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Purine-based carbenes at rhodium and iridium ☆

Jan Schütz, Wolfgang A. Herrmann *

Anorganisch-Chemisches Institut der Technischen Universität München, Lichtenbergstraße 4, D-85747 Garching, Germany

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Abstract

Purine-based carbenes can be attached to catalysis-related metals like rhodium and iridium through the standard method of in situ deprotonation of the respective azolium salts. Thus, 1,3,7,9-tetramethylxanthinium tetrafluoroborate is obtained by the reaction of trimethyloxonium tetrafluoroborate and caffeine. The salt and 7,9-dimethylhypoxanthinium iodide were used as a consecutive precursor to form rhodium (I) and iridium (I) carbene complexes of the type $[M(L)(L_{Carbene})_2]I$ and $M(L)(L_{Carbene})(I)$ (M = Rh, Ir, $L_{Carbene}$ = 1,3,7,9-tetramethylxanthine-8-ylidene, 7,9-dimethylhypoxanthine-8-ylidene, L = η^4-1,5-COD, CO) (COD = 1,5-cyclooctadiene). All compounds were characterized by ^{1}H NMR, ^{13}C NMR, mass spectrometry and/or elemental analysis.

Keywords: *N*-heterocyclic carbenes; Rhodium/iridium complexes; Caffeine; Xanthine

1. Introduction

In 1968 Wanzlick and Schönherr [1], and Öfele [2] discovered that *N*-heterocyclic carbenes (NHCs) derived from imidazolium and pyrazolium salts form remarkably stable transition metal complexes. The renaissance of this chemistry began after the isolation of the related free carbenes by Arduengo et al. [3]. There are many ways to deprotonate azolium salts to obtain free NHCs or NHC metal complexes [4]. This resulted in a search for NHCs beyond the common imidazol- and imidazolin-2-ylidenes. Examples are triazol-5-ylidenes [5], acyclic carbenes [6] and, recently, a pyrimidine-based carbene [7]. Our group concentrated on the possibility to generate NHC transition metal complexes from derivates of purine bases. A very recent report by Youngs and coworkers [8] describing silver and rhodium complexes of 1,3,7,9-tetramethylxanthine-8-ylidene prompts us to publish our own results in this case.

Our first aim was the investigation of methyl-caffeine (1,3,7,9-tetramethylxanthine). There are complexes reported in literature in which caffeine is linked to transition metals through the N-9 atom [9–11]. Ru(II), Ru(III), and Os (III) complexes in which caffeine is bound at C-8 are also reported [9,10,12]. The first 1,3,7,9-tetramethylxanthine-8-ylidene complex was reported in 1975 as a mercury bis-carbene complex [13].

2. Results and discussion

The generation of the 1,3,7,9-tetramethylxanthinium salt was attempted by reaction of methyl iodide with caffeine in refluxing DMF [14]. However, even heating the mixture for 24 h in a closed vessel only resulted in a 1:1-mixture of the desired product and caffeine. Changing to the more reactive methylating reagent trimethyloxonium tetrafluoroborate and to a reaction time of only 1 h in

☆ *N*-heterocyclic carbenes, part 37. For part 36, see S.K. Schneider, K. Öfele, E. Herdtweck, W.A. Herrmann, J. Organomet. Chem. 689 (2004) 2441.

* Corresponding author. Tel.: +49-89-28913080; fax: +49-89-28913473.
E-mail address: lit@arthur.anorg.chemie.tu-muenchen.de (W.A. Herrmann).

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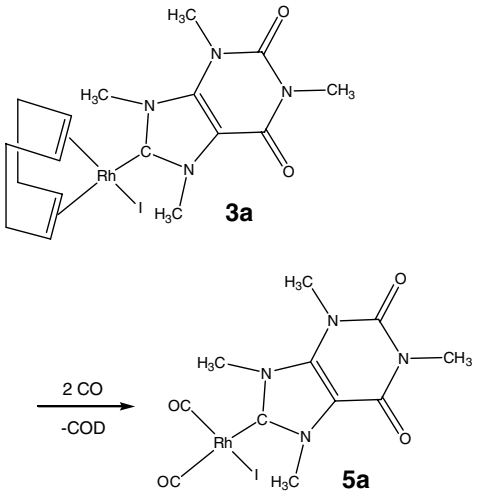
refluxing 1,2-dichloroethane produces 1,3,7,9-tetramethylxanthinium tetrafluoroborate (**2**) in high purity.

