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BENZO[*c*]CINNOLINE COMPLEXES OF RHODIUM *

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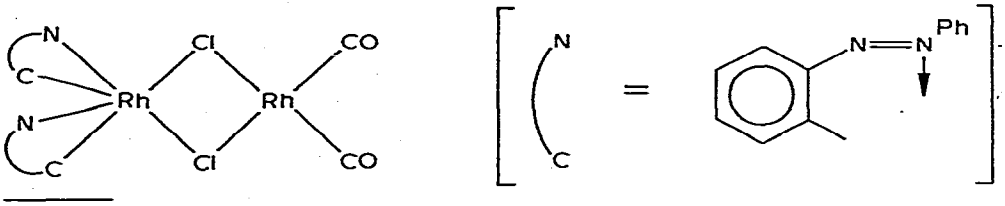
Summary

Complexes of the type $RhCl(L_2)$ (benzo[*c*]cinnoline) (L = CO, PF_3, or PPh_3) have been synthesised and their steroechemistry deduced by NMR spectroscopy. Treatment of $RhCl(PF_3)(PPh_3)_2$ with $Me_3SiN{=}NPh$ did not give the expected phenyldiazorhodium complex; instead attack occurs at the P—F bond to give Me_3SiF and $RhCl(PF_2N{=}NPh)(PPh_3)_2$.

Introduction

The coordination chemistry of azo-compounds is of current interest [1,2]. Previously we described some benzo[*c*]cinnoline and phenanthridine pentacarbonyl complexes of chromium, molybdenum and tungsten [3]. Unlike azobenzene, benzo[*c*]cinnoline is unlikely to undergo any cyclometallation reactions [4]. Previously, *ortho*-metallated complexes containing more than one metal—carbon σ-bond were obtained from reactions between rhodium complexes and azobenzene [5,6]. The reaction between $[RhCl(CO)_2]_2$ and azobenzene (HAzb) is known to produce the 2-(phenylazo)phenylrhodium complex $[Rh_2Cl_2(CO)_2(azb)_2]$ [6]. The postulated mechanism involves (i) initial coordination of *trans*-azobenzene to rhodium(I), (ii) an oxidative addition step to form a metal hydride, (iii) reaction with a second molecule of azobenzene via an *ortho*-metallation with H_2 elimination, and (iv) subsequent reaction with $[RhCl(CO)_2]_2$. Since



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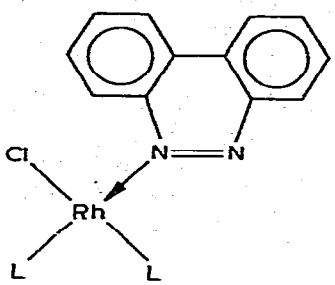
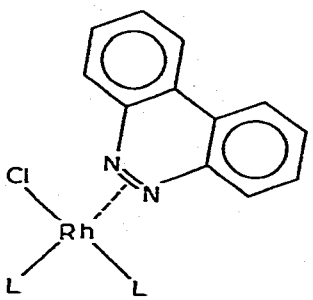
the intermediate azobenzene complex could not be isolated, we have studied the syntheses of some related benzo[*c*]cinnoline rhodium complexes. An attempt to obtain a phenyldiazorhodium(I) complex by treating $RhCl(PF_3)(PPh_3)_2$ with $Me_3SiN{=}NPh$ unexpectedly gave a complex containing the previously unknown difluorophosphine $PF_2N{=}NPh$.

Results and discussion

Benzo[*c*]cinnoline reacts readily with di-μ-chlorotetracarbonyldirhodium, $[RhCl(CO)_2]_2$, in refluxing benzene for 3 h to give a yellow crystalline material, I, and an insoluble red powder, II, in almost equal amounts. Longer reflux times led to higher yields of the insoluble product.

