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Carbon–fluorine bond cleavage as a route to hybrid ligands

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Abstract

Intramolecular C–F bond cleavage provides a convenient, high yield route to metal complexes of hybrid cyclopentadienyl-phosphine ligands. On treatment with proton sponge the complexes $[Cp^*RhCl\{(C_6F_5)_2PCH_2CH_2P(C_6F_5)_2\}]BF_4$ and $[Cp^*RhCl\{(C_6F_5)_2PC_6H_4SMe\text{-}2\}]BF_4$ undergo dehydrofluorinative C–C coupling to give the complexes $[\{\eta^5,\eta^1,\eta^1\text{-}C_5Me_3[CH_2C_6F_4P(C_6F_5)CH_2]_2\text{-}1,3\}RhCl]BF_4$ and $[\{\eta^5,\eta^1,\eta^1\text{-}C_5Me_4CH_2C_6F_4P(C_6F_5)C_6H_4SMe\text{-}2\}RhCl]BF_4$ rapidly and in quantitative yield. The reaction between $[Cp^*RhCl\{PPh_2(C_6F_5)\}(CNR)]BF_4$ (R = phenyl or cyclohexyl) and proton sponge produces $[(\eta^5,\eta^1\text{-}C_5Me_4CH_2C_6F_4PPh_2)RhCl(CNR)]BF_4$, but is far slower and much less clean than for the complexes of the chelating ligands. The neutral complex $[Cp^*RhCl_2\{PPh_2(C_6F_5)\}]$ undergoes no reaction with proton sponge. The proposed mechanism involves initial formation of an η^4-fulvene complex, and subsequent nucleophilic attack (S_NAr) at an *ortho*-C–F bond.

Keywords: C–F bond cleavage; S_NAr reactions; Polyfluoroarylphosphines; Hybrid ligands

1. Introduction

We have reported that on heating in ethanol the organometallic salt $[Cp^*RhCl(dfppe)]BF_4$, **1**, [$Cp^* = \eta^5\text{-}C_5Me_5$; dfppe = $(C_6F_5)_2PCH_2CH_2P(C_6F_5)_2$] undergoes stepwise intramolecular C–F cleavage with concomitant C–C bond formation to give $[\{\eta^5,\eta^1,\eta^1\text{-}C_5Me_3[CH_2C_6F_4P(C_6F_5)CH_2]_2\}RhCl]BF_4$, **2**, in quantitative yield (Scheme 1) [1]. The product is a complex of a novel trifunctional hybrid cyclopentadienyl-bis(phosphine) ligand, and the reaction is a rare example of intramolecular coupling of a η^5-cyclopentadienyl ligand to a phosphine ligand. The coupling of these two ligand types, which are two of the most common and important employed in organometallic chemistry, in chelating bi- or trifunctional ligands is currently an area of intense activity, since the resulting hybrid ligands are expected to exert different effects on metal reactivity compared to the separated ligands [2]. Despite the anticipated advantages of these ligands, the number of complexes of chelating hybrid cyclopentadienyl-phosphine ligands is limited [2]. One reason for this scarcity is the lack of convenient syntheses. The strategy most commonly employed is that of synthesis of the hybrid ligand and subsequent complexation. However, the ligand syntheses are often elaborate, involving multiple steps, and consequently poor overall yields are obtained [2]. The alternative strategy of coordination of both functionalities of the hybrid ligand to the metal followed by a metal-templated intramolecular coupling offers a method of overcoming this problem. The two functionalities are held in close proximity by coordination to the metal and therefore fast rates and high yields coupled with high regioselectivities are expected. Despite the appeal of this approach there are very few reports of intramolecular reactions leading to complexes of hybrid cyclopentadienyl-phosphine ligands: decafluorodiazabenzene reacts with $CpRuMe(PPh_3)_2$ to give $[(\eta^5,\eta^1\text{-}C_5H_4C_6H_4PPh_2)RuC_6F_4N{=}NC_6F_5]$ in moderate yield [3,4], the rhodium complex cation $[Cp^*RhCl(PPh_2CH{=}CH_2)_2]^+$ undergoes radical or base promoted hydroalkylation to give a mixture of the 1,2 and 1,3 isomers of $[\{\eta^5,\eta^1,\eta^1\text{-}C_5Me_3(CH_2CH_2CH_2PPh_2)_2\}RhCl]^+$ in 35–42% yield, respectively [5,6], and in addition to reaction 1, we have reported that in refluxing ethanol the salts $[(\eta^5\text{-}C_5Me_4R)MX(dfppe)]BF_4$ (M = Rh, X = Cl or Br, R = H, Me or Et, M = Ir, X = Cl, R = Me) undergo dehydrofluorinative C–C coupling, to give $[\{\eta^5,\eta^1,\eta^1\text{-}C_5Me_2R[CH_2C_6F_4P(C_6F_5)CH_2]_2\}MX]BF_4$ in virtually quantitative yield [1,7–9]. Here we report that reaction 1 can be performed by addition of 1,8-bis(dimethylamino)naphthalene (proton sponge), propose a mechanism involving S_NAr nucleophilic attack at an aryl C–F bond, and use this mechanism as a basis for developing a convenient route to other hybrid ligands, exemplified by the synthesis of a rhodium complex of the first η^5,η^1,η^1-cyclopentadienyl-phosphine-thioether ligand. Part of this work has been communicated [10].

