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Activation of aliphatic carbon–carbon bonds of esters and amides by rhodium(II) porphyrin

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Abstract

Aliphatic carbon–carbon bonds of esters and amides were activated successfully with rhodium(II) porphyrin radical to give rhodium(III) porphyrin alkyls in moderate yields.

Keywords: Rhodium(II) porphyrin; Esters; Amides; Carbon–carbon bond activation

1. Introduction

The activations of inert chemical bonds are of fundamental importance in chemical research and industrial applications [1–3]. The activation of an aliphatic carbon–carbon bond (CCA) is one of the most challenging reactions. The development of efficient methods for selective cleavage of carbon–carbon bonds in homogeneous media by transition metal complexes is an important topic of organometallic chemistry [4]. Carbon–carbon bond activation could be applied in developing an environmentally benign process by the depolymerization of synthetic polymer waste into lower and biodegradable hydrocarbon fragments [5].

Examples of carbon–carbon bonds activation by transition metal complexes mainly involved ring strain relief [6–8], proximal carbonyl group assistance [9–11], pre-anchored phosphine ligands [12], and nucleophilic openings of three-membered rings [13]. Also photochemically induced C–CN bond cleavage mediated by iron complexes was documented by Nakazawa et al. [14,15]. Silylation of nitriles involving C–CN bond cleavage in catalytic manner was reported by Chatani recently [16]. The difficulties of the activation of unstrained aliphatic carbon–carbon bonds [17] lie in the activation of kinetically more favorable, sterically unhindered carbon–hydrogen over carbon–carbon bonds.

We have reported the first examples of activation of aliphatic carbon–carbon bonds of nitroxide [18] and ketones [19] by 5,10,15,20-tetra(2,4,6-trimethylphenyl)porphyrinate rhodium(II) [Rh(tmp)]. We have explored the activation of carbon–carbon bond of esters and amides to examine the importance of heteroatom assisted pre-coordination [20–22] in CCA. The CCA results of ketones have demonstrated that non-enolizable ketones showed higher selectivity than that of enolizable ketones towards to carbon–carbon bond activation due to elimination of competitive carbon hydrogen bond activation [19]. The CCA reactions were also promoted by Ph_3P. Hence, we now report the successful activation of aliphatic carbon–carbon bonds in non-enolizable esters and amides by Rh(tmp) radical in the presence of triphenylphosphine as a promoter ligand [23–25].

2. Results and discussion

The metal centered radical Rh^{II}(tmp) (**2**) was generated in about 80% yield by the photolytic cleavage of Rh(tmp)CH_3 (**1**) in benzene for ~8 h (Eq. (1)) [19] (see Fig. 1).

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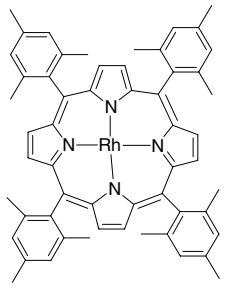


Rh(tmp)

Fig. 1. Structure of Rh(tmp).

$$\underset{\mathbf{1}}{\mathrm{Rh(tmp)CH_3}} \xrightarrow[8\,\mathrm{h},\ \mathrm{N_2}]{\mathrm{C_6H_6},\ h\nu,\ 6\text{–}10^{\circ}\mathrm{C}} \underset{\mathbf{2}}{\mathrm{Rh(tmp)}} \qquad (1)$$

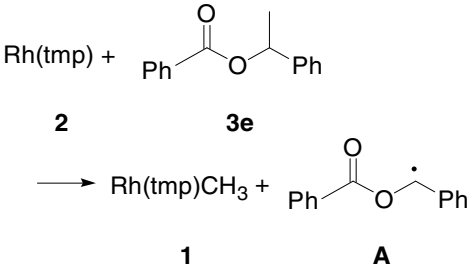
2.1. CCA results of Rh(tmp) and esters

$$\mathrm{Rh(tmp)} + \mathrm{ester} \xrightarrow[\mathrm{C_6H_6},\ \mathrm{N_2},\ 130\,^{\circ}\mathrm{C}]{\mathrm{PPh_3}\ (1\ \mathrm{equiv.})} \mathrm{Rh(tmp)CH_3} \qquad (2)$$

When Rh(tmp) reacted with ethyl benzoate (**3a**) in the presence of Ph_3P at 130 °C in 2 days, $Rh(tmp)CH_2CH_3$ (**4**) was observed in about 7% yield. No reaction occurred at 100 °C for 2 days.

