

# ISOLATION OF $[\text{Rh}(\text{diolefin})\text{X}_2]^-$ SPECIES AND THEIR REACTIONS WITH P- OR N-DONOR LIGANDS, TIN(II) HALIDES AND CARBON MONOXIDE

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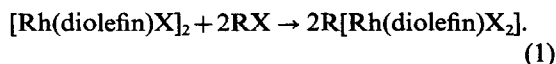
**Abstract**—Anionic  $[\text{Rh}(\text{diolefin})\text{X}_2]^-$  species ( $\text{X} = \text{Cl}, \text{Br}$ ) have been prepared and their reactions studied. The reactions with monodentate ligands led to neutral tetracoordinated complexes, and with N-donor bidentate ligands ( $\text{Rh} : \text{LL} = 2 : 1$ ) gave  $\text{Rh}(\text{X})(\text{diolefin})(\text{LL})$ ,  $[\text{Rh}(\text{diolefin})(\text{LL})]^+[\text{Rh}(\text{diolefin})\text{X}_2]^-$ , or  $[\text{Rh}(\text{diolefin})(\text{LL})]\text{X}$  compounds, depending on the nature of LL or X. Reactions with carbon monoxide involved diolefin displacement. A trichlorostannato complex was obtained from the  $[\text{Rh}(\text{COD})\text{Cl}_2]^-$  species. Reactions of  $[\text{Rh}(\text{COD})\text{Br}]_2$  with bidentate N-donor ligands were also studied.

The reaction between  $[\text{Rh}(\text{diolefin})\text{Cl}]_2$  and some bidentate N-donor ligands ( $\text{Rh} : \text{LL} = 2 : 1$ ) may give, depending on the nature of LL, ion-pair  $[\text{Rh}(\text{diolefin})(\text{LL})]^+[\text{Rh}(\text{diolefin})\text{Cl}_2]^-$  complexes, that contain diolefinic rhodium(I) anions.<sup>1-4</sup> While anionic carbonylated rhodium(I) species  $[\text{Rh}(\text{CO})_2\text{X}_2]^-$  have been extensively studied<sup>5-12</sup> and the crystal structure of  $[\text{Rh}(\text{CO})_2\text{Cl}_2]^-$  has been determined,<sup>6-8</sup> similar diolefinic  $[\text{Rh}(\text{diolefin})\text{X}_2]^-$  species have received less attention.  $\text{AsPh}_4[\text{Rh}(\text{COD})\text{Cl}_2]$  has been isolated<sup>13</sup> and the molecular structure of an ionic complex containing the  $[\text{Rh}(\text{COD})\text{Cl}_2]^-$  anion has been reported.<sup>14</sup>

In the present paper, we describe the synthesis of different  $[\text{Rh}(\text{diolefin})\text{X}_2]^-$  species and their reactivity with mono- and bidentate ligands containing N- or P-donor atoms, with carbon monoxide and with tin(II) halides.

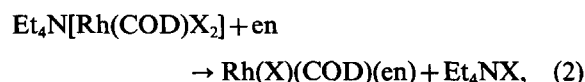
## RESULTS AND DISCUSSION

$[\text{Rh}(\text{diolefin})\text{X}_2]^-$  species may be easily prepared by the reaction of  $[\text{Rh}(\text{diolefin})\text{X}]_2$  dimers with a stoichiometric amount of the corresponding halide as the salt of bulky cations that favour precipitation [eq. (1)]



The complexes (I–III) thus obtained (see Table 1) are yellow and air stable, behave as 1 : 1 electrolytes in acetone solution and their IR spectra show, along with the bands due to the corresponding cations and diolefins, one or two bands in the far-IR due to  $\nu(\text{Rh}-\text{X})$ . The <sup>1</sup>H-NMR spectra of the two anions containing 1,5-cyclooctadiene ( $\text{X} = \text{Cl}, \text{Br}$ ) show three groups of resonances, around 4.3, 2.4 and 1.7 ppm. These values agree with those found in the literature for the anion  $[\text{Rh}(\text{COD})\text{Cl}_2]^-$  isolated as the tetraphenylarsonium salt by a more complicated method.<sup>13</sup> The anion  $[\text{Rh}(\text{NBD})\text{Cl}_2]^-$  shows the corresponding signals around 3.8, 3.6 and 1.1 ppm. All these signals are very similar to those corresponding to the starting dimers  $[\text{Rh}(\text{diolefin})\text{X}]_2$ .<sup>15</sup>