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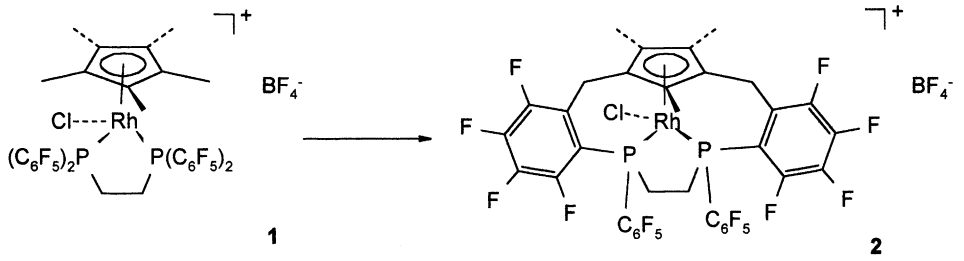


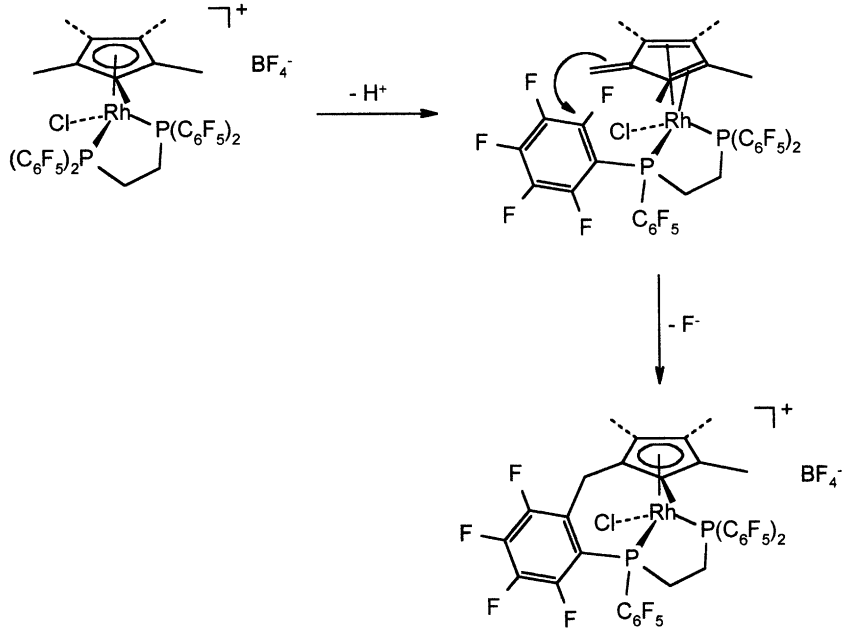
Scheme 1. Reaction 1.

