Journal of Organometallic Chemistry, 476 (1994) 121–126

Protonation of diphosphine and phosphite derivatives of dodecacarbonyltriruthenium

Shariff E. Kabir, Arzu Miah and Khabir Uddin

Department of Chemistry, Jahangirnagar University, Dhaka 1342 (Bangladesh)

Antony J. Deeming

Department of Chemistry, University College London, 20 Gordon Street, London WC1H 0AJ (UK)

(Received June 30, 1993; in revised form November 17, 1993)

Abstract

Protonation of the clusters $[Ru_3(CO)_{10}(\mu\text{-diphos})]$ where diphos = $Ph_2P(CH_2)_nPPh_2$ [dppm ($n = 1$), dppe ($n = 2$), dppp ($n = 3$) or dppb ($n = 4$)] with CF_3CO_2H gave the monohydrido cations $[Ru_3(\mu\text{-H})(CO)_{10}(\mu\text{-diphos})]^+$, initially characterised by NMR methods. The compounds $[Ru_3(\mu\text{-H})(CO)_{10}(\mu\text{-diphos})]$ $[PF_6]$ were isolated for dppp and dppb, but deprotonation occurred more readily for dppm and dppe and in these cases the salts could not be isolated. The hydride and the diphos ligands span the same edge of the metal triangle in the dppp and dppb cations $[Ru_3(\mu\text{-H})(CO)_{10}(\mu\text{-diphos})]^+$ but different edges in the dppm species. The two interconverting isomers are observed in solution when diphos = dppe correspond to these different forms. Protonation of $[Ru_3(CO)_{10}\{P(OMe)_3\}_2]$ with CF_3CO_2H gives $[Ru_3(\mu\text{-H})(CO)_{10}\{P(OMe)_3\}_2]^+$ as two isomers in solution, the major with equivalent and the minor with non-equivalent phosphite ligands.

Key words: Ruthenium; Carbonyl; Hydride; Diphosphine; Phosphite; Fluxionality

1. Introduction

Protonation of dodecacarbonyltriosmium [1–3] and its monophosphine [1,2,4] and diphosphine [5–7] substituted derivatives has been investigated. Protonation of dodecacarbonyltriruthenium has been reported but the tertiary phosphine substituted derivatives do not seem to have been studied previously. We report in this paper the synthesis and protonations of the bridging diphosphine compounds $[Ru_3(CO)_{10}(\mu\text{-diphos})]$, where diphos = dppm, dppe, dppp, and dppb. We also describe the protonation of the monophosphite compound 1,2-$[Ru_3(CO)_{10}\{P(OMe)_3\}_2]$. We wanted to compare the protonation behaviour of the Os_3 and Ru_3 complexes because the isomers commonly obtained for Os_3 had relatively slow conversion rates and consequently it was not always possible to identify the thermodynamically most stable product or products. The greater reactivity of Ru_3 clusters led us to expect products of thermodynamic control and fluxionality involving isomers.