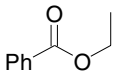
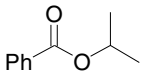
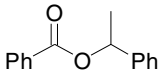
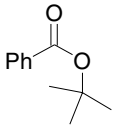
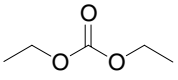
Two possible pathways exist for this formation of $Rh(tmp)CH_2CH_3$. First, the ethyl group cleavage via carbon–oxygen bond activation of $PhCO_2Et$ (**3a**) occurred. Second, CCA of $–OCH_2–CH_3$ occurred to give $Rh(tmp)CH_3$, $Rh(tmp)CH_3$ then reacted with the unreacted Rh(tmp) to give Rh(tmp)H and $Rh(tmp)CH_2$, which further underwent a bimolecular substitution [26,27] to give $Rh(tmp)CH_2CH_3$ and Rh(tmp) (Scheme 1). Therefore, the reaction conditions were optimized to be 130 °C for a shorter time of 1 day to prevent the formation of Rh(tmp)Et and other side reactions.

To our delight, when Rh(tmp) reacted with esters at 130 °C in the presence of 1 equiv. of triphenylphosphine in 1 day or less, carbon–carbon bond activation occurred with Rh(tmp)Me formed (Eq. (2)). Table 1 lists results of the CCA.



(3)

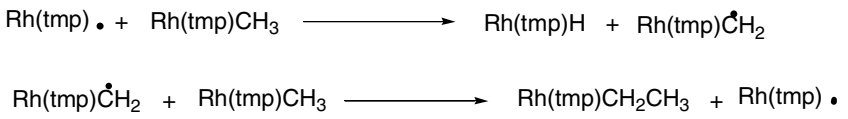
Table 1
Results of CCA at the alkoxy group (type **A**)

Entry	Esters	Reaction time (h)	$Rh(tmp)CH_3$ (**1**)/%[a]
1	 **3a**	24	29
2	 **3b**	24	30
3	 **3c**	6	38
4	 **3e**	24	27
5	 **3f**	24	23

[a] Yield (%) was based on 80% of Rh(tmp) generated through photolysis.

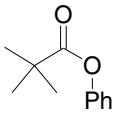
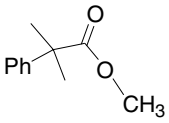
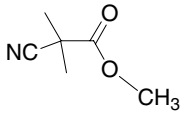
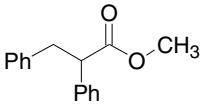
1-Benzoyloxy-1-phenylethane (**3c**) (entry 3) was the most reactive among the series of esters examined, and reacted with Rh(tmp) at 130 °C for 6 h to produce $Rh(tmp)CH_3$ in 38% yield. The electron withdrawing influence of phenyl ring weakened the methyl–carbon bond energy ($BDE_{PhCH–CH_3} = 76.4$ kcal mol^{-1}) [28], and facilitated the CCA. As a radical mechanism has been proposed of the CCA of ketones with Rh(tmp) [26,27], the resultant radical **A** would also be further resonance-stabilized (Eq. (3)) [29]. In this case, the steric hindrance of the phenyl group did not slow down the reactivity (Table 1, entry 3 vs. 1 and 2).

Ethyl benzoate (**3a**) ($BDE_{OCH_2–CH_3} = 90.2$ kcal mol^{-1}) [28], isopropyl benzoate (**3b**) ($BDE_{OCH–CH_3} = 88.2$ kcal mol^{-1}) [28], and *tert*-butyl benzoate (**3e**) gave similar product yields, further demonstrating the steric hindrance



Scheme 1. Proposed mechanism for forming $Rh(tmp)CH_2CH_3$.

