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PHOSPHAALKYNES AS LIGANDS IN ORGANOMETALLIC CHEMISTRY. SYNTHESES AND ^{31}P NMR SPECTRA OF PLATINUM(II), PALLADIUM(II) AND RHODIUM(I) COMPLEXES OF DI-η^5-CYCLOPENTADIENYLTETRACARBONYL-μ-(3,3-DIMETHYL-1-PHOSPHABUTYNE)DIMOLYBDENUM, $[Mo_2(CO)_4(\eta^5\text{-}C_5H_5)_2(^tBuCP)]$

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Summary

Syntheses of the complexes *trans*-$[PtCl_2(PR_3)Mo_2(CO)_4(\eta^5\text{-}C_5H_5)_2(^tBuCP)]$, ($PR_3 = PEt_3$, PPr_3, PBu_3, PPh_2Me, $PPhMe_2$) *trans*-$[PdCl_2(PBu_3)Mo_2(CO)_4(\eta^5\text{-}C_5H_5)_2(^tBuCP)]$, and *trans*-$[RhCl\{(PF_2NMe)_2CO\}Mo_2(CO)_4(\eta^5\text{-}C_5H_5)_2(^tBuCP)]$ are described and their ^{31}P NMR spectra presented and discussed.

Introduction

The coordination chemistry of phosphaalkynes RC≡P, and phosphaalkenes $R_2C{=}PR'$ is rapidly developing [1–15]. Previously [2,4,7,14,15] we have shown that both the phosphorus–carbon π-system and the phosphorus lone pair electrons of ^{t}BuC≡P can be utilised in bonding to transition metals.

Thus ^{t}BuC≡P readily adds across the metal–metal triple bond of $[Mo_2(CO)_4(\eta^5\text{-}C_5H_5)_2]$ to yield $[Mo_2(CO)_4(\eta^5\text{-}C_5H_5)_2(^tBuCP)]$ (I) [4,14], which can then interact further as a ligand via its phosphorus lone pair with other metal carbonyl fragments e.g. $[W(CO)_5]$ [16] and $[M_3(CO)_{11}]$ (M = Ru, Os) [14], to give hetero tri- and penta-metallic complexes II and III.

We now describe an extension of this work to the syntheses of complexes of I with the d^8 transition metals rhodium(I), palladium(II) and platinum(II) and the use of ^{31}P NMR studies on such complexes to compare the relative donor properties of I with more conventional tertiary phosphine ligands R_3P.

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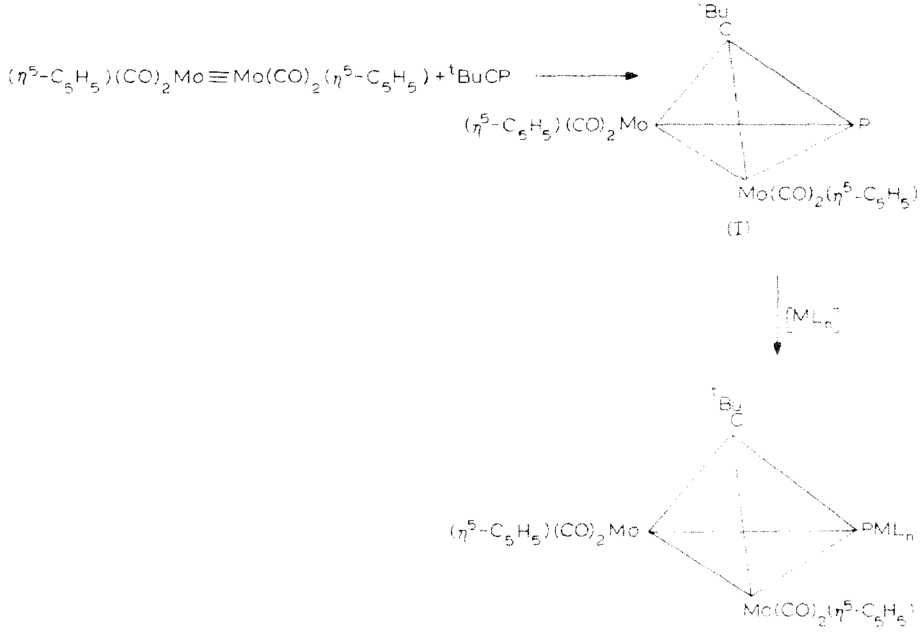