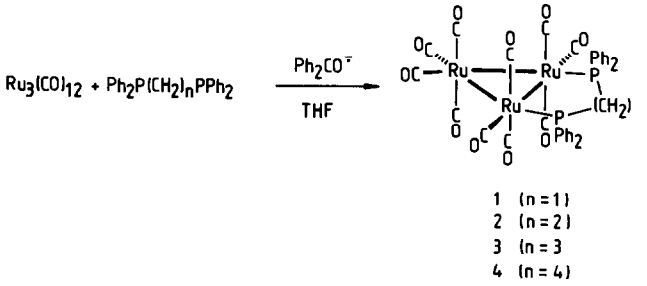
2. Results and discussion

2.1. Synthesis of diphosphine complexes

Reactions of $[Ru_3(CO)_{12}]$ with the series of diphosphines $Ph_2P(CH_2)_nPPh_2$ [$n = 1$ (dppm), 2 (dppe), 3 (dppp) or 4 (dppb)] at room temperature in the presence of catalytic amounts of Ph_2CO^- give the bridging diphosphine clusters $[Ru_3(CO)_{10}\{\mu\text{-}Ph_2P(CH_2)_nPPh_2\}]$ (Scheme 1). The cluster $[Ru_3(CO)_{10}(\mu\text{-dppm})]$ (**1**) was previously prepared from the reaction of $[Ru_3(CO)_{12}]$ with dppm at 50°C and the cluster $[Ru_3(CO)_{10}(\mu\text{-dppe})]$ (**2**) was synthesised from $[Ru_3(CO)_{12}]$ with dppe at 40°C in the presence of Ph_2CO^-. The diphosphines occupy equatorial sites on adjacent Ru atoms in both the dppm and dppe compounds; they were characterised spectroscopically [8,9] and by X-ray diffraction [9,10]. The new compounds $[Ru_3(CO)_{10}(\mu\text{-dppp})]$ (**3**)

Correspondence to: Professor A.J. Deeming.

0022-328X/94/$7.00
SSDI 0022-328X(94)24329-4



Scheme 1.

and $[Ru_3(CO)_{10}(\mu\text{-dppb})]$ (**4**) were characterised by IR, ^1H and $^{31}P\{^1H\}$ NMR and elemental analysis. The $\nu(CO)$ IR spectra for **3** and **4** are very similar to those for **1** [8] and **2** [9], indicating that they form an isostructural series. As expected the $^{31}P\{^1H\}$ NMR spectra of **3** and **4** show singlets (δ 22.35 for **3** and δ 20.45 for **4**).

2.2. Protonation of the diphosphine complexes

We followed the protonation reactions initially by changes in the ^1H NMR spectra upon addition of trifluoroacetic acid (5 mol/mol Ru_3) to $CDCl_3$ or CD_2Cl_2 solutions of the clusters. In the case of the dppp and dppb complexes, the cationic hydrides were isolated as crystals of the hexafluorophosphate salts, which were fully characterised (see Experimental section). However, suitable crystals for single-crystal structure determination were not obtained. The dppm and dppe species are more readily deprotonated, and attempted isolation led to such deprotonation.

Addition of a five-fold molar excess of CF_3CO_2H to a CD_2Cl_2 solution of cluster **1** at room temperature gave a ^1H NMR spectrum (hydride triplet at δ −18.68, J(PH) 6.6 Hz) consistent with the formation of $[Ru_3(\mu\text{-H})(CO)_{10}(\mu\text{-dppm})]^+$ (**5**). The observation of a triplet might be due to protonation at the more electron-rich dppm-bridging site, so that equal coupling to the ^{31}P nuclei would occur, but this would be in direct contrast to our earlier results [7] on the corresponding Os_3 system, for which protonation was at the Os–Os edge not bridged by dppm, the hydride being observed to couple to only one ^{31}P nucleus. The cluster $[Os_3(\mu\text{-H})(CO)_{10}(\mu\text{-dppm})]^+$ gives a ^1H NMR doublet at δ −19.45 [J(PH) 13.6 Hz]. If the protonation sites for Os and Ru are the same, there must be a rapid degenerate hydride migration between the two unbridged edges of the Ru_3 cluster to give time-averaged coupling to both ^{31}P nuclei. This was confirmed by the low-temperature ^1H NMR spectrum (−50°C) (Fig. 1), which shows a double doublet at δ −18.71 [J(PH) 17.7 and 3.5 Hz] and the Os and Ru compounds are therefore probably structurally the same. A rapid fluxional process for Ru (but not for Os), as in Scheme 2, leads to the high-temperature triplet in Fig. 1. In general ruthenium complexes are more labile than osmium ones.