2. Results and discussion

Although reaction 1 does not occur in the presence of triethylamine [1] and on treatment with potassium *t*-butoxide **1** gives a complicated mixture of products, but not **2**, treatment of **1** with two equivalents of the strong, non-nucleophilic base, proton sponge, at room temperature yields **2** in quantitative yield. An NMR tube experiment indicated that the reaction is rapid, reaching completion within 15 min. The difference in reactivity between proton sponge and the other bases is ascribed to the non-nucleophilic and non-coordinating nature of the former. On the basis of this observation, we propose a mechanism for this reaction involving initial formation of an η^4-fulvene complex by loss of a pentamethylcyclopentadienyl proton, followed by nucleophilic attack of the methylene carbon atom at the *ortho*-position of the pentafluorophenyl group (Scheme 2). In support of this mechanism it has previously been established that the methylene carbon atoms of η^4-fulvene rhodium complexes are nucleophilic [11,12] and that polyfluorinated arenes are susceptible to nucleophilic attack [13,14]. This mechanism has previously been proposed by Hughes et al. for the similar dehydrofluorinative C–C coupling reaction between the pentamethylcyclopentadienyl and perfluorobenzyl ligands of $[Cp^*CoI(CF_2C_6F_5)(PMe_3)]$ [15]. The concept of an intramolecular S_NAr reaction involving C–F bond cleavage coupling two functionalities provides a simple route to transition metal complexes of other hybrid cyclopentadienyl-phosphine ligands. Furthermore, the quantitative yield and fast rate of reaction 1 suggest that this route may offer the desired advantages over the more traditional approach to preparing hybrid ligands. In order to establish the limitations of this methodology we have investigated the action of proton sponge on a range of pentamethylcyclopentadienylrhodium pentafluorophenylphosphine complexes.

The neutral monodentate phosphine complex $[Cp^*RhCl_2\{PPh_2(C_6F_5)\}]$ (**3**) has been described previously [8]. Intramolecular dehydrofluorinative C–C coupling of **3** would be expected to yield the complex $[(\eta^5,\eta^1\text{-}C_5Me_4CH_2\text{-}C_6F_4PPh_2)RhCl_2]$. However, on addition of proton sponge to **3** no reaction occurred, even after prolonged periods.