(I)

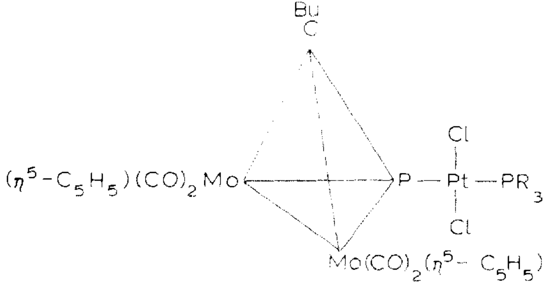
(II, $ML_n = W(CO)_5$;
III, $M = Ru_3$, $L_n = (CO)_{11}$;
$M = Os_3$, $L_n = (CO)_{11}$)

Results and discussion

(a) Platinum(II) complexes

Treatment of the dinuclear chloro-bridged platinum(II) complexes $[PtCl_2(PR_3)]_2$, ($PR_3 = PEt_3$, PPr_3, PBu_3, PMe_2Ph, $PMePh_2$), with I in THF solution readily results in the formation of the wine-red *trans*-square planar complexes IV–VIII.

Unlike the related phospha-alkene complex *trans*-$[PtCl_2(PEt_3)(P(mesityl){=}CPh_2)]$ [5] the complexes IV → VIII do not rearrange to the corresponding *cis*-isomers on treatment with hexane and this is most likely due to the increased steric bulk of I.



(IV, $PR_3 = PEt_3$;
V, $PR_3 = PPr_3$;
VI, $PR_3 = PBu_3$;
VII, $PR_3 = PMe_2Ph$;
VIII, $PR_3 = PMePh_2$)

The $^{31}P\{^1H\}$ NMR spectra of IV → VIII show the expected typical [AB] pattern of lines, (each exhibiting ^{195}Pt satellites), and the resonances of coordinated I occur ca. 50 ppm downfield of that of the free "ligand" I (See Fig. 1 and Table 1).

The values observed for ^2J(PPtP′) in complexes IV–VIII are very large (557–601 Hz) as is expected for a *trans*-isomer [17] and Table 1 also lists ^2J(PPtP′) and ^1J(PtP) coupling constant data for other *trans*-$[PtCl_2(PR_3)(P'R_3)]$ complexes for comparison.

In mixed tertiary phosphine complexes of the type *trans*-$[PtCl_2(PR_3)(P'R_3)]$ (R = alkyl, aryl), the values of ^1J(PtP) and ^1J(PtP′) lie in the 2400–2500 Hz region and ^2J(PPtP′) is ca. 470 Hz. As expected [17] replacement of one tertiary phosphine ligand by a phosphite $(RO)_3P$ is shown to lead to a marked increase in ^1J(PtP) (phosphite) coupling constant (typically ca. 4070 Hz) and a corresponding increase in ^2J(PPtP′) (ca. 710 Hz) (Table 1).

Interestingly the values of ^1J(PtP) measured in complexes IV–VIII for the coordinated cluster ligand I are unusually small, lying in the range (2020 ± 25 Hz), and are much smaller than found in other *trans*-$[PtCl_2(PR_3)(PR_3')]$ systems. Indeed ^1J(PtP) in complexes IV–VIII is only slightly larger than the values for ^1J(PtP) found for R_3P *trans* to Me in complexes of the type *cis*-$[PtMe_2(PR_3)_2]$ which are typically ca. 1850 Hz [18].

The low ^1J(PtP) values presumably reflect the unusual *s*-character of the lone pair on the phosphorus in I which is attached to two molybdenum atoms and one carbon atom. The angles at the phosphorus which are known in I and in its complexes II from single crystal X-ray crystallographic studies are much smaller than the normal CPC angles in R_3P systems (e.g. In I Mo^{1}PMo^2 75.2(0)°, Mo^{1}PC 60.4(1)°, Mo^{2}PC 61.7(1)°; in (II) Mo^{1}PMo^2 75.01(9)°, Mo^{1}PC 61.0(4)°, Mo^{2}PC 66.5(4)°). Similarly small angles at phosphorus have been observed for the related complexes $[Co_2(CO)_6(^tBuCP)W(CO)_5]$ and $[Mo_2(CO)_4(\eta^5\text{-}C_5H_5)_2P_2]$.