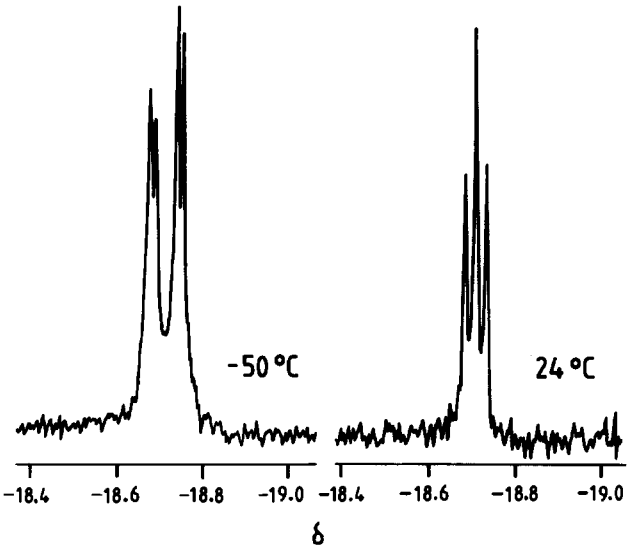
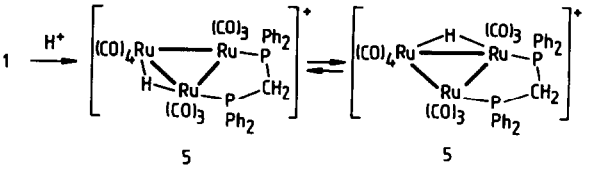
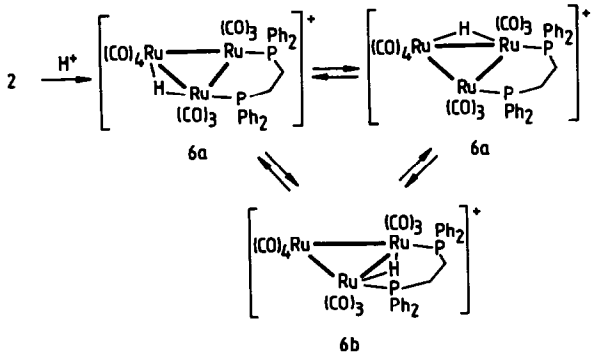


Fig. 1. Variable temperature ^1H NMR spectra of $[Ru_3(\mu\text{-H})(CO)_{10}(\mu\text{-dppm})]^+$ (**5**) in $CDCl_3$ in the hydride region.

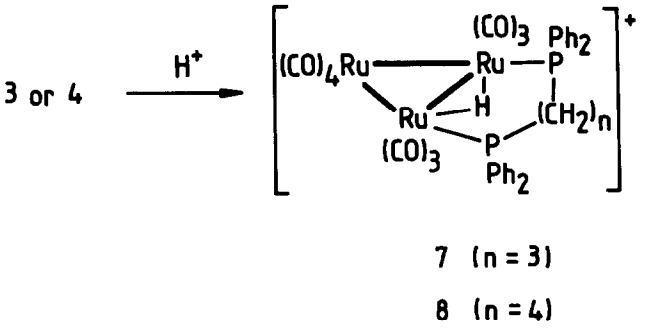
As reported previously for $[Os_3(\mu\text{-H})(CO)_{10}(\mu\text{-dppm})]^+$, the cation **5** does not accommodate the hydride at the dppm-bridged edge because of steric constraint. There is a clash because both ligands want to lie in the M_3 plane and this would bring them unacceptably close [7]. However, the cluster $[Ru_3(\mu\text{-H})(CO)_6(\mu\text{-dppm})_3]^+$ has been shown recently to have a dppm and a hydride ligand bridging the same Ru–Ru edge. In this case there is little alternative because all the edges are dppm bridged [11]. The hydride is above and the dppm below the Ru_3 plane in order to minimise as far as possible steric interaction between them. This is achieved by significant distortion of the $Ru_3(dppm)_3$ framework to allow the proton entry. When there is an unbridged edge as in $[Ru_3(CO)_8(\mu\text{-dppm})_2]$, a proton is incorporated at that edge [12]. Consistent with the fact that the hydride is not on the most electron-rich edge in cation **5**, ready deprotonation occurs under conditions that gave the PF_6-salts in other cases. The addition of NH_4PF_6 in methanol to a methanolic solution of the cation, followed by addition



Scheme 2.



Scheme 3.



Scheme 4.

of water, led to the precipitation of the starting cluster **1**.