Elemental analyses of the two products I and II were similar, but owing to the insolubility of the red powder, difficulty was encountered in obtained a pure sample. The analytical data for I was consistent with its formulation as either



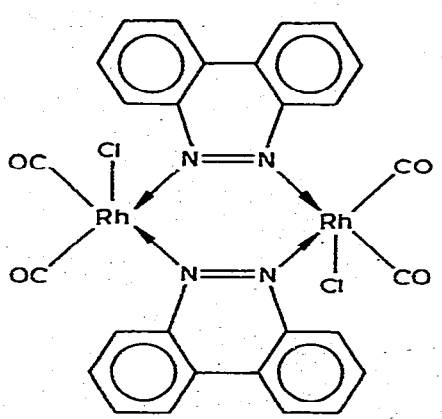


(Ia, L = CO; IIIa, L = PF_3; IVa, L = PPh_3)

(Ib, L = CO; IIIb, L = PF_3; IVb, L = PPh_3)

Ia or Ib (L = CO), and the monomeric nature of the compound was established by a molecular weight determination. The IR spectrum of I in chloroform solution shows two bands at 2090 and 2015 cm^{-1} in the carbonyl stretching region as expected for a complex containing two mutually *cis*-carbonyl ligands [6—10].

The IR spectrum of the insoluble red product, II, is similar to I in the carbonyl stretching region showing bands at 2090 and 2000 cm^{-1} and on the basis of its insolubility and the fact that both I and II gave benzo[*c*]cinnoline and *trans*-$RhCl(CO)(PPh_3)_2$ on treatment with triphenylphosphine is tentatively assigned the dimeric structure shown below.

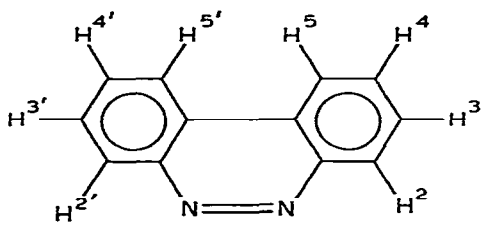


Benzo[*c*]cinnolinerhodium complexes of the type $RhClL_2$(benzo[*c*]cinnoline) (III L = PF_3; IV, L = PPh_3) are also readily obtained in good yield from the reaction of benzo[*c*]cinnoline and $[RhCl(PF_3)_2]_2$ and $RhCl(PPh_3)_3$ respectively. III reacts with triphenylphosphine to give benzo[*c*]cinnoline and the known complex *trans*-$RhCl(PF_3)(PPh_3)_2$.

The reaction of benzo[*c*]cinnoline with hydrated rhodium trichloride in refluxing ethanol gives the insoluble 1 : 1 complex $RhCl_3$(benzo[*c*]cinnoline) V, which in treatment with pyridine gives benzo[*c*]cinnoline and the known complex *cis*-$RhCl_3(py)_3$ [11].

Two types of coordination of the benzo[*c*]cinnoline to the metal atom are possible for complexesI, III and IV: (i) σ-bonding via one nitrogen lone pair as in Ia, IIIa, IVa; or (ii) π-bonding via the N=N double bond as in Ib, IIIb, IVb. Both types of bonding are known in related metal—azo systems.

Further information on the nature of the bonding comes from ^1H and ^{13}C NMR studied. The ^1H NMR spectrum of free benzo[*c*]cinnoline shows three well-resolved patterns of lines in the ratio 1 : 1 : 2, which are readily assigned to pairs of equivalents protons (H^2, $H^{2'}$, H^3, $H^{3'}$) and (H^4, $H^{4'}$, H^5, $H^{5'}$) respectively, viz.



In the room temperature ^1H NMR spectrum of I the low field pattern for H^2 and $H^{2'}$ is much broader than the other resonances, suggesting that in I the H^2 and $H^{2'}$ are non equivalent, i.e., the azo ligand is coordinated to rhodium via one nitrogen lone pair as in Ia. This ^1H resonance broadens considerably at lower temperatures and since the ^{13}C NMR spectrum of I is very similar in appearance to free benzo[*c*]cinnoline, the metal may well be 'shuttling' between the two nitrogen atoms positions, as found in other systems [3,12,13].

The ^{19}F and ^{31}P NMR spectra of III (Fig. 1 and 2) show that the two PF_3 ligands are in different environments, thereby establishing the same *cis*-configuration as found for the dicarbonyl complex I. At room temperature the ^{19}F NMR spectrum of III exhibits four broad patterns which sharpen on cooling until at —70°C the spectrum consists of four widely spaced sets of doublets of equal intensity (from coupling to phosphorus, and rhodium ^{103}Rh). Further fine structure is evident from coupling to the ^{31}P and ^{19}F nuclei of the other PF_3 ligand and the complexity of the spectrum suggests that it is not strictly suitable for a first-order analysis. The loss of fine structure in the ^{19}F NMR spectrum at room temperature is most likely due to an intermolecular phosphine exchange process similar to that previously observed [14,15] for $[RhCl(PF_3)_2]_2$.

