(\eta^5\text{-}C_5Me_5)RhCl(CPI)(AsPh_3)]PF_6$ (3)

It was prepared by following the above procedure (**2**) except that $AsPh_3$ was used in place of PPh_3. It was isolated as orange crystalline solid. Yield (0.608 g, 68%) (Found: C, 51.25; H, 4.45; N, 4.88% M 894 $AsC_{38}ClF_6H_{37}N_3PRh$: requires C, 51.01; H, 4.14; N, 4.69%). IR (cm^{-1}, nujol): 2229.3. λ_{max}/nm, CH_2Cl_2 (ε/$dm^3\ mol^{-1}\ cm^{-1}$): 380(4428), 255(215560), 234(183465).^1H(300 MHz; $CDCl_3$, $SiMe_4$, *J* Hz): δ 1.55 (s, 15H, η^5-C_5Me_5), 7.24–7.56 (br. m, aromatic protons of $AsPh_3$), 8.04(s, 1H), 7.24(d, 1H, 9.0), 7.75(d, 2H, 8.7Hz), 7.64(d, 2H, 8.7Hz), 7.62(d, 2H, 8.9Hz), ^{31}P NMR (300 MHz): −150.20 (sep., PF_6). $^{13}C\{^1H\}$ NMR ($CDCl_3$): 151.60(αC–Imd), 148.91 (α′C–Imd), 132–136.68(m, Ph), 130.12(αC–CNPh), 129.20(γC–CNPh), 128.60(βC–CNPh), 125.01(βC–Imd), 122.80 (δC–CNPh), 115.50(CN–CNPh), 98.14 (CC–Cp*), 9.52(CH_3–Cp*). FAB-MS *m/z* 748(749), $[M]^+$; 579(580), $[M]^+$–CPI; 544(544), $[M]^+$–CPI–Cl; 237(238) $[M]^+$–CPI–Cl–$AsPh_3$.

3.1.4. Preparation of $[(\eta^5\text{-}C_5Me_5)RhCl(CPI)(SbPh_3)]PF_6$ (4)

It was prepared by following the above procedure (**2**) except that $SbPh_3$ was used in place of PPh_3. It was isolated as orange crystalline solid. Yield (0.641 g, 68%) (Found: C, 48.50; H, 3.96; N, 4.60% M 942 $C_{38}ClF_6H_{37}N_3PRhSb$: requires C, 48.41; H, 3.93; N, 4.46%). IR (cm^{-1}, nujol): 2224.1. λ_{max}/nm, CH_2Cl_2 (ε/$dm^3\ mol^{-1}\ cm^{-1}$): 386(3336), 258(204321), 236(181260). ^1H NMR (300 MHz; $CDCl_3$, $SiMe_4$, *J* Hz): δ 1.64 (s, 15H, η^5-C_5Me_5), 7.27–7.61(br. m, aromatic protons of $AsPh_3$), 8.21(s, 1H), 7.33(d, 1H, 9.1), 7.85(d, 2H, 8.9Hz), 7.70(d, 2H, 8.9Hz), 7.69(d, 2H, 9.0Hz), ^{31}P NMR (300 MHz): −149.16(sep., PF_6). $^{13}C\{^1H\}$ NMR ($CDCl_3$): 151.21(αC–Imd), 148.54(α′C–Imd), 132–136.67(m, Ph), 130.52(γC–CNPh), 129.51(αC–CNPh), 128.21(βC–CNPh), 124.62(βC–Imd), 121.50(δC–CNPh), 120.80(CN–CNPh), 90.31(CC–Cp*), 8.82(CH_3–Cp*). FAB-MS *m/z* 797(796), $[M]^+$; 628(629), $[M]^+$–CPI; 592(591), $[M]^+$–CPI–Cl; 238(238) $[M]^+$–CPI–Cl–$SbPh_3$.

Acknowledgments

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