Addition of a five-fold molar excess of CF_3CO_2H to a $CDCl_3$ solution of $[Ru_3(CO)_{10}(\mu\text{-dppe})]$ (**2**) at room temperature resulted in quantitative conversion to $[Ru_3(\mu\text{-H})(CO)_{10}(\mu\text{-dppe})]^+$ (**6**). The ^1H NMR spectrum at $-50°C$ gave two approximately equal intensity hydride signals: a double doublet at δ -18.52 [J(PH) 18.0 and 3.6 Hz] and a triplet at δ -20.91 [J(PH) 18.0 Hz], indicating two isomers, in a time-independent mol ratio of 0.53:0.47, which differ in the hydride sites (Scheme 3). We believe that the δ -18.52 signal is associated with **6a**, with a geometry corresponding to that of the dppm cation **5**. The hydride triplet at δ -20.91 corresponds to cation **6b** with equivalent ^{31}P nuclei. The osmium analogue of cation **6b** has been structurally characterised [5]. As in the case of cluster **5**, fluxional hydride migrations can be inferred for **6** from the 360 MHz ^1H NMR spectra shown in Fig. 2. Coalescence gives the broad singlet at δ -19.94 at 24°C; the spectrum of the same sample at 80 MHz at 24°C exhibits a sharp triplet [apparent J(PH) 9.1 Hz].

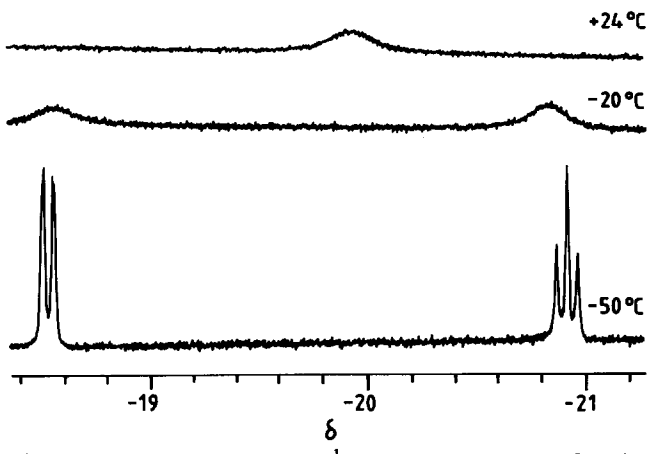


Fig. 2. Variable temperature ^1H NMR spectra of $[Ru_3(\mu\text{-H})(CO)_{10}(\mu\text{-dppe})]^+$ (**6**) in $CDCl_3$ in the hydride region.

The fluxional process can be rationalised in terms of the hydride ligand migrating over all three edges of the cluster. The interconversion of the degenerate forms of **6** can be no faster than the interconversion of **6a** and **6b**, otherwise there would be a time-averaged triplet for **6a**. Since the isomers have the mol ratio 0.53:0.47, there is a 94:53 preference for the hydride to be on an edge bridged by dppe. The Ru behaviour contrasts greatly with that for Os. Protonation of $[Os_3(CO)_{10}(\mu\text{-dppe})]$ gives initially major and minor isomers, corresponding to **6a** and **6b** respectively [5]. Then there is a rather slow conversion into a mixture dominated by the isomer corresponding to **6b**. Kinetically, protonation is preferred on the more open edge of the cluster, and hydride migration is slow for osmium. The fluxionality apparent in Fig. 2 for ruthenium implies a very much faster rate of hydride migration for that metal.

As in the protonation of $[Os_3(CO)_{10}(\mu\text{-dppp})]$, the protonation of $[Ru_3(CO)_{10}(\mu\text{-dppp})]$ (**3**) with CF_3CO_2H gives a single isomeric form of $[Ru_3(\mu\text{-H})(CO)_{10}(\mu\text{-dppp})]^+$ (**7**), in which the hydride gives a ^1H NMR triplet [δ -19.94, J(PH) 10.5 Hz] both at $-50°C$ and at room temperature, implying that there is a strong preference for the isomer with the hydride and dppp bridging the same cluster edge (Scheme 4). This cluster cation is less readily deprotonated because it may be isolated as the hexafluorophosphate salt (Experimental section). No ^1H NMR evidence for any other isomer was obtained. Protonation of $[Os_3(CO)_{10}(\mu\text{-dppb})]$ initially gave two hydrido intermediates, which are slowly converted into another species which was identified as $[Os_3(\mu\text{-H})(CO)_{10}(\mu\text{-dppb})]^+$, having the hydride and dppb bridging the same Os–Os edge. Whether such intermediates are formed in the ruthenium case is unknown, because the first ^1H NMR spectrum that could be recorded showed a single isomeric product, $[Ru_3(\mu\text{-H})(CO)_{10}(\mu\text{-dppb})]^+$ (**8**), which was isolated as the hexafluorophosphate salt.