Table 2
Results of CCA α- to the carbonyl group (type **B**)

Entry	Esters	Reaction time (h)	Rh(tmp)CH_3 (**1**)/%[a]
1	 **3g**	24	33
2	 **3h**	24	25
3	 **3i**	24	34
4	 **3j**	48	No reaction

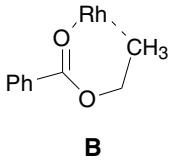
[a] Yield (%) was based on 80% of Rh(tmp) generated through photolysis.

did not play a significant role. Comparing with isopropyl benzoate (**3b**), 1-benzoyloxy-1-phenylethane (**3c**) gave higher ~8% yield of CCA product. It also supported that electron withdrawing group closer to the activation position increased the reactivity of substrates. For the case of diethyl carbonate (**3f**), about 23% yield of Rh(tmp)CH_3 was produced.

The CCA at the α,β position to the carbonyl group of esters was also successful. Phenyl-2,2-dimethyl-propionoate (**3g**) (Table 2, entry 1) was slightly more reactive than *tert*-butyl benzoate (**3e**) (Table 1, entry 4), which might be due to the carbonyl group closer to the activation position in **3g** ($BDE_{(CH_3)_3-CCOOCH_3} = 84.5$ kcal mol^{-1}) [28].

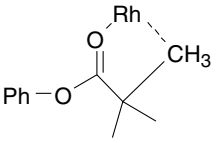
Substituent effect was also examined. Compound **3h** was less reactive (Table 2, entry 2) than **3g**. Presumably, it is due to the sterically more hindered phenyl group. Compound **3i** was more reactive as the nitrile group is more electron-withdrawing and less sterically hindered to result in a higher yield of CCA product. However, in the more bulky case of 2,3-diphenylmethyl propionate (**3j**) (Table 2, entry 4), no CCA or COA reaction occurred.

Type **B** substrates were more reactive than type **A** substrates. For both types **A** and **B** substrates, the pre-coordination of rhodium with carbonyl likely occurred. We rationalized that in the transition states of type **A** (Fig. 2)



B

Fig. 2. Six-membered ring transition state of CCA (type **A**).



C

Fig. 3. Five-membered ring transition state of CCA (type **B**).

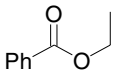
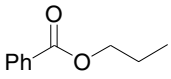
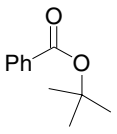
and type **B** (Fig. 3), six-membered and five-membered rings were formed, respectively. The more facile formation of five-membered ring transition state [30] may account the higher reactivity of type **B** substrates.

2.2. CCA of Rh(tmp) and esters under solvent-free conditions

$$\mathrm{Rh(tmp)} + \mathrm{ester} \xrightarrow[N_2,\ \text{temp.,}\ \text{time}]{\text{solvent-free,}\ PPh_3} \mathrm{product} \quad (4)$$

Solvent-free conditions were carried out for the CCA to increase the rate through high concentration of substrate. However, **3a** did not react at 70 °C for 5 days. At 100 °C, only about 10% Rh(tmp)CH_3 was isolated after 3 days.

Table 3
CCA results of esters under solvent-free conditions

Entry	Esters	Temperature (°C)	Time (days)	Product (yield/%[a])
1		70	5	No reaction
2	 **3a**	100	3	Rh(tmp)CH_3 (**1**) (10)
3	 **3d**	100	3	Rh(tmp)CH_2CH_3 (**4**) (trace)
4	 **3e**	130	1	Rh(tmp)CH_3 (**1**) (18)

[a] Yield (%) was based on 80% of Rh(tmp) generated through photolysis.