The ^{31}P NMR spectrum of III at —80°C contains two overlapping 1 : 3 : 3 : 1 quartet patterns, indicating the presence of two different PF_3 environments, each line showing considerable further fine structure. At room temperature the spectrum is broader, but the PF_3 ligands remain non-equivalent. The magnitude

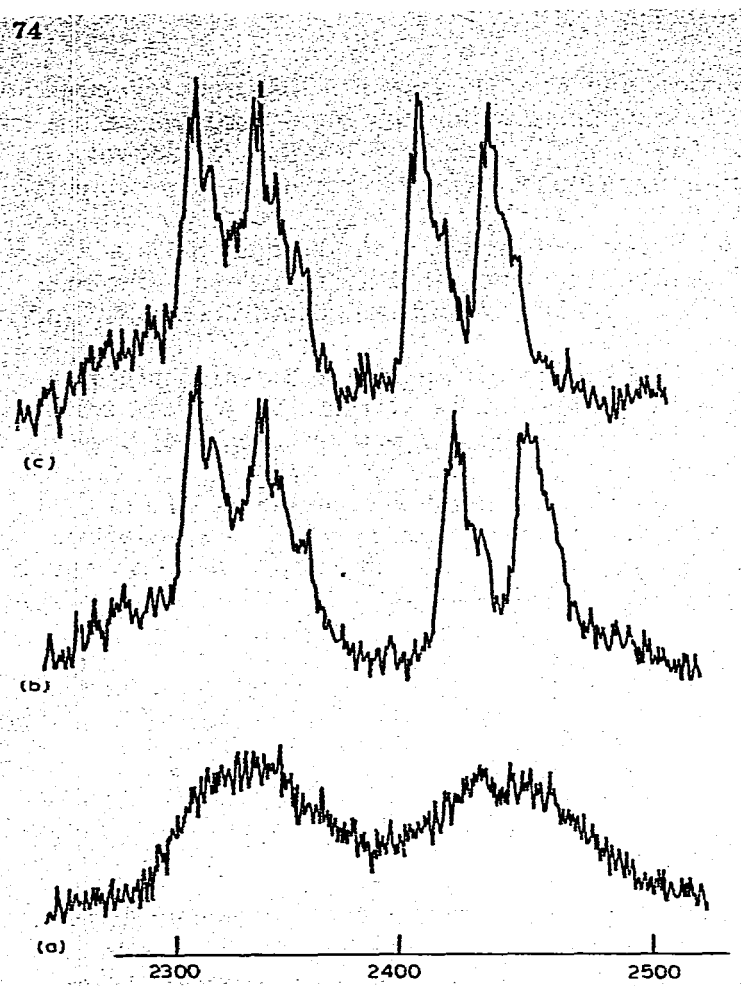


Fig. 1. The high field half of the ^{19}F NMR spectrum (at 94.1 MHz) of *cis*-RhCl(cinn)$(PF_3)_2$ at room temperature (a), —50°C (b), —70°C (c) (scale in Hz to high field of CCl_3F). An exact mirror image of these patterns occurs at low field.

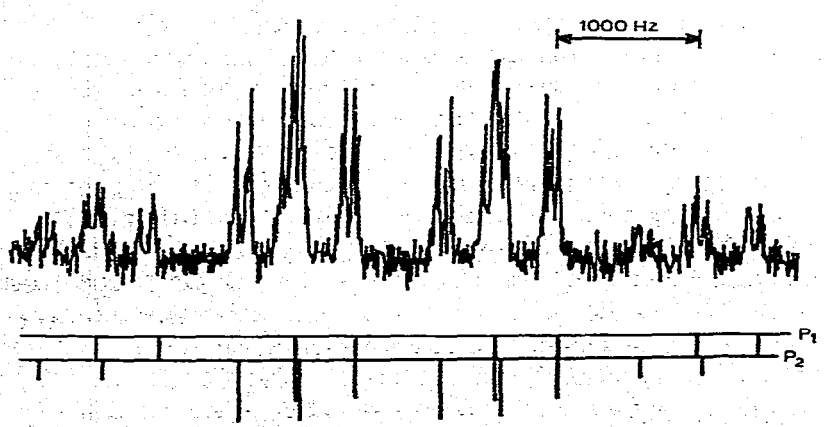


Fig. 2. ^{1}H decoupled ^{31}P NMR spectrum (at 40.49 MHz) of $CHCl_3$ solution of *cis*-RhCl(cinn)$(PF_3)_2$ at —80°C.