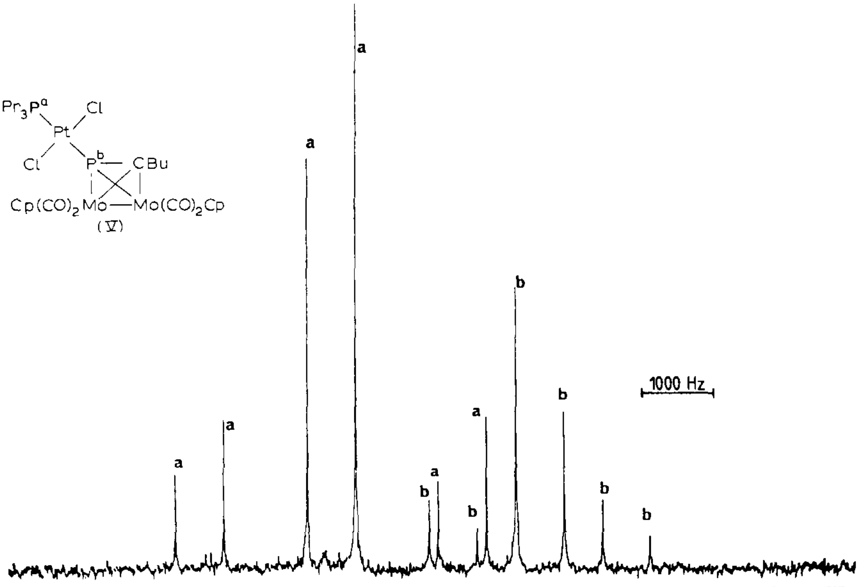


Fig. 1. $^{31}P\{^1H\}$ NMR spectrum of complex V.

TABLE 1

$^{31}P\{^1H\}$ NMR DATA FOR SOME *trans*-[$PtCl_2(PR_3)L$] COMPLEXES

Complexes	$^1J(PtP)$	$^1J(PtP_I)$ [a]	$^2J(PP_I)$ [a]	Ref.
trans-[$PtCl_2(PEt_3)\{Mo_2(\mu\text{-}^tBuCP)(CO)_4(\eta^5\text{-}C_5H_5)_2\}$] (IV) [b]	3089	1995	557	This work
trans-[$PtCl_2(PPr_3)\{Mo_2(\mu\text{-}^tBuCP)(CO)_4(\eta^5\text{-}C_5H_5)_2\}$] (V) [c]	3071	2034	562	This work
trans-[$PtCl_2(PBu_3)\{Mo_2(\mu\text{-}^tBuCP)(CO)_4(\eta^5\text{-}C_5H_5)_2\}$] (VI) [d]	3074	2033	564	This work
trans-[$PtCl_2(PMe_2Ph)\{Mo_2(\mu\text{-}^tBuCP)(CO)_4(\eta^5\text{-}C_5H_5)_2\}$] (VII) [e]	3080	2051	601	This work
trans-[$PtCl_2(PMePh_2)\{Mo_2(\mu\text{-}^tBuCP)(CO)_4(\eta^5\text{-}C_5H_5)_2\}$] (VIII) [f]	3176	2222	599	This work
trans-[$PtCl_2(PEt_3)\{P(Cl)(mesityl)(CHPh_2)\}$]	2647	2539	547	[5]
trans-[$PtCl_2(PEt_3)\{P(mesityl)=CPh_2\}$]	2844	2590	544	[5]
trans-[$PtCl_2(PBu_3)(PEt_3)$]	2409	2412	462	[19]
trans-[$PtCl_2(PBu_3)(PMe_2Ph)$]	2442	2423	483	[19]
trans-[$PtCl_2(PBu_3)(PPh_3)$]	2537	2462	473	[19]
trans-[$PtCl_2(PBu_3)\{P(OPh_3)\}$]	2516	4068	709	[20]
trans-[$PtCl_2(PEt_3)_2$]	2400	–	–	[21]
trans-[$PtCl_2(PPr_3)_2$]	2385	–	–	[21]
trans-[$PtCl_2(PBu_3)_2$]	2392	–	–	[21]
trans-[$PtCl_2(PMePh_2)_2$]	2437	–		This work
trans-[$PtCl_2\{P(OR)_3\}_2$] (R = *Ortho*-tolyl)	4405			[22]