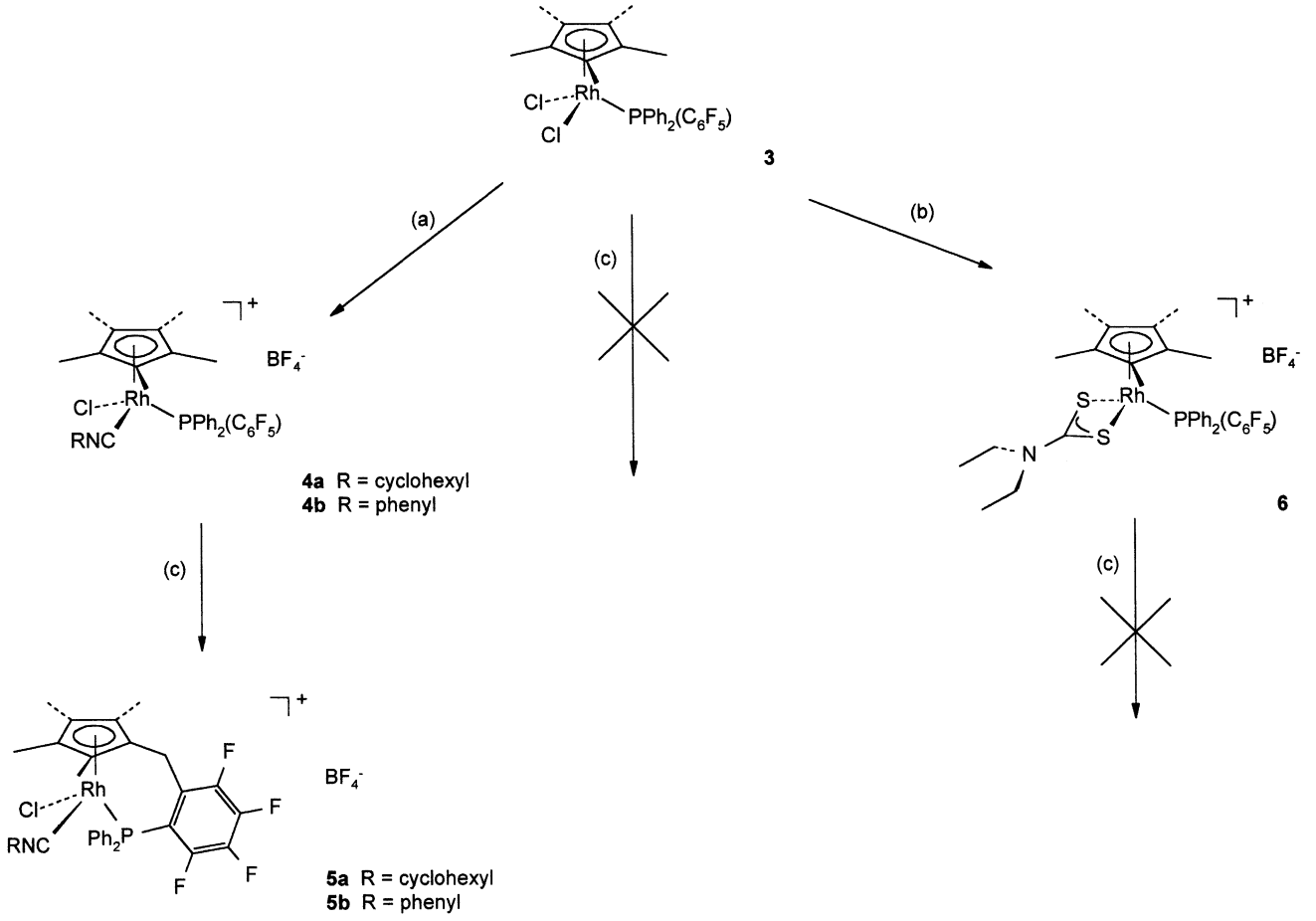
The contrast between reaction 1 and the lack of reaction of **3** with proton sponge suggests that it may be necessary for



Scheme 2. Proposed mechanism for the dehydrofluorinative carbon–carbon coupling reaction of **1**.

the complex to be cationic for dehydrofluorinative C–C coupling to occur. It is anticipated that the acidity of the pentamethylcyclopentadienyl hydrogen atoms would be enhanced by the positive charge. In order to test this hypothesis cationic complexes of formulation $[Cp^*RhXL\{PPh_2(C_6F_5)\}]^+$, where X and L are one and two-electron donor ligands, respectively, were investigated. Attempts to prepare the bis(phosphine) complex cation $[Cp^*RhCl\{PPh_2(C_6F_5)\}_2]^+$ were unsuccessful, presumably for steric reasons [8]. On treatment with phenyl- or cyclohexyl-isonitrile in the presence of an excess of sodium tetrafluoroborate **3** yielded the salts $[Cp^*RhCl(CNR)\{PPh_2(C_6F_5)\}]BF_4$ (**4a** R = cyclohexyl, **4b** R = phenyl) as yellow oils (Scheme 3).