of ^1J(PRh) for the PF_3 ligands *cis* and *trans* to chlorine are found to be approx. 300 and 350 Hz respectively, which are similar to the values observed in $[RhCl(PF_3)_2]_2$ [16].

The $\{^1H\}$—^{31}P NMR spectrum of IV (Fig. 3) shows two overlapping AB patterns expected for two non-equivalent PPh_3 ligands coupled to the ^{103}Rh nucleus. The resonance exhibiting the larger ^{103}Rh—^{31}P coupling constant is assigned to PPh_3 *trans* to chlorine (see Table 1).

Abel and Burton [17] have obtained an arylazomanganese carbonyl complex $[Mn(CO)_4N_2Ph]_2$ by treating $MnBr(CO)_5$ with $Me_3SiN{=}NPh$ [18], and suggested that the reaction mechanism involves initial displacement of carbon monoxide followed by elimination of bromotrimethylsilane. The related reaction of trimethylsilylpyrazole with $[RhCl(CO)_2]_2$ affords the bridged dinuclear pyrazolylrhodium carbonyl complex [19].

When, however, $Me_3SiN{=}NPh$ is treated with $RhCl(PF_3)(PPh_3)_2$, no elimination of trimethylchlorosilane results. Instead phosphorus—fluorine bond cleavage occurs to give Me_3SiF and the rhodium(I) complex VI, containing a coordinated $PF_2N{=}NPh$ ligand. The free fluorophosphine has not been previously reported.

$$RhCl(PF_3)(PPh_3)_2 + Me_3SiN{=}NPh \rightarrow \underset{(VI)}{RhCl(PF_2N_2Ph)(PPh_3)_2} + Me_3SiF$$

The formulation of VI was confirmed by elemental analysis and NMR spectroscopy. The ^{19}F NMR spectrum shows the expected 12 line pattern arising from two doublets of overlapping 1 : 2 : 1 triplets (from coupling to the directly bonded phosphorous, rhodium and two equivalent PPh_3 groups). The $\{^1H\}$—^{31}P NMR spectrum shows the expected low field widely spaced triplet pattern for the PF_2 moiety, each line being split by coupling to ^{103}Rh and the two equivalent PPh_3 groups. The resonances of the PPh_3 ligands appear as a doublet of doublets of triplets from coupling to ^{103}Rh, ^{31}P and two ^{19}F nuclei. Chemical

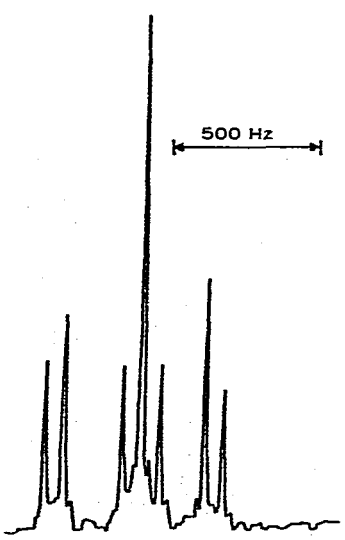


Fig. 3. ^1H decoupled ^{31}P NMR spectrum (at 40.49 MHz) of *cis*-RhCl(cinn)$(PPh_3)_2$ in $CHCl_3$ solution.