Table 4
CCA results of amides

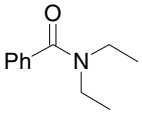
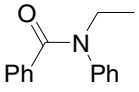
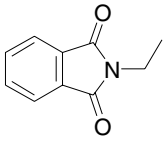
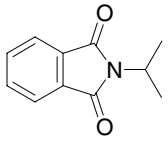
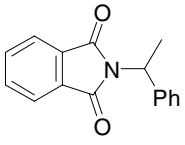
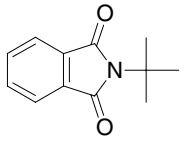
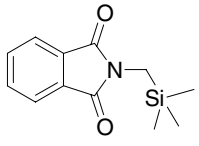
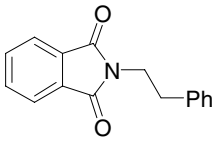
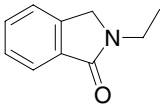
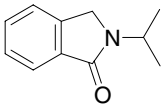
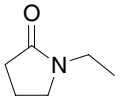
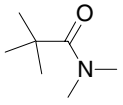
Entry	Amides	Temperature (°C)	Time (h)	Rh(tmp)CH_3 (**1**)/%[a]
1	 **5a**	130	24	33
2		130	6	41
3		100	6	37
4	 **5b**	100	6	29
5	 **5c**	130	24	40
6		130	6	37
7		100	6	39
8	 **5d**	100	6	33
9	 **5e**	100	6	39
10	 **5f**	130	6	43
11		100	6	40
12	 **5g**	100	24	No reaction

Table 4 (*continued*)

Entry	Amides	Temperature (°C)	Time (h)	Rh(tmp)CH_3 (**1**)/%[a]
13	 **5h**	100	48	No reaction
14	 **5i**	100	6	28
15	 **5j**	100	6	22
16	 **5k**	100	6	25
17	 **5l**	100	6	32

[a] Yield (%) was based on 80% of Rh(tmp) generated through photolysis.

Propyl benzoate (**3d**) reacted at 100 °C for 3 days to give trace amount of Rh(tmp)CH_2CH_3 via CCA at –OCH_2–CH_2CH_3 moiety. Ester **3e** reacted at 130 °C for 1 day to produce about 18% yield of Rh(tmp)CH_3. From Table 3, it suggested that increasing concentration of substrates did not result in the higher activity of CCA, likely due to change of solvent property [31].

2.3. Sealed tube experiments to confirm CCA of Rh(tmp) and esters

The reaction mixture of Rh(tmp), Ph_3P and **3a** was heated in anaerobic conditions at 130 °C for 30 h in an NMR tube. The extent of the reaction was monitored by ^{1}H NMR spectroscopy. The increasing intensity with time of the characteristic peaks (doublet, $^2J_{Rh-H} = 3.0$ Hz, δ: −5.25 ppm) of Rh–CH_3 confirmed the occurrence of carbon–carbon bond activation in the substrate.

2.4. CCA results of Rh(tmp) and amides

$$\mathrm{Rh(tmp)} + \text{amide} \xrightarrow[\mathrm{C_6H_6,\ N_2,\ temp.,\ time}]{\mathrm{PPh_3}} \mathrm{Rh(tmp)CH_3} \qquad (5)$$

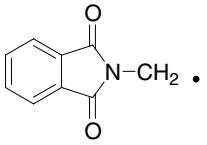
Table 4 shows the results of CCA of the more coordinating substrates, amides, with Rh(tmp). Carbon–carbon bond activation occurred when Rh(tmp) reacted with amides at 100–130 °C within 1 day to give $Rh(tmp)CH_3$.

The effect of reaction time was examined. When **5a** was heated at 130 °C and 100 °C for 6 h, similar yields of $Rh(tmp)CH_3$ were obtained in 41% and 37%, respectively. The same results were observed for **5c** and **5f**. Temperatures from 100 to 130 °C did not affect the yields of $Rh(tmp)CH_3$ (Table 4, entries 2, 3; 6, 7; 10, 11) and extent of reaction time giving similar results (Table 4, entries 1, 2; 5, 6). Even longer time of 24 h did not result in higher yields. Therefore, the CCA of Rh(tmp) and amides were carried out at 100 °C for 6 h.