Thus we conclude that the ring size of the bridge has a marked effect on the incorporation of proton and

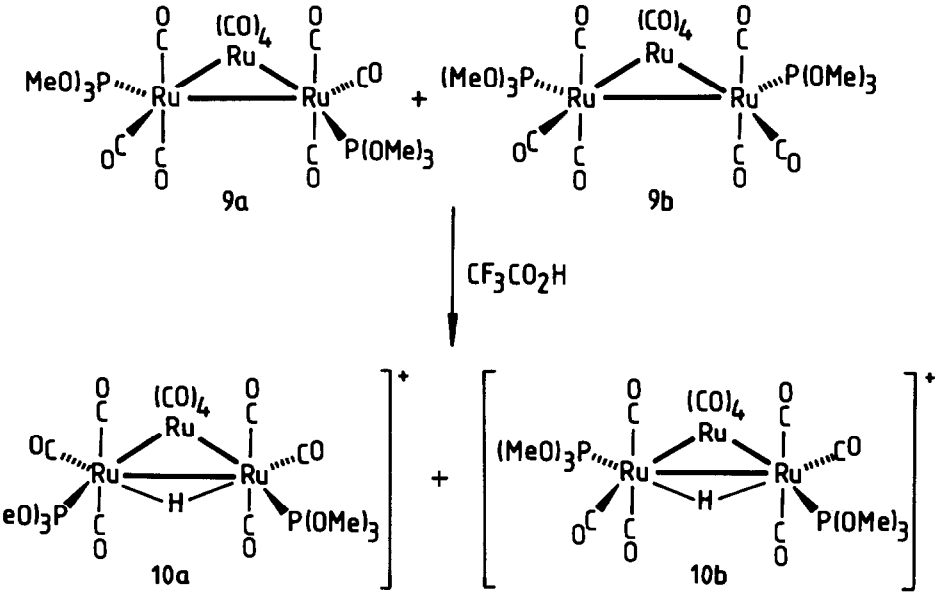
the acidity of the cation formed. Increase in the diphosphine chain length leads to greater flexibility and ease of positioning of the hydride ligand on the edge bridged by the diphosphine, which would seem to be the most electron-rich site in the cluster.

2.3. Protonation of a trimethylphosphite cluster

The cluster $[Ru_3(CO)_{10}\{P(OMe)_3\}_2]$ exists in solution as a mixture of two interconverting isomeric forms, **9a** (70%) and **9b** (30%), the major isomer having non-equivalent phosphite ligands and the minor one having equivalent ones [13]. At room temperature the $^{31}P\{^1H\}$ NMR spectrum in $CDCl_3$ is a singlet at δ 150.0 but at $-40°C$ has resolved into separate signals for the two isomers [**9a**, δ 150.9 and 150.3; **9b** δ 150.9]. The rate of isomer interconversion is greater than for osmium. This inseparable mixture reacts with a five-fold excess of CF_3CO_2H at room temperature to give two isomeric cations, $[Ru_3(\mu\text{-}H)(CO)_{10}\{P(OMe)_3\}_2]^+$, **10a** (90%) and **10b** (10%) (Scheme 5), which were characterised spectroscopically in solution. Attempted isolation led to decomposition. In $CDCl_3$ solution, the major isomer (**10a**) has equivalent phosphite ligands and its ^1H NMR spectrum contains a hydride triplet at δ -19.22 [J(PH) 8.5 Hz], the low value of J(PH) being consistent with a structure in which the phosphite and the hydride have a *cis* relationship at ruthenium as shown in Scheme 5. The minor isomer (**10b**) gives a double doublet [δ -19.69, J(PH) 15.2 (*trans*) and 7.5 (*cis*) Hz] for the hydride, showing that the phosphite ligands are non-equivalent and that these are *cis* and *trans* with respect to the hydride respectively. The $^{31}P\{^1H\}$ NMR spectrum of this mixture appeared as a singlet at δ 130.9, the signals for the separate isomers not being resolved. The minor isomer (**10b**) is derived by protonation of the major neutral species (**9a**), while the major protonated species (**10a**) has no observable neutral counterpart. The minor neutral isomer (**9b**) is not observed in its protonated form. We have discussed these preferences for different isomers on protonation for the PMe_2Ph, PPh_3 and $P(OMe)_3$ substituted osmium analogues [5]. The hexafluorophosphate salt of the PMe_2Ph complex of osmium has been structurally characterised [5]. The metal–hydride, rather than the metal–metal vector, defines the octahedral coordination directions in the protonated form. This leads to an opening of the angles between adjacent ligands on neighbouring metal atoms that are hydride bridged. This creates less crowding at the protonated metal atoms *cis* to the hydride, and this is where the phosphites predominantly reside. These are the most crowded coordination sites in the neutral precurser. Since there is rapid interconversion of neutral isomers, and probably easy interconversion of the cationic isomers, the isomer composition is thermodynamically controlled.