The synthesis of the new metal complexes follows established procedures [15] for the preparation of carbene complexes of Rh(I) and Ir(I) with imidazol-2-ylidene ligands from the alkoxide-bridged dimer [M(COD)-OEt]$_2$ **1a** (M = Rh) and **1b** (M = Ir), respectively, of Scheme 1.

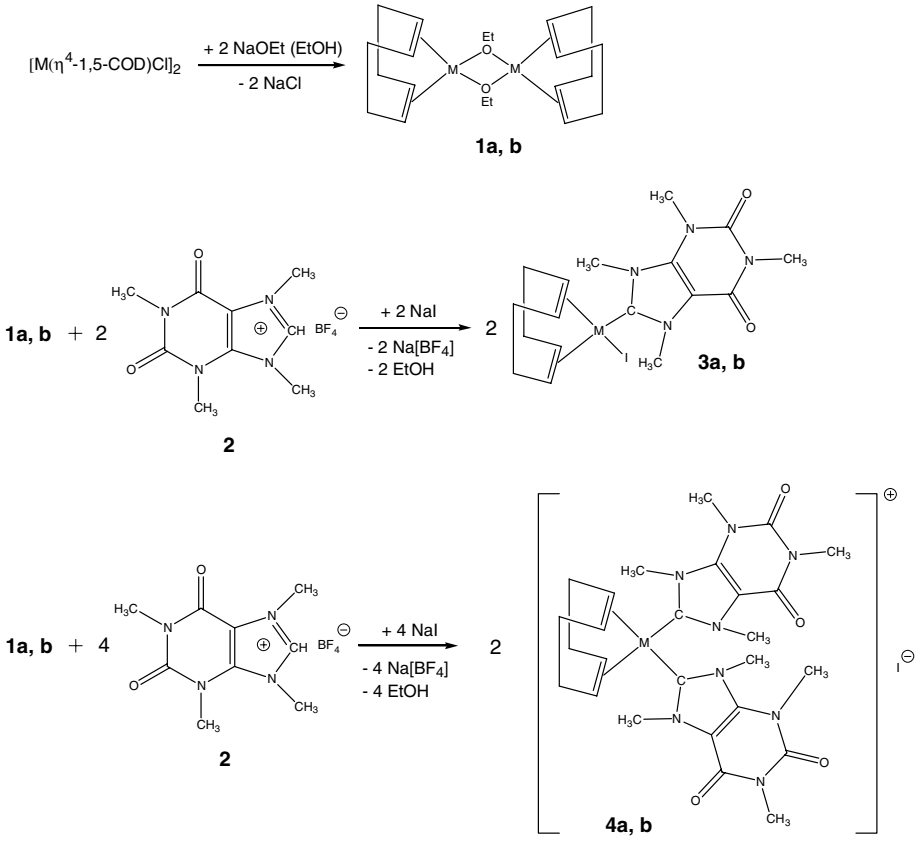
Substitution of the chloro bridge in the precursor dimer by an ethoxy bridge leads to complexes **1a**, **b**. Deprotonating the xanthinium salt **2** in situ with the internal base finally gives the desired complexes **3a**, **b** and **4a**, **b**. Reaction of **1a**, **b** with two equivalents of xanthinium salt results in the neutral complexes **3a**, **b**, whereas four equivalents of xanthinium salt and a longer reaction time leads to the cationic dicarbene complexes **4a**, **b**.

From **3a**, **b** or **4a**, **b** cyclooctadiene may be displaced by two molecules of carbon monoxide to yield, e.g., compound **5a** and **6a** with ν(CO) = 2080, 2009 (cm^{-1}) and ν(CO) = 1962 (cm^{-1}), respectively, indicating a *cis*-configuration for compound **5a** (Eq. (1)). The products **5a** and **6a** were formed within minutes as air stable pale yellow solids. Due to the strong donor capability of the carbene ligands, the cyclooctadiene ligand can be quantitatively substituted by the stronger acceptor ligand carbon monoxide [16].