For *N*-phenyl benzanilide (**5b**), the CCA yield was lower than that of **5a**. It might be due to the steric hindrance of phenyl ring. Comparing the cases of **5c**, **5d**, **5e**, and **5f**, similar reactivity was observed and $Rh(tmp)CH_3$ was formed in moderate yields. However, the more hindered trimethylsilyl substituted amide **5g** and benzyl substituted amide **5h** did not react with Rh(tmp). The amides with one carbonyl group such as **5i** and **5j**, gave lower CCA yields. Substrates **5k** and **5l** gave about 25% and 32% yield of CCA product, respectively.

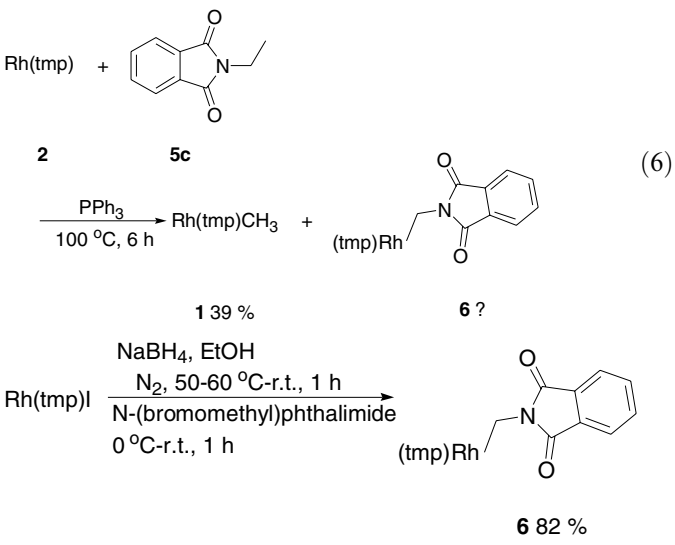
2.5. Identification of co-product

Since only $Rh(tmp)CH_3$ was isolated in low to moderate yields, other coproducts formed was therefore searched. For **5c**, compound **6** may also form (Eq. (6)). An authentic sample of compound **6** was obtained by reductive alkylation of Rh(tmp)I (Eq. (7)). However, analysis of the crude reaction mixture showed the absence of **6**. As**6** was thermally stable in C_6D_6 with 1 equiv. Ph_3P at 100 °C for 3 days, its decomposition was not possible. We have earlier proposed a bimolecular substitution mechanism for the CCA reaction, so it is likely that radical **D**, once formed, underwent more efficient hydrogen abstraction rather than coupled [32] with Rh(tmp) (see Fig. 4).



D

Fig. 4. Proposed intermediate for **5c**.



(6)

(7)

2.6. Sealed tube experiment to confirm CCA of Rh(tmp) and amides

The mixture of a solution of Rh(tmp), 1 equiv. of Ph_3P and 10 equiv. of substrate **5f** in C_6D_6 was placed in the NMR tube, degassed and sealed under vacuo and heated at 100 °C for 10 h. The increasing intensity with time of the characteristic peaks (doublet, $^2J_{Rh-H} = 3.0$ Hz, δ: −5.25 ppm) of Rh–CH_3 confirmed the formation of Rh(tmp)Me as the CCA product.

2.7. Comparison of CCA of esters and amides

The pre-coordination of carbonyl groups with rhodium–metal center was proposed in the esters and amides CCA. The higher reactivities of amides than those of esters are likely due to the more electron donating ability of nitrogen in amides than oxygen in esters [33].

3. Summary

In conclusion, esters and amides underwent carbon–carbon bond activation by Rh(tmp) successfully to give Rh(tmp) alkyls in moderate yields.