TABLE 1

^{19}F AND ^{31}P NMR DATA FOR COMPLEXES III, IV AND V

Complex	$\varphi_{(F)}$ [a]	$\delta_{(P)}$ [b]	$^1J(PF)$ [c]	$^3J(PF')$ [c]	$^2J(PP')$ [c]	$^1J(RhF)$ [c]	$^1J(RhP)$ [c]
cis-RhCl(benzo[*c*]-cinnoline)$(PF_3)_2$ [d] (III)	19.2	15.7	1304	14	86.0	28 [f]	300 [f]
	20.2	24.7	1324	19		31 [f]	350 [f]
cis-RhCl(benzo[*c*]-cinnoline)$(PPh_3)_2$ [e] (IV)		88.0			48.8		200.2
		93.6					166.0
trans-RhCl$(PF_2N_2Ph)(PPh_3)_2$	47.7	51.2	1095	11	46.4	15	275.9

[a] In ppm (rel. CCl_3F. [b] In ppm [rel. $(MeO)_3P$]. [c] In Hz. [d] The higher values for $\varphi_{(F)}$ and $\delta_{(P)}$ refer to PF_3 *trans* to chlorine. [e] The lower value for $\delta_{(P)}$ refers to PPh_3 *trans* to chlorine. [f] Approximate values.

shift and coupling constant data are listed in Table 1. As expected, the analogous $RhCl(CO)(PPh_3)_2$ did not react with $Me_3SiN{=}NPh$.

Experimental

Experimental procedures were carried out as described earlier [3]. Benzo[*c*]-cinnoline (Ralph Emanuel Ltd.) was used as supplied. Trimethylsilylphenyldiimine [18] was prepared by the literature method, and the purity of the intensely blue liquid (b.p. 72—74°C/2.5 mmHg) was checked by IR and ^1H NMR spectroscopy. $[RhCl(CO)_2]_2$ was prepared by a slight modification of the method of Powell and Shaw [20], while $[RhCl(PF_3)_2]_2$ [15], $RhCl(PPh_3)_3$ [21], $RhCl(PF_3)(PPh_3)_2$ [15], and $RhCl(CO)(PPh_3)_2$ [21,22] were prepared by literature methods.

Reaction of di-μ-chlorotetracarbonyldirhodium(I) with benzo[c]cinnoline

A mixture of di-μ-chlorotetracarbonyldirhodium (0.4 g, 1 mmol) and benzo[*c*]cinnoline (0.450 g, 2.5 mmol) in benzene (20 ml) was refluxed for 2 h and the resulting red precipitate was filtered off, washed with benzene and hexane and dried in vacuo to afford chloro(benzo[*c*]cinnoline)dicarbonylrhodium(I) dimer (II), (0.280 g, 37%), m.p. 138°C (dec.). (Found: C, 43.2; H, 2.3; N, 7.5. $C_{14}H_8ClN_2O_2Rh$ calcd.: C, 44.85; H, 2.13; N, 7.47%.) Infrared spectrum: 2080vs, 2000vs, 1612m, 1580m, 1504s, 1292w, 1265w, 1190s, 1175m, 1146vs, 1108s, 1100s, 1040m, 978w, 970m, 962w, 951m, 889w, 775vs, 768vs, 726s, 718s, 705vs, 640w, 625m, 569m, 524s, 498s cm^{-1} (Nujol mull). The filtrate was concentrated under vacuum and addition of hexane yielded yellow crystals of *cis*-chloro(benzo[*c*]cinnoline)dicarbonylrhodium(I),(I), (0.330 g, 44%), m.p. 128°C (dec.). (Found: C, 44,4; H, 2.3; Cl, 9.6; N, 7.5. $C_{14}H_9ClN_2O_2Rh$ calcd.: C, 44.85; H, 2.13; Cl, 9.47; N, 7.47%.) Mol. wt. found 407 (osmometrically in CCl_2H_2 solution). $RhCl_2(C_{12}H_8N_2)(CO)_2$ calcd. 375.5. Infrared spectrum: 3078vw, 3062vw, 2090vs *, 2015vs *, 1910w, 1580m, 1500m, 1283w, 1269m, 1147w, 1198m, 1176m, 1147s, 1133s, 1107m, 1000w, 960w, 950m, 786m, 768vs,

* CCl_3H solution.

716s, 633m, 610s, 542w, 537w, 510m, 487, 480m, 402w cm^{-1} (Nujol mull). The ^1H NMR spectrum shows multiplets at τ1.2 (2H), 1.5 (2H) and 2.1 (4H) ($CDCl_3$ solution) and the ^{13}C NMR spectrum five separate resonances at 145.4, 133.4, 130.8, 130.3 and 121.7 ppm (rel. TMS).