3. Experimental section

All reactions were carried out under nitrogen, but subsequent work-up was in air. Tetrahydrofuran (THF) was freshly distilled from sodium benzophenone ketyl



Scheme 5.

prior to use. All the diphosphines, $[Ru_3(CO)_{12}]$ and $P(OMe)_3$ were used as received from Aldrich Chemical Company. IR spectra were recorded on a PE983 or PE1420 spectrometer, NMR on an IBM NR80, Jeol GX270/89, Bruker AMX360 or a Bruker AM400 spectrometer. The clusters $[Ru_3(CO)_{10}(\mu\text{-dppm})]$ (**1**) [8] and $[Ru_3(CO)_{10}(\mu\text{-dppe})]$ (**2**) [9] were prepared by modifications of reported methods by treating $[Ru_3(CO)_{12}]$ with dppm or dppe in THF in the presence of catalytic amounts of $Na[Ph_2CO]$ [9], and were purified by TLC on silica, with hexane-dichloromethane (10:3 v/v) as eluent.

3.1. Synthesis of $[Ru_3(CO)_{10}(\mu\text{-dppp})]$ (3)

Sodium benzophenone ketyl, $Na[Ph_2CO]$, (0.025 mol cm^{-3}) was added dropwise to a stirred solution of $[Ru_3(CO)_{12}]$ (0.300 g, 0.469 mmol) in THF (50 ml) until the IR absorption for the starting ruthenium carbonyl at 2059 cm^{-1} had disappeared. The solution changed from orange to red, and after the removal of the solvent, separation of the mixture by TLC [SiO_2; eluent: hexane-CH_2Cl_2 (10:3, v/v)] gave one main band, which afforded cluster **3** as orange-red crystals (0.248 g, 52%) from a dichloromethane-pentane mixture at −20°C (Found: C, 44.7; H, 2.75; P, 6.35. $C_{37}H_{26}O_{10}P_2Ru_3$ requires C, 44.65; H, 2.65; P, 6.2%); IR ν(CO): 2073m, 2006vs, 1994s, 1951m, 1909w cm^{-1}; ^1H NMR ($CDCl_3$): δ 7.45 (Ph), 2.26 and 2.05 (CH_2); $^{31}P\{^1H\}$ NMR ($CDCl_3$): δ 22.35 (s).

3.2. Synthesis of $[Ru_3(CO)_{10}(\mu\text{-dppb})]$ (4)

A similar reaction of $[Ru_3(CO)_{12}]$ (0.300 g, 0.49 mmol) with dppb (0.200 g, 0.47 mmol) in THF (50 ml) followed by a similar chromatographic work-up gave $[Ru_3(CO)_{10}(\mu\text{-dppb})]$ (**4**) as red crystals (0.218 g, 46%) from a hexane-dichloromethane mixture at −20°C (Found: C, 45.55; H, 3.0; P, 6.35. $C_{38}H_{28}O_{10}P_2Ru_3$ requires C, 45.2; H, 2.8; P, 6.15%); IR ν(CO) ($CDCl_3$): 2074m, 2010s, 2000vs, 1985m, 1908w cm^{-1}; ^1H NMR ($CDCl_3$): δ 7.50 (Ph), 2.50 and 1.75 (CH_2); $^{31}P\{^1H\}$ NMR ($CDCl_3$): δ 20.45 (s).