4. Experimental

All materials were obtained from commercial suppliers and used without further purification unless otherwise specified. Benzene was distilled from sodium. Benzene-d_6 was vacuum distilled from sodium, degassed thrice by freeze–thaw–pump cycle and stored in a Teflon scrawhead stoppered flask. Pyridine was distilled over KOH under N_2. Triphenylphosphine was recrystallized from EtOH. Thin layer chromatography was performed on Merck pre-coated silica gel 60 F_{254} plates. Silica gel (Merck, 70–230 and 230–400 mesh) or neutral aluminum oxide

(Merck, activity I, 70–230 mesh) was used for column chromatography.

^{1}H NMR spectra were recorded on a Brüker DPX 300 (300 MHz) spectrometer. Spectra were referenced internally to the residual proton resonance in $CDCl_3$ (δ 7.26 ppm), tetramethylsilane (TMS, δ 0.00 ppm) or with C_6D_6 (δ 7.15 ppm) as the internal standard. Chemical shifts (δ) were reported as part per million (ppm) in δ scale downfield from TMS or $(Me_3Si)_4Si$.

4.1. Preparation of 5,10,15,20-tetramesitylporphyrinatorhodium(II) [Rh(tmp)] (2) [34]

To a Teflon screwheaded stoppered flask, Rh(tmp)CH_3 (**1**) (10.0 mg, 0.011 mmol) was charged and dissolved in C_6H_6 (4.0 mL) to obtain a clear orange solution. The reaction mixture was then degassed by the freeze–pump–thaw method (three cycles) and refilled with N_2. The reaction mixture was irradiated under a 400 W Hg-lamp at 6–10 °C until all the starting material was consumed as indicated by TLC analysis (~8 h) to give Rh(tmp) (**2**) in 80% yield.

4.2. Reaction of [Rh(tmp)] (2) and esters 3a–3j with PPh_3 added

Triphenylphosphine solution (0.1 mL, 0.01 mmol, 0.1 M in C_6H_6, 1 equiv.) was added to the solution of [Rh(tmp)] (**2**) at r.t. Degassed ester solution (5 equiv.) in benzene was added to the adduct solution, and the mixture was heated under N_2 in the absence of light. The crude product was purified by chromatography on silica gel to give the product.

4.3. Preparation of (5,10,15,20-tetramesitylporphyrinato)rhodium(III) ethyl [Rh(tmp)CH_2CH_3] (4) [34]

Red suspension of Rh(tmp)I (200 mg, 0.15 mmol) in EtOH (100 mL) and the solution of $NaBH_4$ (28 mg, 0.74 mmol) in 0.5 M NaOH (8 mL) wre purged with N_2 separately for about 15 min. The $NaBH_4$ solution was added to the suspension of Rh(tmp)I via cannular. The reaction mixture was heated at 55 °C for 1 h under N_2. After cooled to r.t., EtI was added via syringe. A bright red suspension formed immediately and it was stirred overnight at room temperature. The reaction was worked up by addition of CH_2Cl_2 and H_2O. The crude product was extracted with CH_2Cl_2 (200 mL), washed with H_2O (25 mL × 3), dried over anhydrous $MgSO_4$, filtered and rotary evaporated off to dryness. After purification by column chromatography on silica gel using hexane:CH_2Cl_2 (10:1) to hexane:CH_2Cl_2 (5:1) as the gradient eluent, bright red solid (**4**) (113 mg, 0.12 mmol, 83%) was obtained which was further purified by recrystallization from CH_2Cl_2/hexane. $R_f = 0.67$ (hexane:CH_2Cl_2 = 5:1); ^{1}H NMR (300 MHz, $CDCl_3$) δ −4.74 (dq, 2H, $J = 7.5$ Hz, $J_{Rh-CH_2} = 3.6$ Hz), −4.22 (t, 3H, $J = 5.7$ Hz), 1.93 (s, 12H), 1.90 (s, 12H), 2.61 (s, 12H), 7.25 (s, 4H), 8.45 (s, 8H).