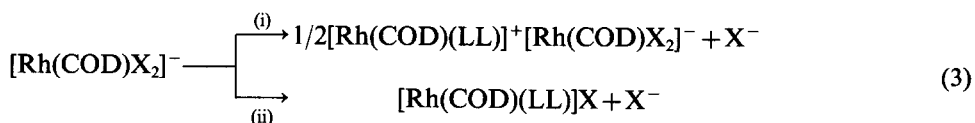
The reactivity of  $[\text{Rh}(\text{COD})\text{X}_2]^-$  with aliphatic diamines (LL) depends on the degree of N-substitution in the diamine. Thus, when LL = ethylenediamine (en), pentacoordinated neutral  $\text{Rh}(\text{X})(\text{COD})(\text{en})$  compounds are obtained, irrespective of the stoichiometric ratios employed ( $\text{Rh} : \text{LL} = 2 : 1$  or  $1 : 1$ ) [eq. (2)]



but when the more crowded LL = N,N,N',N'-tetramethylethylenediamine (tmen) is used, no reaction occurs and the starting products are recovered unchanged.

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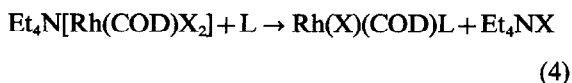
The anions  $[\text{Rh}(\text{COD})\text{X}_2]^-$  react with bidentate heterocyclic N-donor ligands to give products that depend on the type of halogen, X. Thus,  $\text{Et}_4\text{N}[\text{Rh}(\text{COD})\text{Cl}_2]$  reacts with 2,2'-bipyridine (bipy), 1,10-phenanthroline (phen), 5,6-dimethyl-1,10-phenanthroline (5,6-Me<sub>2</sub>phen) or 4,7-diphenyl-1,10-phenanthroline (4,7-Ph<sub>2</sub>phen) to yield ion-pair compounds  $[\text{Rh}(\text{COD})(\text{LL})]^+[\text{Rh}(\text{COD})\text{Cl}_2]^-$  when Rh:LL = 2:1 or ionic tetracoordinated  $[\text{Rh}(\text{COD})(\text{LL})]\text{Cl}$  when Rh:LL = 1:1. These products may also be obtained by the reaction of  $[\text{Rh}(\text{COD})\text{Cl}]_2$  with these ligands.<sup>3,16</sup> On the other hand, the reaction of  $\text{Et}_4\text{N}[\text{Rh}(\text{COD})\text{Br}_2]$  with these ligands gave only tetracoordinated ionic  $[\text{Rh}(\text{COD})(\text{LL})]\text{Br}$  complexes, irrespective of the stoichiometric ratios employed (Rh:LL = 2:1 or 1:1). The elemental analyses, molar conductivities and yields for the complexes obtained (IV–VII) are collected in Table 1.



(i) = 1/2LL (X = Cl); (ii) = LL (X = Cl, Br).

These results indicate a lower tendency to form the ion-pair compounds, when the anionic species contain bromine.

Monodentate P- or N-donor ligands, such as quinoline (quin) or triphenylphosphine, react easily with  $[\text{Rh}(\text{diolefin})\text{X}_2]^-$  species (Rh:L = 1:1), and displace one halide atom, yielding neutral tetra-coordinated compounds [eq. (4)]

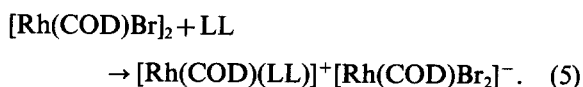


L = quin, Ph<sub>3</sub>P.