[a] In Hz. [b] $\delta(PR_3)$ = 115.6 ppm, $\delta(P_I)$ 192.3 ppm. [c] $\delta(PR_3)$ = 130.3 ppm, $\delta(P_I)$ = 206.6 ppm. [d] $\delta(PR_3)$ = 129.6 ppm, $\delta(P_I)$ = 205.6 ppm. [e] $\delta(PR_3)$ = 146.7 ppm, $\delta(P_I)$ = 206.9 ppm. [f] $\delta(PR_3)$ = 134.7 ppm, $\delta(P_I)$ = 206.6 ppm.

The low value of ^1J(PtP) to the cluster ligand in IV–VIII is paralleled by correspondingly larger ^1J(PtP) coupling constants to the R_3P in the *trans*-position (ca. 3080 ± 10 Hz) and the ^2J(PPtP′) values (ca. 560–600 Hz) are likewise also increased, to become comparable with data observed in complexes of the type *trans*-$[PtCl_2(PEt_3)(P(mesityl){=}CPh_2)]$ and *trans*-$[PtCl_2(PEt_3)(PCl(mesityl)(CHPh_2)]$ [5] (See Table 1).

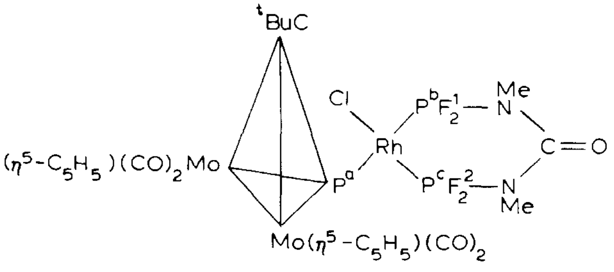
(b) Palladium(II) complexes

Treatment of I with the palladium(II) complex $[PdCl_2(PBu_3]_2$ readily yields the brown complex *trans*-$[PdCl_2(PBu_3)\{Mo_2(CO)_4(\eta^5\text{-}C_5H_5)_2(^t\text{BuCP})\}]$ (IX).

The $^{31}P\{^1H\}$ NMR spectrum of IX showed clear evidence of an intermolecular ligand exchange process at room temperature exhibiting two singlets at −103.5 and −247.5 ppm (broad). On cooling a solution of IX to −80°C the expected two sets of AB doublets (δ(P) −108.7; −206.6 ppm) were observed and the large value of ^2J(PPdP′) (573 Hz) is indicative of a *trans*-square planar complex similar to that found for IV–VIII. On raising the temperature the AB pattern gradually is replaced by the two singlets.

(c) Rhodium(I) complexes

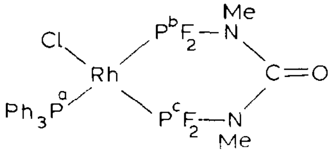
Treatment of the recently described dimeric chloro-bridge rhodium(I) complex of the urea based fluorophosphine ligand $[RhCl\{OC(NMePF_2)_2\}]_2$ [23] with I in dichloromethane results in ready bridge cleavage to produce the brick-red monomeric complex X.



(X)

Complex (X) also readily undergoes ligand exchange at room temperature but the sharp $^{31}P\{^1H\}$ NMR spectrum observed at −70°C which has been fully analysed is consistent with the formulation. The experimental and calculated $^{31}P\{^1H\}$ NMR spectra of the PF_2 region of X are shown in Fig. 2. Chemical shift and coupling constant data are listed in Table 2. In order to compare the ligand behaviour of I with PPh_3 towards Rh^I the ^{31}P NMR spectrum of the analogous complex $[RhCl\{OC(NMePF_2)_2)(PPh_3\}]$ (XI), which is also fluxional at room temperature [23] was reexamined at −50°C (Table 2).