4.4. Sealed tube experiments

Triphenylphosphine solution (0.01 mL, 0.001 mmol, 0.1 M in C_6D_6, 1 equiv.) was added to the solution of [Rh(tmp)] (**2**) in the NMR tube at r.t. Degassed ester **3a** solution (10 equiv.) in C_6D_6 was added to the adduct solution, then the NMR tube was sealed under vacuum. The mixture was heated at 130 °C for 30 h under N_2 in the absence of light. The reaction was monitored with ^{1}H NMR spectroscopy.

4.5. Reaction of [Rh(tmp)] (2) and amides 5a–5l with PPh_3 added

Triphenylphosphine solution (0.1 mL, 0.01 mmol, 0.1 M in C_6H_6, 1 equiv.) was added to the solution of [Rh(tmp)] (**2**) at r.t. Degassed amide solution (5 equiv.) in benzene was added to the adduct solution, and the mixture was heated under N_2 in the absence of light. The crude product was purified by chromatography on silica gel to give the product.

4.6. Preparation of (5,10,15,20-tetramesitylporphyrinato)(N-phthalimido)methylrhodium(III) [Rh(tmp)CH_2N-Pht] (6) [34]

To a solution of Rh(tmp)I (50 mg, 0.049 mmol) in EtOH (30 mL), a solution of $NaBH_4$ (9.5 mg, 0.25 mmol) in aqueous NaOH (0.1 M, 2 mL) was added under N_2. The solution mixture was heated at 50–60 °C under N_2 for 1 h and then cooled to r.t. *N*-(bromomethyl)phthalimide (157 mg, 0.49 mmol) was added under N_2. The mixture was stirred at r.t. for 15 min. A reddish orange suspension was formed. The reaction was worked up by addition of CH_2Cl_2 and H_2O. the crude product was extracted with CH_2Cl_2 (200 mL), washed with H_2O (25 mL × 3), dried over anhydrous $MgSO_4$, filtered and rotary evaporated off to dryness. After purification by column chromatography on silica gel using hexane:CH_2Cl_2 (5:1) to hexane:CH_2Cl_2 (2:1) as the gradient eluent, a red solid of **6** was obtained (42.0 mg, 0.040 mmol, 82%). $R_f = 0.51$ (hexane:CH_2Cl_2 = 1:1). ^{1}H NMR (300 MHz, C_6D_6) δ −2.19 (d, 2H, $J_{Rh-CH_2} = 3.6$ Hz), 1.88 (s, 12H), 2.25 (s, 12H), 2.45 (s, 12H), 6.42 (d, 2H, $J = 8.4$ Hz), 6.55 (dd, 2H, $J = 3.0$ Hz, $J = 5.4$ Hz), 6.90 (s, 2H), 7.22 (s, 4H), 7.42 (s, 2H), 8.80 (s, 8H). ^{13}C NMR ($CDCl_3$, 100 MHz) 21.98, 22.12, 22.66, 120.61, 122.54, 128.41, 131.42, 131.56, 133.37, 138.08, 139.15, 139.48, 140.19, 143.64, 163.43. Anal. Calc. for $C_{65}H_{58}N_5O_2Rh$: C, 74.77; H, 5.60; N, 6.70. Found: C, 74.40; H, 5.99; N, 6.43%. HRMS (FAB): Calc. for $(C_{65}H_{58}N_5O_2Rh)^+$: *m*/*z* 1043.3640. Found: *m*/*z* 1043.36097.

4.7. Sealed tube experiments

Triphenylphosphine solution (0.01 mL, 0.001 mmol, 0.1 M in C_6D_6, 1 equiv.) was added to the solution of

[Rh(tmp)] (**2**) in the NMR tube at r.t. Degassed amide **5f** solution (10 equiv.) in C_6D_6 was added to the adduct solution, then the NMR tube was sealed under vacuum. The mixture was heated at 100 °C for 10 h under N_2 in the absence of light. The reaction was monitored with ^1H NMR spectroscopy.

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