Carbon monoxide displaces the coordinated diolefin, leading to the well-known dicarbonylated anions  $[\text{Rh}(\text{CO})_2\text{Cl}_2]^{-5}$  identified through the IR spectra of the solutions obtained.

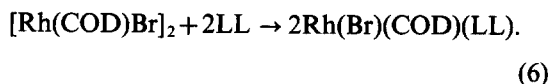
$\text{Et}_4\text{N}[\text{Rh}(\text{COD})\text{Cl}_2]$  reacts with  $\text{SnCl}_2$  but needs Rh:Sn = 1:3 molar ratios to react completely and give  $(\text{Et}_4\text{N})_2[\text{Rh}(\text{SnCl}_3)_3(\text{COD})]$ ,<sup>17</sup> while  $\text{Et}_4\text{N}[\text{Rh}(\text{COD})\text{Br}_2]$  does not react with  $\text{SnBr}_2$  under the same conditions and the starting product is recovered unchanged.

In the light of the above results, we have studied the reaction between  $[\text{Rh}(\text{COD})\text{Br}_2]$ , prepared *in situ* from  $[\text{Rh}(\text{COD})\text{Cl}]_2$ ,<sup>1</sup> and bidentate N-donor ligands (Rh:LL = 2:1). The reaction with LL = bipy, phen, 5,6-Me<sub>2</sub>phen, 4,7-Ph<sub>2</sub>phen or tmen, gave ion-pair compounds [eq. (5)]



The complexes obtained are collected in Table 1 (X–XVI), they behave as 1:1 electrolytes in acetone solution and their IR spectra show bands due to  $\nu$  (Rh—Br) of the anion  $[\text{Rh}(\text{COD})\text{Br}_2]^-$ . In keeping with previous suggestions<sup>18</sup> we could attribute the behaviour of  $[\text{Rh}(\text{COD})\text{Br}_2]$  to a bridge cleavage mechanism via five-coordinate intermediates that could avoid free bromine appearing in solution.

The reaction of  $[\text{Rh}(\text{COD})\text{Br}_2]$  with ethylenediamine, 2,9-dimethyl-1,10-phenanthroline (2,9-Me<sub>2</sub>phen) or 2,9-dimethyl-4,7-diphenyl-1,10-phenanthroline (2,9-Me<sub>2</sub>-4,7-Ph<sub>2</sub>phen) (Rh:LL = 1:1 or 2:1) gave only pentacoordinated neutral compounds  $\text{Rh}(\text{Br})(\text{COD})(\text{LL})$  (XV–XVII), analogous to those obtained from  $[\text{Rh}(\text{COD})\text{Cl}]_2$ <sup>3,4</sup> [eq. (6)]



These compounds behave as non-electrolytes in acetone solution and their IR spectra show one band around 200 cm<sup>-1</sup> that may be due to  $\nu$  (Rh—Br), thus confirming the bromine being bonded to the rhodium atom.  $\text{Rh}(\text{Br})(\text{COD})(\text{en})$  shows bands due to the coordinated diamine at  $\nu$  (N—H) = 3190 ( $\Delta\nu$  = 180), 3170 ( $\Delta\nu$  = 200) and 3080 ( $\Delta\nu$  = 205) cm<sup>-1</sup>. The band at the higher frequency is split, probably due to crystal lattice effects. The  $\nu$  (N—H) decreases upon coordination are smaller than in the analogous  $\text{Rh}(\text{Cl})(\text{COD})(\text{en})$ .<sup>4</sup>

## EXPERIMENTAL

$[\text{Rh}(\text{COD})\text{Cl}]_2$ , *in situ*  $[\text{Rh}(\text{COD})\text{Br}_2]$ <sup>1</sup> and  $[\text{Rh}(\text{NBD})\text{Cl}]_2$ <sup>19</sup> compounds were prepared by published methods. The C, H and N microanalysis were carried out with a Perkin–Elmer 240C microanalyser. Conductivities were measured in *ca*  $2.5 \times 10^{-4}$  M acetone solutions with a Metrom E518 conductimeter. Proton NMR spectra were recorded at room temperature with an XL-200 Varian spectrometer with tetramethylsilane as the internal standard and deuteriochloroform as the solvent. IR spectra were recorded on a Perkin–Elmer 598 spectrophotometer in the range 4000–200 cm<sup>-1</sup> using Nujol mulls between polyethylene sheets and