In complex XI although the ^{31}P chemical shifts of the non-equivalent PF_2 groups



(XI)

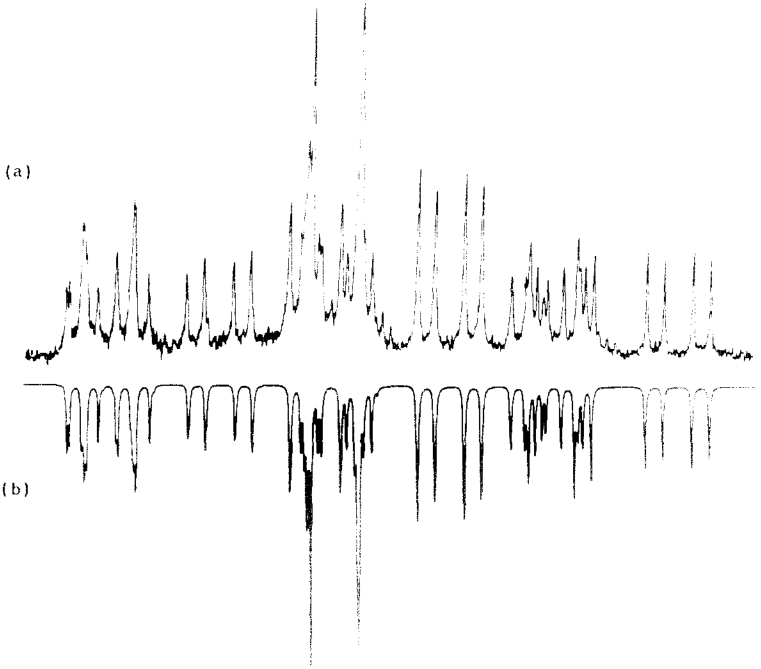


Fig. 2. Observed (a) and calculated (b) $^{31}P\{^1H\}$ NMR spectrum of the rhodium complex X.

are almost the same $\delta(P^a) - \delta(P^b) = 0.4$ ppm) and were very similar to those observed for the dimer $[RhC\{(OC(NMePF_2)_2\}]_2$ [23] the resonance of the phosphorus nucleus *trans* to the phosphaalkyne in X is shifted upfield about 10 ppm. The large values of $^2J(P^aP^b)$ in both X and XI (626 and 519 Hz. respectively) confirm that P^a and P^b are mutually *trans* and both $^2J(P^aP^b)$ and the *cis*-coupling constant $^2J(P^aP^c)$ are ca. 20% larger in X than in XI which is similar to the behaviour observed for the platinum complexes.

TABLE 2

^{31}P CHEMICAL SHIFT [a] AND COUPLING CONSTANT [b] DATA FOR COMPLEXES X AND XI [d]

	Complex X	Complex XI
$\delta(P^a)$	[242.0] [c]	116.5
$\delta(P^b)$	−22.8	−13.1
$\delta(P^c)$	−12.5	13.5
$^2J(P^aP^b)$	625.7	519.3
$^2J(P^bP^c)$	92.4	96.9
$^2J(P^aP^c)$	74.9	58.7
$^1J(P^bF)$	1221.7	1219.8
$^1J(P^cF)$	1177.4	1178.1
$^2J(P^aRh)$	[140] [c]	111.1
$^1J(P^bRh)$	247.4	213.7
$^1J(P^cRh)$	270.4	282.8

[a] In ppm (rel. TMP). [b] In Hz. [c] Estimated value. [d] For labelling of P^a, P^b and P^c see text.

Unfortunately the value of ^1J(RhP) for the coordinated ^{t}BuCP could not be accurately measured from the ^{31}P NMR spectrum of X owing to the complexity of the spectrum, however, an estimated value of ca. 140 Hz is certainly significantly larger than the corresponding value of ^1J(PRh) found for the *trans*-PPh$_3$ ligand in XI.

As expected the ^{19}F NMR spectrum of X at room temperature shows a single, rather broad 1-1 doublet centred at -41.7 ppm ($^1J(\text{PF}) \simeq 1212$ Hz), which on cooling the sample to -50°C changes into two 1-1 doublets (δ(F^2) -33.2 ppm, ^1J(PF) 1176 Hz; δ(F^1) -50.3 ppm, ^1J(PF) 1226 Hz) which are still broad enough not to allow resolution of ^2J(RhF) or other intramolecular coupling constants.