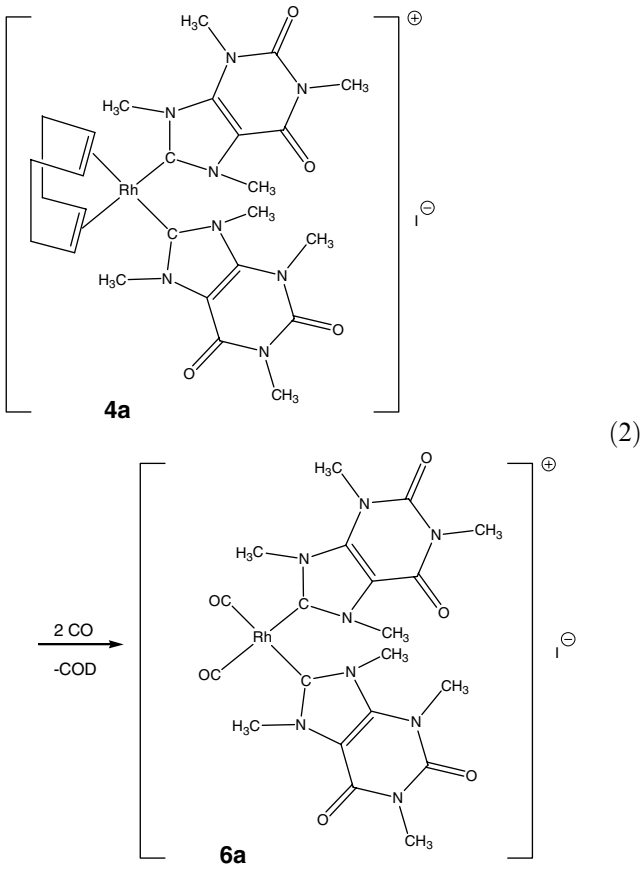
The CO stretching vibrations at ν_{CO} = 2080 and 2009 cm^{-1} for **5a** can be used to gauge the donor ability of the ligand and comparison of the values with other imidazol-2-ylidene-based complexes indicates that 1,3,7,9-tetramethylxanthine-8-ylidene is a weaker electron donor than e.g., pyrimidine-based carbenes [7] or imidazol-2-ylidenes [16]. There appears not to be a comparable NHC ligand in literature that is a stronger electron acceptor than 1,3,7,9-tetramethylxanthine-8-ylidene.



(1)

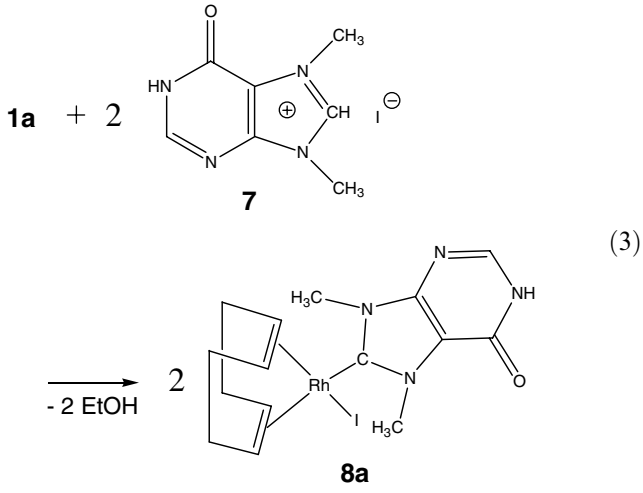


Scheme 1. Synthesis of **3a**, **b** and **4a**, **b**



(2)

Hypoxanthine is an another purine base that has an imidazole moiety. The transformation into an azolium cation is accomplished by the reaction of hypoxanthine with methyl iodide [17]. The resulting 7,9-dimethylxanthinium iodide **7** reacts in an analogous way to the 1,3,7,9-tetramethylxanthinium salt with $[Rh(COD)OEt]_2$ (Eq. (3))



(3)