NMR tube experiments, performed in $CDCl_3$, indicated that both compounds **4a** and **4b** underwent reactions on treatment with proton sponge as evidenced by the appearance of new resonances in the ^1H, ^{19}F and $^{31}P\{^1H\}$ NMR spectra, which increased in intensity with time, whilst those assigned to **4a** and **4b** diminished. However, in neither case was a single product formed. After 68 h the $^{31}P\{^1H\}$ NMR spectrum of the reaction of **4a** with proton sponge showed only a doublet resonance at $\delta = 41.7$ with a coupling $^1J_{RhP} = 132$ Hz, and the ^{19}F NMR spectrum showed predominantly four resonances of equal intensity at $\delta = -120.61$, -137.09, -146.30 and -152.01, in addition to the BF_4^- resonances, indicative of the loss of one fluorine atom from the pentafluorophenyl ring. The ^1H NMR spectrum, although complicated by the presence of resonances assigned to protonated proton sponge, clearly showed that the Cp^* ligand had undergone reaction. The doublet resonance at $\delta = 1.67$ was no longer evident and the region 1–2.5 ppm, although further complicated by cyclohexylisonitrile resonances, contained two new doublets at $\delta = 2.12$ and 1.86 with couplings $J_{PH} = 4.7$ and 6.6 Hz and two new singlets at $\delta = 1.78$ and 1.32. These resonances are consistent with four non-equivalent methyl groups of a η^5-tetramethylcyclopentadienyl ligand of a chiral-at-metal complex, and occur at similar δ as those of the methyl groups of $[(\eta^5\text{-}C_5Me_4H)RhCl(CNC_6H_{11})\{PPh_2(C_6F_5)\}]BF_4$ ($\delta = 1.91$, 1.82, 1.76 and 1.39) [16]. These data are consistent with the expected product $[(\eta^5,\eta^1\text{-}C_5Me_4CH_2C_6F_4PPh_2)RhCl(CNC_6H_{11})]BF_4$ (**5a**) (Scheme 3). Further, two new doublet of doublet resonances, each integrating for one hydrogen, are observed at $\delta = 3.64$ and 3.30, with a mutual coupling of $J = 16.0$ Hz and coupling to phosphorus of 11.8 and 5.6 Hz, respectively, which are consistent with two non-equivalent methylene hydrogen atoms [1]. The shift of the phosphorus resonance to higher frequency on going from **4a** to **5a** is also consistent with



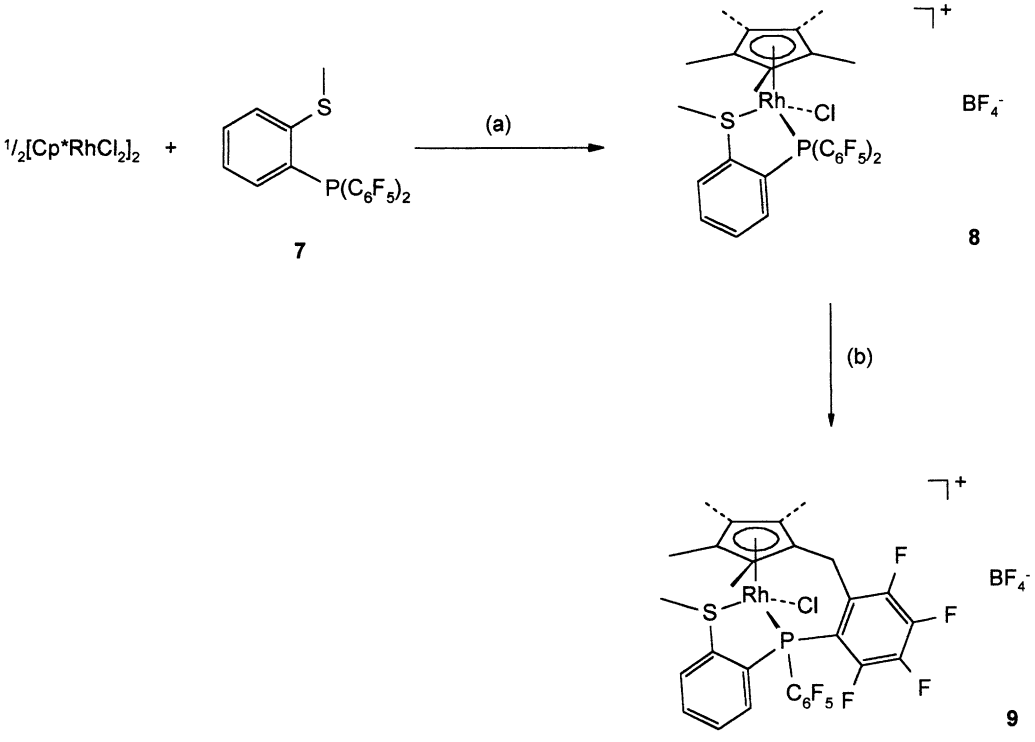
Scheme 3. (a) RNC, $NaBF_4$, CH_2Cl_2–MeOH; (b) NaS_2CNEt_2, CH_2Cl_2–MeOH; (c) proton sponge, $CDCl_3$.

the shift to higher frequency on going from **1** ($\delta = 35.1$) to **2** ($\delta = 77.1$) [1].