Table 1. Analytical, conductivity and IR data and yields for the isolated compounds

| Compound  | Found (calc.)<br>%C %H %N |              |              | $\Lambda_{\text{M}}$<br>( $\Omega^{-1} \text{cm}^2$<br>$\text{mol}^{-1}$ ) | $\nu$ (Rh—X)<br>( $\text{cm}^{-1}$ ) | Yield<br>(%) |
|---|---------------------------|--------------|--------------|--|--------------------------------------|--------------|
| $\text{Et}_4\text{N}[\text{Rh}(\text{COD})\text{Cl}_2]$ (I)                                   | 45.3<br>(46.4)            | 7.4<br>(7.8) | 3.2<br>(3.4) | 106  | 270(sh)<br>258(s)                    | 61           |
| $\text{AsPh}_4[\text{Rh}(\text{NBD})\text{Cl}_2]$ (II)  | 56.2<br>(57.3)            | 4.3<br>(4.3) | —            | 112  | 275(m)<br>260(m)                     | 76           |
| $\text{Et}_4\text{N}[\text{Rh}(\text{COD})\text{Br}_2]$ (III)                                 | 38.4<br>(38.3)            | 7.1<br>(6.4) | 2.8<br>(2.8) | 136  | 188(s)                               | 74           |
| $[\text{Rh}(\text{COD})(\text{phen})]\text{Br}$ (IV)  | 49.9<br>(51.0)            | 4.3<br>(4.3) | 5.9<br>(5.9) | <sup>a</sup>   | —                                    | 52           |
| $[\text{Rh}(\text{COD})(\text{bipy})]\text{Br}$ (V)   | 46.9<br>(48.4)            | 4.7<br>(4.5) | 6.1<br>(6.3) | 109  | —                                    | 70           |
| $[\text{Rh}(\text{COD})(5,6\text{-Me}_2\text{phen})]\text{Br}$ (VI)                           | 50.4<br>(52.9)            | 4.5<br>(4.8) | 5.6<br>(5.6) | 121  | —                                    | 75           |
| $[\text{Rh}(\text{COD})(4,7\text{-Ph}_2\text{phen})]\text{Br}$ (VII)                          | 60.1<br>(61.6)            | 4.8<br>(4.5) | 4.3<br>(4.5) | 114  | —                                    | 66           |
| $\text{Rh}(\text{Br})(\text{COD})(\text{quin})$ (VIII)  | 47.5<br>(48.6)            | 4.8<br>(4.6) | 3.5<br>(3.3) | 10   | 193(s)                               | 53           |
| $\text{Rh}(\text{Cl})(\text{COD})(\text{quin})$ (IX)  | 54.0<br>(54.3)            | 5.0<br>(4.5) | 4.4<br>(3.7) | 10   | 285(sh)<br>265(m)                    | 31           |
| $[\text{Rh}(\text{COD})(4,7\text{-Ph}_2\text{phen})][\text{Rh}(\text{COD})\text{Br}_2]$ (X)   | 51.8<br>(52.5)            | 4.4<br>(4.4) | 3.3<br>(3.1) | 96   | 178(m)                               | 72           |
| $[\text{Rh}(\text{COD})(\text{phen})][\text{Rh}(\text{COD})\text{Br}_2]$ (XI)                 | 43.8<br>(44.1)            | 4.3<br>(4.2) | 3.7<br>(3.7) | <sup>a</sup>   | 191(s)                               | 69           |
| $[\text{Rh}(\text{COD})(5,6\text{-Me}_2\text{phen})][\text{Rh}(\text{COD})\text{Br}_2]$ (XII) | 45.4<br>(45.6)            | 4.6<br>(4.6) | 3.4<br>(3.5) | <sup>a</sup>   | 179(s)                               | 65           |
| $[\text{Rh}(\text{COD})(\text{bipy})][\text{Rh}(\text{COD})\text{Br}_2]$ (XIII)               | 42.7<br>(42.3)            | 4.3<br>(4.4) | 4.1<br>(3.8) | 104  | 190(m)                               | 47           |
| $[\text{Rh}(\text{COD})(\text{tmen})][\text{Rh}(\text{COD})\text{Br}_2]$ (XIV)                | 36.0<br>(37.8)            | 5.8<br>(5.8) | 4.1<br>(4.0) | 116  | 195(s)                               | 79           |
| $\text{Rh}(\text{Br})(\text{COD})(2,9\text{-Me}_2\text{phen})$ (XV)                           | 51.6<br>(52.9)            | 4.8<br>(4.8) | 5.4<br>(5.6) | 2  | 202(s)                               | 48           |
| $\text{Rh}(\text{Br})(\text{COD})(2,9\text{-Me}_2\text{-}4,7\text{-Ph}_2\text{phen})$ (XVI)   | 61.6<br>(62.6)            | 5.0<br>(4.9) | 4.5<br>(4.3) | 2  | 202(s)                               | 85           |
| $\text{Rh}(\text{Br})(\text{COD})(\text{en})$ (XVII)  | 33.6<br>(34.2)            | 5.7<br>(5.7) | 7.2<br>(8.0) | <sup>a</sup>   | 202(s)                               | 65           |