For all complexes both the ^1H and ^{13}C NMR spectra prove the complexation of the transition metal atom with one (**3a**, **3b**, **5a**, **8a**) or two (**4a**, **4b**, **6a**) xanthine-8-ylidene moieties. In the ^1H NMR spectra the signals for C(8)–H disappear while the rest of the proton signals shift slightly downfield. Comparison of the ^{13}C resonances of the carbene carbon atoms of the corresponding rhodium and iridium complexes shows a downfield shift for the rhodium complexes: $\delta = 189.6$ (**3a**), 186.8 and 186.7 (**4a**) compared to 185.2 (**3b**), 181.1 and 181.0 (**4b**). There is also a downfield shift for the cationic dicarbene complexes **4a**, **b** compared to the neutral monocarbene complexes **3a**, **b**. The coupling constant increases for the rhodium compounds from $^1J_{C-Rh} = 52.2$ Hz (**3a**) to 55.1 Hz (**4a**) when two instead of one carbene coordinates to the metal centre. The coupling constants of the other rhodium complexes are also in the region to those previously reported for other NHC–rhodium complexes [8,16,18]: $^1J_{C-Rh} = 42.6$ Hz (**5a**), 53.4 Hz (**6a**) and 52.9 Hz (**8a**). The variance of the chemical shift or the coupling constant of the different ligands 1,3,7,9-tetramethylxanthine-8-ylidene and 7,9-dimethylhypoxanthine-8-ylidene is not significant: 189.6, $^1J_{C-Rh} = 52.2$ Hz (**3a**) and 188.1 Hz, $^1J_{C-Rh} = 52.9$ Hz (**8a**). This is probably due to the similar imidazol moiety of both ligands. The changes in the 6-membered ring do apparently not affect the properties of the carbene.

3. Conclusion

Purine bases appear to be excellent sources for carbene-type ligands, which act as relatively weak σ-donor ligands to metal centers like rhodium(I) and iridium(I). Catalytic applications are under investigation in our laboratories.

4. Experimental

4.1. General considerations

All manipulations were carried out using standard Schlenk techniques under an atmosphere of argon or nitrogen. Ethanol, diethyl ether and *n*-pentane were dried over Na and distilled prior to use applying conventional procedures.

The compounds trimethyloxonium tetrafluoroborate, 1,5-cyclooctadiene, $IrCl_3$ and $RhCl_3$ were purchased from Merck–Schuchardt, caffeine from Acros without further purification. 7,9-dimethylhypoxanthinium iodide [17], $[Rh(COD)Cl]_2$ [19] and $[Ir(COD)Cl]_2$ [20] and were prepared according to literature methods.

^1H and ^{13}C NMR spectra were recorded on a JEOL JNM-GX 400 spectrometer in $CDCl_3$ or d_6-DMSO and referenced to the residual ^1H and ^{13}C signals of the solvents. Elemental analysis were performed by the micro analytical laboratory in our institute. IR spectra were recorded on a FTS 575C BIO-RAD spectrometer.

Mass spectra were recorded on a Varian MAT 311a spectrometer using FAB ionization (xenon/*p*-nitrobenzylalcohol matrix).

4.2. Synthesis of 1,3,7,9-tetramethylxanthinium tetrafluoroborate **2**

Trimethyloxonium tetrafluoroborate (1.07 g, 7.21 mmol) was slowly added to a solution of caffeine (1.40 g, 7.22 mmol) in 1,2-dichloroethane (20 mL). The mixture was refluxed for 1 h at 100 °C. The remaining 1,2-dichloroethane was removed in vacuo to give a slightly yellow oil. Addition of diethyl ether and stirring for 14 h gave the product as a white solid (1.40 g, 66%). ^{1}H NMR (400 MHz, d_6-DMSO): δ = 9.27 (s, 1H, NCHN), 4.12 (s, 3H, CH_3), 4.04 (s, 3H, CH_3), 3.26 (s, 3H, CH_3), 2.49 (s, 3H, CH_3); ^{13}C NMR (100.5 MHz, d_6-DMSO): δ 154.2 (C(6)O), 150.7 (C(2)O), 140.3 (*C(4)*=C(5)), 139.8 (NCHN), 108.9 (C(4)=*C(5)*), 37.3, 36.2, 31.9, 28.9 (N*C* H_3); Elemental Anal. Calc. for $C_9H_{15}N_4O_2BF_4$ (296.11): C, 36.52; H, 4.43; N, 18.93. Found: C, 35.98; H, 4.88; N, 19.00%.