The reaction between **4b** and proton sponge was much less clean than that of **4a**. After several hours the $^{31}P\{^1H\}$ NMR spectra of the reaction of **4b** contained a prominent doublet at $\delta = 40.9$ with a coupling $^1J_{RhP} = 133$ Hz, along with a number of resonances in the region 20–30 ppm. The presence of several resonances in the ^{19}F NMR spectra confirmed the formation of a number of different products. However, four resonances of equal integration were evident at $\delta = -119.22$, -136.07, -145.54, -151.82, which are very similar to those assigned to **5a**. The disappearance of the Cp^* resonance in the ^1H NMR spectrum confirmed that a reaction involving the Cp^* ligand had occurred. The similarity of the ^{19}F and ^{31}P NMR data with that of **5a** suggests that treatment of **4b** with proton sponge yields **5b**.

In contrast to **4a** and **4b**, no reaction was observed on treatment of the dithiocarbamate complex $[Cp^*Rh(S_2CNEt_2)\{PPh_2(C_6F_5)\}]BF_4$ (**6**), formed by treatment of **3** with sodium dithiocarbamate, with stoichiometric or excess quantities of proton sponge, even after prolonged periods.

The observation that the intramolecular dehydrofluorinative C–C coupling reaction of **1** proceeds rapidly and in high yield, whereas that for **4a** and **4b** proceeds considerably more slowly and less cleanly, suggests that chelation may be an important factor in determining both the kinetics of the reaction and the absence of side reactions. Whereas in **4a** and **4b**, there is rotation about the Rh–P bond, in complex **1** this cannot occur and one pentafluorophenyl ring of each $P(C_6F_5)_2$ moiety is held close to the pentamethylcyclopentadienyl ring [1]. Thus the reaction of **1** is expected to be more rapid, and if the η^4-fulvene complexes derived from **4a** and **4b** are reactive towards other molecules, such as the solvent, then their slow rate of intramolecular dehydrofluorinative C–C coupling would allow side reactions to occur. In order to confirm the effects of chelation we studied the reaction involving the bifunctional phosphine-thioether $(C_6F_5)_2PC_6H_4SMe$-1,2 (**7**) [10]. The reaction of $[Cp^*RhCl_2]_2$ with **7** in the presence of an excess of tetrafluoroborate afforded $[Cp^*RhCl(\mathbf{7})]BF_4$ (**8**) in ca. 80% yield [10]. An NMR tube experiment indicated that on treatment with proton sponge **8** undergoes a rapid and clean reaction. The $^{31}P\{^1H\}$ NMR spectrum showed a doublet resonance at $\delta = 58.6$, with coupling $^1J_{RhP} = 150$ Hz, which is ca. 30 ppm to higher frequency than that of **8** ($\delta = 30.2$), and the ^{19}F NMR spectrum showed the presence of only nine fluorine atoms. The ^1H NMR spectrum showed four resonances between $\delta = 1.5$ and 2.5 consistent with four non-equivalent methyl groups of a η^5-tetramethylcyclopentadienyl ligand of a chiral-at-metal complex [16], and two mutually coupled methylene resonances at $\delta = 4.36$ and 3.02, the first of which is also coupled to phosphorus, as well as resonances due to the SMe and C_6H_4 hydrogen atoms. These data are entirely consistent with the expected product $[\{\eta^5,\eta^1,\eta^1\text{-}C_5Me_4CH_2C_6F_4P(C_6F_5)C_6H_4SMe\text{-}2\}RhCl]BF_4$ (**9**). Compound **9** was synthesized on a preparative scale in 92% yield by treatment of **8** with proton sponge in chloroform (Scheme 4) and its structure has been determined by single-crystal X-ray diffraction [10]. The rapid and clean conversion of **8–9** mirrors that of **1–2** and confirms that