Reaction of di-μ-chlorotetrakis(trifluorophosphine)dirhodium(I) with benzo[c]cinnoline

In a similar fashion to the above, $[RhCl(PF_3)_2]_2$ (0.257 g, 0.4 mmol) and benzo[*c*]cinnoline (0.180 g, 1 mmol) in refluxing benzene (20 ml) for 3 h gave yellow crystals (from benzene/hexane) of *cis*-chloro(benzo[*c*]cinnoline)bis(trifluorophosphine)rhodium(I) (III), (0.260 g, 67%), m.p. 140—145°C (dec.). (Found: C, 29.8; H, 1.7; N, 5.7. $C_{12}H_8ClF_6N_2P_2Rh$ calcd.: C, 29.21; H, 1.62; N, 5.6%.) Infrared spectrum: 3068w, 1615m, 1532s, 1502m, 1282m, 1271(sh, 1240w, 1147s, 1135s, 1108m, 976(sh), 952(sh), 900vs(br), 788s, 767vs, 723s, 685w, 633m, 615s, 565vs, 522vs cm^{-1} (Nujol mull). The ^1H NMR spectrum shows three broad resonances at τ0.9 (2H), 1.32 (2H) and 1.88 (4H) ($CDCl_3$ solution).

Reaction of chlorotris(triphenylphosphine)rhodium(I) with benzo[c]cinnoline

A suspension of chlorotris(triphenylphosphine)rhodium (0.200 g, 0.2 mmol) was stirred at room temperature with benzo[*c*]cinnoline (0.090 g, 0.5 mmol) in benzene (20 ml) under dinitrogen gas for 12 h. The initially insoluble rhodium complex dissolved to give an orange solution which yielded *cis*-chloro(benzo[*c*]cinnoline)bis(triphenylphosphine)rhodium, $RhCl(C_{12}H_8N_2)(PPh_3)_2$, (0.120 g, 70%), m.p. 113—115°C (dec.) as a brown-orange powder after recrystallisation from benzene/hexane (Found: C, 69.0; H, 4.8; N, 3.0. $C_{48}H_{38}ClN_2P_2Rh$ calcd.: C, 68.40; H, 4.5; N, 3.32%.) Infrared spectrum: 3062w, 1610w, 1580m, 1500w, 1312w, 1290w(br), 1186m, 1160w, 1138(sh), 1141m, 1094vs, 1075(sh), 1030s, 1002m, 963w, 946m, 850m, 766vs, 753s, 744vs, 720s, 700vs, 678vs, 633m, 624m, 610m, 550vs, 531vs, 520s, 500s, 462w, 443m, 428m cm^{-1} (Nujol mull).

Reaction of rhodium trichloride trihydrate with benzo[c]cinnoline

A mixture of $RhCl_3 \cdot 3H_2O$ (0.270 g, 1 mmol) and benzo[*c*]cinnoline (0.720 g, 4 mmol) in absolute ethanol was stirred at room temperature, but no reaction occurred. On refluxing the solution, a brown-orange precipitate deposited, which was filtered off, washed with hot ethanol and dried under vacuum. Elemental analyses were consistent with formulation of the product as trichloro(benzo[*c*]cinnoline)rhodium (0.250 g, 64% yield based on Rh), m.p. 280°C (dec.). (Found: C, 39.5; H, 2.4; N, 7.5. $C_{12}H_8Cl_3N_2Rh$ calcd.: C, 37.29; H, 2.04; N, 7.24%.). Infrared spectrum: 1610m, 1575m, 1445s, 1347w, 1260vw, 1148m, 1132m, 1102m, 1030vw, 970w, 905w, 760vs, 735sh, 720vs cm^{-1} (Nujol mull).

Reaction of trichloro(benzo[c]cinnoline)rhodium(III) with pyridine

Pyridine (1 ml) was added to a suspension of trichloro(benzo[*c*]cinnoline)-rhodium(III) (0.200 g, 0.5 mmol) in ether (15 ml) and the mixture was stireed for 30 min. The complex dissolved to give a light-orange solution. After filtration and reducing the volume of the solvent, the solution was diluted with water to precipitate yellow crystals of benzo[*c*]cinnoline (0.053 g, 57%). The filtrate

was then concentrated and cooled to give yellow crystals of trichlorotripyridinerhodium(III), $RhCl_3(py)_3$ (0.150 g, 50%). (Found: C, 39.9; H, 4.4; N, 8.9. $C_{15}H_{15}Cl_3N_3Rh$ calcd.: C, 40.0; H, 3.3; N, 9.4%.) The infrared spectrum and melting point were identical with a genuine sample [11].