4.3. General procedure for the preparation of halo-monocarbene complexes

NaH (45 mg, 1.86 mmol) was dissolved in 8 mL ethanol and slowly added to a suspension of $[M(COD)Cl]_2$ (0.45 mmol) in 8 mL ethanol. After the mixture was stirred for 30 min at room temperature the xanthinium salt (290 mg, 0.98 mmol) and NaI (for **2a**, **b**) (160 mg, 1.07 mmol) were added. The suspension was stirred at room temperature for 24 h, filtrated at 0 °C and washed with 10 mL ethanol and 10 mL diethyl ether at 0 °C.

4.3.1. Synthesis of iodo(η^4-1,5-COD)(1,3,7,9-tetramethylxanthine-8-ylidene)rhodium(I) **3a**

The pure product was obtained as a yellow solid (432 mg, 88%). ^{1}H NMR (400 MHz, d_6-DMSO): δ = 4.89 (br, 2H, COD_{vinyl}), 4.45 (s, 3H, CH_3), 4.18 (s, 3H, CH_3), 3.69 (s, 3H, CH_3), 3.42 (m, 2H, COD_{vinyl}), 3.17 (s, 3H, CH_3), 2.34 (m, 4H, COD_{allyl}), 1.90 (m, 4H, COD_{allyl}); ^{13}C NMR (100.5 MHz, d_6-DMSO): δ 189.6 (d, carbene, $^1J_{Rh-C}$ = 52.2 Hz), 152.4 (C(6)O), 150.2 (C(2)O), 140.4 (*C(4)*=C(5)), 109.0 (C(4)=*C(5)*), 97.6 (d, COD_{vinyl}–Rh, $^1J_{Rh-C}$ = 7.2 Hz), 69.4 (d, COD_{vinyl}–Rh, $^1J_{Rh-C}$ = 12.1 Hz), 38.4, 36.6, 31.3, 28.0 (NCH_3), 32.4, 32.0, 28.7, 28.5 (COD_{allyl}); Elemental Anal. Calc. for $C_{17}H_{24}N_4O_2IRh$ (546.0): C, 37.38; H, 4.43; N, 10.26. Found: C, 37.78; H, 4.38; N, 10.55%; FAB-MS *m/z* 546 (M^+, 18%).

4.3.2. Synthesis of iodo(η^4-1,5-COD)(1,3,7,9-tetramethylxanthine-8-ylidene)iridium(I) **3b**

The pure product was obtained as a yellow solid (512 mg, 90%). ^{1}H NMR (400 MHz, d_6-DMSO): δ = 4.58 (m, 2H, COD_{vinyl}), 4.29 (s, 3H, CH_3), 4.05 (s, 3H, CH_3), 3.71 (s, 3H, CH_3), 3.43 (br, 2H, COD_{vinyl}), 3.21 (s, 3H, CH_3), 2.13 (m, 4H, COD_{allyl}), 1.73 (m, 4H, COD_{allyl}); ^{13}C NMR (100.5 MHz, d_6-DMSO): δ 185.2 (carbene), 152.6 (C(6)O), 150.2 (C(2)O), 140.7 (*C(4)*=C(5)), 108.9 (C(4)=*C(5)*), 83.0 (br, COD_{vinyl}), 53.0 (COD_{vinyl}), 37.9, 36.1, 31.3, 28.0 (NCH_3), 32.6, 32.3, 29.8, 29.4 (COD_{allyl}); Elemental Anal. Calc. for $C_{17}H_{24}N_4O_2IIr$ (636.1): C, 32.13; H, 3.81; N, 8.82. Found: C, 32.03; H, 3.73; N, 7.41%; FAB-MS *m/z* 636 (M^+, 39%).