Experimental

Standard Schlenk tube techniques were used throughout. Solvents were dried and distilled prior to use. ^{1}H, ^{19}F and ^{31}P NMR spectra were obtained using a Bruker WP 80 Multinuclear Fourier Transform Spectrometer. Chemical shift data are quoted relative to TMS, CCl_3F and TMP respectively, with upfield shifts negative. Infrared spectra were recorded on a Perkin–Elmer 1430 spectrometer and elemental analyses were performed by Ms. A.G. Olney of the School of Chemistry and Molecular Sciences at Sussex University.

[$Mo_2(CO)_6(\eta^5\text{-}C_5H_5)_2$] was purchased commercially and the Pt^{II} and Pd^{II} complexes synthesised by literature methods. ^{t}BuCP was prepared by the method described in ref. 24.

Preparation of [$Mo_2(CO)_4(\eta^5\text{-}C_5H_5)_2$ (^{t}BuCP)] (I)

A solution of [$Mo_2(CO)_6(\eta^5\text{-}C_5H_5)_2$] (0.5 g, 1 mmol) in xylene (50 cm^3) was refluxed under an atmosphere of dinitrogen gas for 24 h. The resulting [$Mo_2(CO)_4(\eta^5\text{-}C_5H_5)_2$](*Mo≡Mo*) was treated with a solution of ^{t}BuCP (0.1 g, 1 mmol) in toluene (10 cm^3) at room temperature and the mixture stirred for 24 h. After removal of solvents in vacuo the residue was extracted with hexane and removal of hexane under reduced pressure gave wine-red crystals of I (0.33 g 60%) M.p. 68–70°C. Found: C, 42.04; H, 3.98. $C_{19}H_{19}Mo_2O_4P$ calcd.: C, 42.69; H, 3.55%. IR ν(CO)(hexane): 1990m, 1930s, 1850m cm^{-1}. ^{31}P: δ(P) -252.0 ppm (CH_2Cl_2), ^{1}H ($CDCl_3$): δ 1.25 (s, 9H, ^{t}Bu); 4.9 (d, 10H, 2C_5H_5) ppm.

This complex has been prepared independently by Herrmann, Becker and co-workers [25].

Preparation of trans-[$Mo_2PtCl_2(\eta^5\text{-}C_5H_5)_2(CO)_4(PEt_3)$(^{t}BuCP)] (IV)

A solution of [$PtCl_2(PEt_3)$]$_2$ (0.077 g, 0.1 mmol) in THF (5 cm^3) was treated with I (0.11 g, 0.2 mmol) in THF (5 cm^3) at room temperature. After 1 h volatile material was removed in vacuo and the residue was recrystallised from CH_2Cl_2/hexane (1/5) at -40°C to afford wine-red crystals of IV (0.12 g, 64%), m.p. 150°C. Found: C, 32.60; H, 3.93. $C_{25}H_{34}Cl_2Mo_2O_4P_2Pt$ calcd.: C, 32.67; H, 3.70%. IR ν(CO)(nujol): 1980m, 1950s, 1900s cm^{-1}. ^{1}H ($CDCl_3$): δ 1.3 (s, 9H, ^{t}Bu) 0.9–1.5 (m, 9H, 3CH_3), 1.8–2.1 (m, 6H, 3CH_2) 5.4 ppm (d, 10H, 2C_5H_5, ^3J(PH) 0.7 Hz).

Preparation of trans-[($Mo_2PtCl_2(\eta^5\text{-}C_5H_5)_2(CO)_4(PPr_3)$(^{t}BuCP)] (V)

In a similar fashion to the above [$PtCl_2(PPr_3)$]$_2$ (0.1 g, 0.12 mmol) and I (0.13 g, 0.23 mmol) gave pale brown crystals of V (0.17 g, 77%) m.p. 123–124°C. Found: C, 35.05; H, 4.40. $C_{28}H_{40}Cl_2O_4Mo_2P_2Pt$ calcd.: C, 35.0; H, 4.17%. IR ν(CO)(nujol):

2020m, 1970s, 1900m cm^{-1}. ^{1}H ($CDCl_3$): δ 1.1 (t, 9H, $3CH_3$); 1.3 (s, 9H, ^{t}Bu); 1.6–1.7 (m, 6H, $3CH_2$) 1.9–2.0 (m, 6H, $3CH_2$) 5.5 (d, 10H, $2C_5H_5$).