Reaction of cis-chloro(benzo[c]cinnoline)dicarbonylrhodium(I) with triphenylphosphine

Complex I (0.112 g, 0.15 mmol) was treated with triphenylphosphine (0.157 g, 0.6 mmol) in benzene at room temperature. After stirring the mixture for 15 h the solvent was removed and recrystallisation from chloroform/hexane afforded *trans*-$RhCl(CO)(PPh_3)_2$ (0.150 g, 72%) identified by its characteristic infrared spectrum and melting point. Concentration of the filtrate have benzo[c]cinnoline (0.038 g, 70%) after recrystallisation from hexane.

Reaction of chloro(benzo[c]cinnoline)dicarbonylrhodium dimer with triphenylphosphine

A suspension of the insoluble dimeric benzo[c]cinnoline rhodium complex II (0.112 g, 0.07 mmol) in benzene was agitated with an excess of triphenylphosphine (0.160 g, 0.6 mmol) at room temperature for 10 h. Yellow crystals of *trans*-$RhCl(CO)(PPh_3)_2$ (0.180 g, 80%) were obtained after recrystallisation from chloroform/hexane. Benzo[c]cinnoline (0.033 g, 60%) was subsequently isolated from the filtrate and recrystallised from hexane.

Reaction of cis-chloro(benzo[c]cinnoline)bis(trifluorophosphine)rhodium with triphenylphosphine

A mixture of *cis*-chloro(benzo[c]cinnoline)bis(trifluorophosphine)rhodium (0.146 g, 0.3 mmol) and triphenylphosphine (0.263 g, 1 mmol) was stirred in benzene (20 ml) for 8 h at room temperature. The volume of the solvent was reduced under vacuum and hexane was added to yield *trans*-$RhCl(PF_3)(PPh_3)$-$(PPh_3)_2$ (0.170 g, 75%), identified by comparison of its infrared spectrum and m.p. with an authentic sample [15]. Benzo[c]cinnoline (0.040 g, 70%) was recovered from the filtrate by recrystallisation from hexane.

Reaction of trans-chlorotrifluorophosphinebis(triphenylphosphine)rhodium(I) with trimethylsilylphenyldiimine

A mixture of *trans*-chlorotrifluorophosphinebis(triphenylphosphine)rhodium-(I) (0.750 g, 1 mmol) and trimethylsilylphenyldiimine (0.270 g, 1.5 mmol) in benzene (40 ml) was refluxed under dinitrogen for 5 h. The solution changed colour from green to light-red. After reducing the volume of the solvent in vacuo (~10 ml) hexane was added to precipitate the complex *trans*-chlorophenyldiazodifluorophosphinebis(triphenylphosphine)rhodium(I), $RhCl(PF_2N_2Ph)(PPh_3)_2$ as a brown-orange powder (0.560 g, 68%) m.p. 128°C. (Found: C, 59.8; H, 4.6; N, 3.0. $C_{42}H_{35}ClF_2N_2P_3Rh$ calcd.: C, 60.25; H, 4.18; N, 3.34%.) Infrared spectrum: 159m(br), 1570(sh), 1260m, 1185w, 1145w, 1114m, 1092s, 1070w, 1030m, 1000w, 9152, 820s, 790(sh), 745vs, 728s, 693vs, 560m, 547s, 530vs, 515(sh), cm^{-1} (Nujol mull).

Reaction of trans-chlorocarbonylbis(triphenylphosphine)rhodium(I) with trimethylsilylphenyldiimine

No reaction was observed when *trans*-chlorocarbonylbis(triphenylphosphine)-rhodium(I) (0.350 g, 0.5 mmol) was refluxed with trimethylsilylphenyldiimine (0.180 g, 1 mmol) in benzene under nitrogen gas for 5 h. The blue trimethylsilylphenyldiimine was recovered unchanged at the end of the reaction.

Acknowledgement

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