3.3. Protonation of $[Ru_3(CO)_{10}(\mu\text{-dppm})]$ (1)

A red solution of $[Ru_3(CO)_{10}(\mu\text{-dppm})]$ (0.050 g, 0.052 mmol) in CD_2Cl_2 (0.5 ml) turned orange on addition of trifluoroacetic acid (0.020 ml, 0.254 mmol). The ^1H NMR and IR spectra showed that protonation to give **5** was complete; IR ν(CO) (CH_2Cl_2): 2128s, 2080s, 2053vs, 2013sh, 2005s, 1979w cm^{-1}; ^1H NMR (CD_2Cl_2): δ 7.50 (Ph), 4.34 [t, CH_2, J(PH) 10.5 Hz], −18.71 [dd, RuH, J(PH) 17.7 and 3.5 Hz]. The residue, after removal of the solvent under reduced pressure, was dissolved in methanol (5 ml) and a methanolic solution of NH_4PF_6 (0.013 g, 0.080 mmol) was added. Addition of a few drops of water precipitated the starting compound (**1**) in quantitative yield. The solvent was removed from a solution of **1** in trifluoroacetic acid, and recrystallisation of the residue from a dichloromethane-diethylether mixture at −20°C again gave cluster **1** quantitatively.

3.4. Protonation of $[Ru_3(CO)_{10}(\mu\text{-dppe})]$ (2)

The ^1H NMR spectrum of a solution of CF_3CO_2H (0.018 ml, 0.233 mmol) and cluster **2** (0.045 g, 0.046 mmol) in $CDCl_3$ (0.5 ml) indicated the complete formation of $[Ru_3(\mu\text{-H})(CO)_{10}(\mu\text{-dppe})]^+$ (**6**). Spectra were recorded in chloroform or dichloromethane. IR ν(CO) (CH_2Cl_2): 2124m, 2104m, 2081s, 2074vs, 2050sh, 2039vs, 2026s, 1996m, 1978w cm^{-1}; ^1H NMR ($CDCl_3$, −50°C): δ 7.58 (Ph), 3.76, 3.06, 2.39, 2.19 (CH_2), −20.91 [t, RuH, J(PH) 18.0 Hz], −18.52 [dd, RuH, J(PH) 18.0 and 3.6 Hz]. Attempts to isolate the hexafluorophosphate salt were unsuccessful.

3.5. Protonation of $[Ru_3(CO)_{10}(\mu\text{-dppp})]$ (3)

The ^1H NMR spectrum was recorded for a solution of CF_3CO_2H (0.020 ml, 0.254 mmol) and cluster **3** (0.050 g, 0.050 mmol) in CD_2Cl_2, which indicated complete formation of cation **7**. The solvent was removed under vacuum the residue was dissolved in methanol (8 ml), and a solution of NH_4PF_6 (0.012 g, 0.074 mmol) in a minimum of methanol was added. Addition of a few drops of water gave an orange precipitate which was recrystallised from an CH_2Cl_2/Et_2O mixture to give the cluster $[Ru_3[\mu\text{-H}(CO)_{10}(\mu\text{-dppp})]\text{-}[PF_6]$ (**7**) as orange crystals (0.043 g, 75%) (Found: C, 39.15; H, 2.5; H, 8.2. $C_{37}H_{27}F_6O_{10}P_3Ru_3$ requires C, 38.9; H, 2.4; P, 8.1%); IR ν(CO) (CH_2Cl_2): 2115w, 2073vs, 2066s, 2039vs, 2008s cm^{-1}; ^1H NMR ($CDCl_3$): δ 7.55 (Ph), 2.87 and 2.06 (CH_2), −19.94 [t, RuH, J(PH) 10.5 Hz].