4.3.3. Synthesis of iodo(η^4-1,5-COD)(7,9-dimethylhypoxanthine-8-ylidene)rhodium(I) **8a**

The pure product was obtained as a yellow solid (402 mg, 89%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.19 (s, 1H, NCHN), 4.43 (s, 3H, CH_3), 4.27 (s, 3H, CH_3); ^{13}C NMR (100.5 MHz, $CDCl_3$): δ 188.1 (d, carbene, $^1J_{Rh-C}$ = 52.9 Hz), 159.2 (NCHN), 153.4 (CO), 149.3 (*C(4)*=C(5)), 116.3 (C(4)=*C(5)*), 98.0 (d, COD_{vinyl}–Rh, $^1J_{Rh-C}$ = 7.3 Hz), 70.1 (d, COD_{vinyl}–Rh, $^1J_{Rh-C}$ = 15.0 Hz), 36.8, 33.6, 33.4, 29.6, 30.0, 25.6 (NCH_3, COD_{allyl}); Elemental Anal. Calc. for $C_{15}H_{20}N_4OIRh$ (502.0): C, 35.88; H, 4.01; N, 11.16. Found: C, 35.91; H, 4.05; N, 11.38%; FAB-MS *m/z* 502 (M^+, 9%).

4.4. General procedure for the preparation of bis-carbene halide complexes

NaH (160 mg, 6.67 mmol) was dissolved in 10 mL ethanol and slowly added to a suspension of $[M(COD)Cl]_2$ (0.71 mmol) in 8 mL ethanol. After the mixture was stirred for 45 min at room temperature 1,3,7,9-tetramethylxanthinium tetrafluoroborate (1680 mg, 5.68 mmol) and NaI (870 mg, 5.80 mmol) were added. The suspension was stirred at room temperature for 48 h, filtrated at 0 °C and washed with 10 mL ethanol and 10 mL diethyl ether at 0 °C.

4.4.1. Synthesis of [(η^4-1,5-COD)bis(1,3,7,9-tetramethylxanthine-8-ylidene)rhodium(I)]iodide **4a**

The pure product was obtained as a yellow solid (771 mg, 72%). ^{1}H NMR (400 MHz, d_6-DMSO): δ = 4.54 (br, 2H, COD_{vinyl}), 4.47 (br, 2H, COD_{vinyl}), 4.19 (s, 3H, CH_3), 3.70 (s, 3H, CH_3), 3.33 (s, 3H, CH_3), 3.17 (s, 3H, CH_3), 2.48 (br, 4H, COD_{allyl}), 2.18 (br, 4H, COD_{allyl}); ^{13}C NMR (100.5 MHz, d_6-DMSO): δ 186.8 (d, carbene, $^1J_{Rh-C}$ = 55.1 Hz), 186.7 (d, carbene, $^1J_{Rh-C}$ = 55.1 Hz), 152.3, 152.3 (C(6)O), 150.1, 150.0 (C(2)O), 140.8, 140.8 (*C(4)*=C(5)), 109.6, 109.5 (C(4)=*C(5)*), 91.4 (d, COD_{vinyl}–Rh, $^1J_{Rh-C}$ = 7.4 Hz), 91.1 (d, COD_{vinyl}–Rh, $^1J_{Rh-C}$ = 7.4 Hz), 90.4 (d, COD_{vinyl}–Rh, $^1J_{Rh-C}$ = 7.4 Hz), 90.1 (d, COD_{vinyl}–Rh, $^1J_{Rh-C}$ = 7.4 Hz), 37.9, 37.6, 30.3, 29.9 (NCH_3), 31.4, 31.3, 30.1, 30.0 (COD_{allyl}); Elemental Anal. Calc.

for $C_{26}H_{36}N_8O_4IRh$ (754.1): C, 41.39; H, 4.81; N, 14.85. Found: C, 42.01; H, 4.85; N, 14.51%; FAB-MS *m/z* 627 (M^+, 13%).