Scheme 4. (a) $NaBF_4$, CH_2Cl_2–MeOH; (b) proton sponge, $CHCl_3$.

chelation of the phosphine ligand has a profound effect on the kinetics of the reaction. The difference between the reactions involving chelating and monodentate phosphines can be ascribed to the free rotation about the Rh–P bond in the latter. In the complexes **1** and **8** the two reacting fragments, η^5-C_5Me_5 and C_6F_5, are held in close proximity [1], whereas in **4a** and **4b** rotation about the Rh–P bond moves the fragments apart.

3. Conclusion

On treatment with proton sponge rhodium complexes containing η^5-pentamethylcyclopentadienyl and pentafluorophenylphosphine ligands can undergo dehydrofluorinative C–C coupling to produce rhodium complexes of hybrid cyclopentadienyl-phosphine ligands. The proposed mechanism of the reaction involves formation of an η^4-fulvene complex and subsequent nucleophilic attack at an *ortho*-C–F bond. A necessary requirement of the reaction is that the complex is a cation. Reactions involving complexes of the monodentate phosphine $PPh_2(C_6F_5)$ are slow and product formation is not quantitative. In contrast, reactions of complexes of chelating ligands containing at least one $P(C_6F_5)_2$ moiety are rapid and quantitative. It is postulated that this is a result of free rotation about the Rh–P bonds in the monophosphine complexes. In summary, intramolecular dehydrofluorinative C–C coupling provides a convenient, high yield alternative to the more common routes to hybrid cyclopentadienyl-phosphine ligands.

4. Experimental

4.1. Instrumentation

The ^1H, ^{19}F and ^{31}P NMR spectra were recorded using a Bruker DPX300 spectrometer. ^1H (300.01 MHz) were referenced internally using the residual protio solvent resonance relative to $SiMe_4$ ($\delta = 0$), ^{19}F (282.26 MHz) externally to $CFCl_3$ ($\delta = 0$) and ^{31}P (121.45 MHz) externally to 85% H_3PO_4 ($\delta = 0$). All chemical shifts are quoted in δ (ppm), using the high frequency positive convention, and coupling constants in Hz. Elemental analyses were carried out by ASEP, The School of Chemistry, The Queen's University of Belfast.

4.2. Materials

The compounds $[Cp^*RhCl_2]_2$, $NaBF_4$, CNC_6H_{11}, $PPh_2(C_6F_5)$ (Aldrich) were used as supplied. PhNC [17], **1** [1] and **3** [8] were prepared as described.

4.3. Preparations

The preparation and characterising data for compounds **7–9** has been reported [10].

4.3.1. *$[Cp^*RhCl\{PPh_2(C_6F_5)\}(CNC_6H_{11})]BF_4$ (**4a**)*

The compound **3** (0.110 g, 0.170 mmol) was treated with $NaBF_4$ (0.022 g, 0.200 mmol) and cyclohexylisonitrile (0.022 g, 0.200 mmol) as described for the preparation of $[(\eta^5\text{-}C_5Me_4H)RhCl\{PPh_2(C_6F_5)\}(CNC_6H_{11})]^+\cdot BF_4^-$ [16]. The product was obtained as a yellow oil in virtually quantitative yield. Repeated recrystallisation failed to give solid product, and elemental analysis could not be obtained. Characterisation is based on the NMR spectroscopic data and comparison with similar compounds [16]. ^1H NMR (300.1 MHz, $CDCl_3$): $\delta = 7.77$ (2H, m, C_6H_5), 7.47 (8H, m, C_6H_5), 4.17 (^1H, m, CNCH), 2.05 (2H, m, C_6H_{11}), 1.67 (15H, d, $J_{PH} = 4.2$ Hz, Cp^*), 1.61 (8H, m, C_6H_{11}). ^{19}F (282.4 MHz, $CDCl_3$): $\delta = -123.79$ (2F, br, *o*-F), -144.21 (1F, br, *p*-F), -153.91 (0.8F, s, $^{10}BF_4^-$), -153.96 (3.2F, s, $^{11}BF_4^-$), -157.38 (2F, br, *m*-F). $^{31}P\{^1H\}$ (121.5 MHz, $CDCl_3$): $\delta = 22.19$ (dm, $^1J_{RhP} = 129$ Hz).