<sup>a</sup> Insoluble in acetone.

with a Nicolet 60SX spectrophotometer in the range 500–140  $\text{cm}^{-1}$ , using polyethylene pellets.

#### Preparation of $\text{Et}_4\text{N}[\text{Rh}(\text{COD})\text{X}_2]$ complexes

To 5  $\text{cm}^3$  of dichloromethane solutions of  $[\text{Rh}(\text{COD})\text{X}_2]$  (0.12 mmol), the appropriate  $\text{Et}_4\text{NX}$  (0.24 mmol) was added. Upon addition of diethyl ether yellow precipitates were formed, filtered off, washed with diethyl ether and air-dried.

#### Preparation of $\text{Ph}_4\text{As}[\text{Rh}(\text{NBD})\text{Cl}_2]$

Addition of  $\text{Ph}_4\text{AsCl}$  (0.24 mmol) to 5  $\text{cm}^3$  of a dichloromethane solution of  $[\text{Rh}(\text{NBD})\text{Cl}_2]$  (0.12

mmol), followed by addition of n-hexane gave a yellow solid that was filtered off, washed with n-hexane and air-dried.

#### Reactions of $\text{Et}_4\text{N}[\text{Rh}(\text{COD})\text{X}_2]$

(i) *Reaction with ethylenediamine.* Addition of ethylenediamine (0.12 mmol) to solutions of  $\text{Et}_4\text{N}[\text{Rh}(\text{COD})\text{X}_2]$  (0.12 mmol) in 5  $\text{cm}^3$  of dichloromethane yield yellow solids that were filtered off, washed with dichloromethane and air dried. The solids obtained were identified as  $\text{Rh}(\text{X})(\text{COD})(\text{en})$ .

(ii) *Reaction with phenanthroline type ligands.* To  $\text{Et}_4\text{N}[\text{Rh}(\text{COD})\text{X}_2]$  (0.12 mmol) solutions in 5  $\text{cm}^3$  of dichloromethane, the corresponding stoi-

chiometric amounts (0.12 mmol) of 1,10-phenanthroline, 2,2'-bipyridine, 5,6-dimethyl-1,10-phenanthroline or 4,7-diphenyl-1,10-phenanthroline were added, whereupon the solution turned red or violet. The formed  $[\text{Rh}(\text{COD})(\text{LL})]\text{Br}$  complexes precipitated immediately as red crystals when  $\text{LL} = 1,10\text{-phenanthroline}$  or 5,6-dimethyl-1,10-phenanthroline were added and required elimination of  $\text{CH}_2\text{Cl}_2$  and addition of acetone when  $\text{LL} = 2,2'\text{-bipyridine}$  (red crystals) or 4,7-diphenyl-1,10-phenanthroline (violet crystals) was used. The complexes obtained were filtered off, washed with acetone and air-dried.