4.4.2. Synthesis of [(η⁴-1,5-COD)bis(1,3,7,9-tetramethylxanthine-8-ylidene)iridium(I)]iodide ***4b***

The general procedure for bis-carbene halide complexes was followed. The pure product was obtained by removing the solvent in vacuo and washing with 3×10 mL diethyl ether at −40 °C to give a orange solid (730 mg, 61%). ^{1}H NMR (400 MHz, d_6-DMSO): δ = 4.42 (br, 2H, COD_{vinyl}), 4.13 (s, 3H, CH_3), 4.01 (s, 3H, CH_3), 3.71 (s, 3H, CH_3), 3.31 (br, 2H, COD_{vinyl}), 3.19 (s, 3H, CH_3), 2.31 (m, 4H, COD_{allyl}), 1.97 (m, 4H, COD_{allyl}); ^{13}C NMR (100.5 MHz, d_6-DMSO): δ 181.1, 181.0 (carbene), 152.9 (C(6)O), 150.4 (C(2)O), 141.0, 140.9 (*C(4)*═C(5)), 109.3, 109.2 (C(4)═*C(5)*), 78.7, 78.3, 77.6, 77.2 (COD_{vinyl}), 39.1, 37.4, 37.2, 31.5, 31.3, 28.0 (NCH_3), 30.7, 30.6, 30.6, 30.5 (COD_{allyl}); FAB-MS *m/z* 716 (M^+, 21%).

4.5. General procedure for the preparation of carbonyl carbene complexes

Carbon monoxide was passed into a solution of the corresponding (η⁴-1,5-COD) (1,3,7,9-tetramethylxanthin-8-ylidene)rhodium(I) complex (0.22 mmol) in 20 mL methylenchloride for 1 h at room temperature. After the solution changed color from bright yellow to pale yellow the solvent was removed and the residue washed with 15 mL *n*-pentane to give a pale yellow solid (101 mg, 93%).

4.5.1. Synthesis of iodo dicarbonyl(1,3,7,9-tetramethylxanthine-8-ylidene)rhodium(I) ***5a***

Yield: 101 mg (93%). ^{1}H NMR (400 MHz, d_6-DMSO): δ = 4.67 (s, 3H, CH_3), 4.14 (s, 3H, CH_3), 3.73 (s, 3H, CH_3), 3.21 (s, 3H, CH_3); ^{13}C NMR (100.5 MHz, d_6-DMSO): δ 189.0 (d, CO, $^1J_{Rh-C}$ = 42.6 Hz), 180.3 (d, CO, $^1J_{Rh-C}$ = 70.8 Hz), 178.2 (d, carbene, $^1J_{Rh-C}$ = 50.7 Hz), 152.8 (N*C(6)* OC(5)), 150.4 (N*C(2)* ON), 140.7 (C*(4)* = C(5)), 109.8 (C(4)═*C(5)*), 38.8, 37.1, 31.5, 27.5 (NCH_3); Elemental Anal. Calc. for $C_{11}H_{12}N_4O_4IRh$ (493.9): C, 26.74; H, 2.45; N, 11.34. Found: C, 26.78; H, 2.37; N, 10.99%; IR (CH_2Cl_2): ν (cm^{-1}) = 2080 (s), 2009 (s), 1715 (s), 1677 (s).

4.5.2. Synthesis of [dicarbonyl bis(1,3,7,9-tetramethylxanthine-8-ylidene)rhodium(I)]iodide ***6a***

Yield: 148 mg (96%). ^{1}H NMR (400 MHz, d_6-DMSO): δ = 4.30 (s, 3H, CH_3), 3.61 (s, 3H, CH_3), 3.39 (s, 3H, CH_3), 3.08 (s, 3H, CH_3); ^{13}C NMR (100.5 MHz, d_6-DMSO): δ 188.2 (d, CO, $^1J_{Rh-C}$ = 44.6 Hz), 181.8 (d, carbene, $^1J_{Rh-C}$ = 53.4 Hz), 152.8 (N*C(6)* OC(5)), 150.4 (N*C(2)* ON), 140.5 (*C(4)*═C(5)), 108.9 (C(4)═*C(5)*), 38.7, 36.9, 31.5, 28.1 (NCH_3); FAB-MS *m/z* 547 ($M^+ - CO$, 11%); IR (CH_2Cl_2): ν (cm^{-1}) = 1962 (s), 1710 (s), 1673 (s).

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