4.3.2. *$[Cp^*RhCl\{PPh_2(C_6F_5)\}(CNPh)]BF_4$ (**4b**)*

The compound **4b** was prepared as described in Section 4.3.1 from **3**, prepared in situ from $[Cp^*RhCl_2]_2$ (0.200 g, 0.330 mmol) and $PPh_2(C_6F_5)$ (0.236 g, 0.670 mmol) using phenylisonitrile (0.066 g, 0.640 mmol). Yield: 0.500 g, 94%. Repeated recrystallisation failed to give solid product, and elemental analysis could not be obtained. Characterisation is based on the NMR spectroscopic data and comparison with similar compounds [16]. ^1H NMR (300.1 MHz, $CDCl_3$): $\delta = 7.86$ (2H, m, C_6H_5), 7.42 (11H, m, C_6H_5), 6.91 (2H, d, $J = 7.5$ Hz, C_6H_5), 1.75 (15H, d, $J_{PH} = 4.2$ Hz, Cp^*). ^{19}F (282.4 MHz, $CDCl_3$): $\delta = -124.15$ (2F, m, *o*-F), -143.86 (1F, m, *p*-F), -153.81 (0.8F, s, $^{10}BF_4^-$), -153.86 (3.2F, s, $^{11}BF_4^-$), -157.24 (2F, m, *m*-F). $^{31}P\{^1H\}$ (121.5 MHz, $CDCl_3$): $\delta = 22.6$ (dm, $^1J_{RhP} = 130$ Hz).

4.3.3. *$[Cp^*Rh(S_2CNEt_2)\{PPh_2(C_6F_5)\}]BF_4$ (**6**)*

To a solution of $PPh_2(C_6F_5)$ (0.090 g, 0.252 mmol) and $[Cp^*RhCl_2]_2$ (0.102 g, 0.126 mmol) in dichloromethane (30 cm^3) and methanol (20 cm^3), was added $NaBF_4$ (0.143 g, 1.30 mmol) followed by sodium dithiocarbamate (0.056 g, 0.252 mmol) in methanol (10 cm^3). After stirring at room temperature for 12 h, the volatiles were removed by rotary evaporation and the product extracted into dichloromethane (3 cm × 20 cm^3). Removal of the solvent by rotary evaporation gave an orange solid, which was washed with hexane (2 cm × 10 cm^3) and dried in vacuo. Yield: 0.197 g (89%). ^1H NMR (300.1 MHz, $CDCl_3$): $\delta = 7.48$ (10H, m, C_6H_5), 3.36 (4H, quartet, $^3J_{FF} = 7.2$ Hz, NCH_2), 1.49 (15H, d, $J_{PH} = 3.6$ Hz, Cp^*), 1.00 (6H, t, $^3J_{FF} = 7.2$ Hz, NCH_2CH_3). ^{19}F (282.4 MHz, $CDCl_3$): $\delta = -123.93$ (2F, m, *o*-F), -144.39 (1F, m, *p*-F), -154.34 (0.8F, s, $^{10}BF_4^-$), -154.39 (3.2F, s, $^{11}BF_4^-$), -157.50 (2F, m, *m*-F). $^{31}P\{^1H\}$ (121.5 MHz, $CDCl_3$): $\delta = 18.3$ (dm, $^1J_{RhP} = 155$ Hz). Anal. Calcd for $C_{33}H_{35}BF_9NPRhS_2$: C, 48.0; H, 4.3; N, 1.7. Found: C, 48.35; H, 4.6; N, 1.4.

Acknowledgements

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