3.6. Protonation of $[Ru_3(CO)_{10}(\mu\text{-dppb})]$ (4)

Similar treatment of cluster **4** (0.045 g, 0.044 mmol) and CF_3CO_2H (0.017 ml, 0.219 mmol) in CD_2Cl_2 (0.5 ml) and similar work-up gave the cluster $[Ru_3(\mu\text{-H})(CO)_{10}(\mu\text{-dppb})][PF_6]$ (**8**) as orange crystals (0.034 g, 65%) (Found: C, 39.75; H, 2.7; P, 8.15. $C_{38}H_{29}F_6O_{10}P_3Ru_3$ requires C, 39.5; H, 2.55; P, 8.0%); IR ν(CO) (CH_2Cl_2): 2015w, 2074vs, 2028sh, 2006s cm^{-1}; ^1H NMR ($CDCl_3$): δ 7.54 (Ph), 2.74 and 1.75 (CH_2), −19.93 [t, RuH, J(PH) 10.1 Hz].

3.7. Protonation of $[Ru_3(CO)_{10}\{P(OMe)_3\}_3]$ (9)

Trifluoroacetic acid (0.047 ml, 0.605 mmol) was added to a $CDCl_3$ solution (0.5 ml) of the cluster **9** (0.050 g, 0.060 mmol). The product cluster **10** could not be isolated by the method described above, but spectra

were recorded in this solvent or in dichloromethane; IR ν(CO) (CH_2Cl_2): 2139w, 2121m, 2083s, 2065s, 2047vs, 2035sh, 2000m cm^{-1}; ^1H NMR ($CDCl_3$): major isomer, δ −19.22 [t, RuH, J(PH) 8.5 Hz], minor isomer, δ −19.69 [dd, RuH, J(PH) 15.2 and 7.5 Hz], 3.82 [d, Me, J(PH) 12.2 Hz].

Acknowledgments

We thank Professor E. Rosenberg, Department of Chemistry, California State University Northridge, and Professor K.A. Azam, Department of Chemistry, Jahangirnagar University, while on leave at Oxford University, for help in obtaining NMR spectra, and the University of London Central Research Fund for support.

References

1 A.J. Deeming, B.F.G. Johnson and J. Lewis, *J. Chem. Soc. (A)*, (1970) 2517.
2 J. Knight and M.J. Mays, *J. Chem. Soc. (A)*, (1970) 711.
3 A.A. Koridze, O.A. Kizas, N.M. Astakhova, P.V. Petrovskii and Yu. K. Grishin, *J. Chem. Soc., Chem. Commun.*, (1981) 853.
4 A.J. Deeming, S. Donovan-Mtunzi, S.E. Kabir, M.B. Hursthouse, K.M.A. Malik and N.P.C. Walker, *J. Chem. Soc., Dalton Trans.*, (1987) 1869.
5 A.J. Deeming, S. Donovan-Mtunzi, K.I. Hardcastle, S.E. Kabir, K. Henrick and M. McPartlin, *J. Chem. Soc., Dalton Trans.*, (1988) 579.
6 A.J. Deeming, K.I. Hardcastle and S.E. Kabir, *J. Chem. Soc., Dalton Trans.*, (1988) 827.
7 A.J. Deeming and S.E. Kabir, *J. Organomet. Chem., 340* (1988) 359.
8 F.A. Cotton and B.E. Hanson, *Inorg. Chem., 16* (1977) 3369.
9 M.I. Bruce, T.W. Hambley, B.K. Nicholson and M.R. Snow, *J. Organomet. Chem., 235* (1982) 83.
10 A.W. Coleman, D.F. Jones, P.H. Dixneuf, C. Brisson, J.-J. Bonnet and G. Lavigne, *Inorg. Chem., 23* (1984) 952.
11 H.A. Mirza, J.J. Vittal and R.D. Puddephatt, *Inorg. Chem., 32* (1993) 1327.
12 G. Lavigne, N. Lugan and J.-J. Bonnet, *Organometallics, 1* (1982) 1040; J.A. Ladd and A.L. Balch, *Organometallics, 3* (1984) 1838.
13 S.E. Kabir, unpublished results.