(iii) *Reaction with triphenylphosphine.*  $\text{Ph}_3\text{P}$  (0.08 mmol) was added to yellow solutions of  $\text{Et}_4\text{N}[\text{Rh}(\text{COD})\text{X}_2]$  (0.08 mmol) in 5  $\text{cm}^3$  of  $\text{CH}_2\text{Cl}_2/\text{MeOH}$  (1:1) whereupon the solutions became orange. The elimination of dichloromethane yielded orange  $\text{Rh}(\text{X})(\text{COD})(\text{Ph}_3\text{P})$  compounds that were filtered off, washed with methanol and air-dried.

(iv) *Reaction with quinoline.* Quinoline (0.12 mmol) was added to 5  $\text{cm}^3$  of yellow dichloromethane solutions of  $\text{Et}_4\text{N}[\text{Rh}(\text{COD})\text{X}_2]$  (0.12 mmol). Addition of methanol (3  $\text{cm}^3$ ) and elimination of dichloromethane led to yellow precipitates of  $\text{Rh}(\text{X})(\text{COD})(\text{quin})$  complexes that were filtered off, washed with methanol and air-dried.

(v) *Reaction with carbon monoxide.* Carbon monoxide ( $P = 1$  atm) was bubbled through dichloromethane solutions of  $\text{Et}_4\text{N}[\text{Rh}(\text{COD})\text{X}_2]$  (0.12 mmol) for 30 min. IR spectra of the yellow solutions obtained confirmed the formation of  $\text{Et}_4\text{N}[\text{Rh}(\text{CO})_2\text{X}_2]$  complexes.

(vi) *Reaction with tin(II) halides.*  $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$  (0.30 mmol) in 5  $\text{cm}^3$  of methanol was added to 5  $\text{cm}^3$  of  $\text{Et}_4\text{N}[\text{Rh}(\text{COD})\text{Cl}_2]$  (0.10 mmol) in dichloromethane. The addition of  $\text{Et}_4\text{NCl}$  (0.10 mmol) followed by elimination of dichloromethane gave  $(\text{Et}_4\text{N})_2[\text{Rh}(\text{SnCl}_3)_3(\text{COD})]$  that was filtered off, washed with methanol and air-dried.

$\text{Et}_4\text{N}[\text{Rh}(\text{COD})\text{Br}_2]$  did not react with  $\text{SnBr}_2$  under the same conditions.

#### *Preparation of $[\text{Rh}(\text{COD})(\text{LL})]^+[\text{Rh}(\text{COD})\text{Br}_2]^-$ complexes*

Addition of the corresponding ligand (0.06 mmol) to 5  $\text{cm}^3$  of dichloromethane solutions of *in situ* prepared  $[\text{Rh}(\text{COD})\text{Br}_2]$  (0.06 mmol) gave red solids when  $\text{LL} = 1,10\text{-phenanthroline}$ , 2,2'-bipyridine or 5,6-dimethyl-1,10-phenanthroline. When  $\text{LL} = 4,7\text{-diphenyl-1,10-phenanthroline}$  or  $\text{N,N,N',N'-tetramethylethylenediamine}$ , addition of diethyl ether was necessary to afford precipitation of the corresponding compounds. The

complexes obtained were filtered off, washed with diethyl ether and air-dried.

#### *Preparation of $\text{Rh}(\text{Br})(\text{COD})(\text{LL})$ complexes*

0.12 mmol of the corresponding ligand were added to 5  $\text{cm}^3$  of dichloromethane solutions of *in situ* prepared  $[\text{Rh}(\text{COD})\text{Br}_2]$  (0.06 mmol). When  $\text{LL} = \text{ethylenediamine}$ , a yellow solid appeared immediately. When  $\text{LL} = 2,9\text{-dimethyl-1,10-phenanthroline}$  or 2,9-dimethyl-4,7-diphenyl-1,10-phenanthroline, addition of diethyl ether gave yellow solids. The complexes thus obtained, were filtered off, washed with diethyl ether and air-dried.

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