Preparation of trans-[$Mo_2PtCl_2(\eta^5\text{-}C_5H_5)_2(CO)_4(PBu_3)(^{t}BuCP)$] (VI)

Likewise [$PtCl_2(PBu_3)]_2$ (0.06 g, 0.06 mmol) and I (0.07 g, 0.12 mmol) afforded brown crystals of VI (0.05 g, 43%) m.p. > 240°C. Found: C, 35.0, H, 5.70. $C_{31}H_{46}Cl_2Mo_2O_4P_2Pt$ calcd.: C, 37.13; H, 4.59%. IR ν(CO)(nujol): 2110w, 1965s, 1920s, 1860w cm^{-1}. ^{1}H ($CDCl_3$): δ 1.0 (t, 9H, $3CH_3$) 1.3 (s, 9H, ^{t}Bu) 1.4–1.5 (m, 6H, $3CH_2$) 1.6–1.7 (m, 6H, $3CH_2$) 1.9–2.0 (m, 6H, $3CH_2$) 5.5 ppm (s, 10H, $2C_5H_5$).

Preparation of trans-[$Mo_2PtCl_2(\eta^5\text{-}C_5H_5)_2(CO)_4(PMe_2Ph)(^{t}BuCP)$] (VII)

Similarly [$PtCl_2(PMe_2Ph)]_2$ (0.081g, 0.1 mmol) and I gave after 1 week at −40°C wine-red crystals of VII (0.125 g, 65%) m.p. 110–112°C. Found: C, 35.69; H, 3.28. $C_{27}H_{30}Cl_2Mo_2O_4P_2Pt$ calcd.: C, 34.54; H, 3.20%. IR (ν(CO)(nujol): 1980m, 1950s, 1900s cm^{-1}. ^{1}H ($CDCl_3$): δ 1.3 (s, 9H, ^{t}Bu) 1.6–2.1 (m, 6H, 2Me, ^{2}J(PH) 11.2 Hz, ^{4}J(PH) 3.4 Hz) 5.4 (s, 10H, $2C_5H_5$) 7.3–7.9 ppm (m, 5H, C_6H_5).

Preparation of trans-[$Mo_2PtCl_2(\eta^5\text{-}C_5H_5)_2(CO)_4(PMePh_2)(^{t}BuCP)$] (VIII)

In an analogous way [$PtCl_2(PMePh_2)]_2$ (0.09 g, 0.09 mmol) and I (0.10 g, 0.19 mmol) gave after 1 h dark brown crystals of VIII (0.15 g, 82%) m.p. > 240°C. Found: C, 37.51; H, 3.49. $C_{32}H_{32}Cl_2Mo_2O_4P_2Pt$ calcd.: C, 38.40; H, 3.20%. IR ν(CO)(nujol): 1990s, 1960s, 1910m cm^{-1}. ^{1}H ($CDCl_3$): δ 1.3 (s, 9H, ^{t}Bu) 2.4 (d, 3H, CH_3, ^{2}J(PH) 12.3 Hz) 5.3 (s, 10H, $2C_5H_5$) 7.3–7.7 ppm (m, 10H, $2C_6H_5$).

Preparation of trans-[$Mo_2PdCl_2(\eta^5\text{-}C_5H_5)_2(CO)_4(PBu_3)(^{t}BuCP)$] (IX)

Likewise [$PdCl_2(PBu_3)]_2$ (0.02 g, 0.03 mmol) and I (0.03 g, 0.06 mmol) gave after 1 h brown crystals of IX (0.03 g, 61%). Found: C, 33.72; H, 4.81. $C_{31}H_{46}Cl_2Mo_2O_4P_2Pd$ calcd.: C, 35.90; H, 3.64%). IR ν(CO)(nujol): 2120w, 1950s, 1915w, 1845s cm^{-1}. ^{1}H ($CDCl_3$): δ 0.9 (t, 9H, 3Me), 1.2 (s, 9H, ^{t}Bu), 1.4–1.8 ppm (m, 18H, $9CH_2$).

Preparation of trans-[$Mo_2RhCl(\eta^5\text{-}C_5H_5)_2(CO)_4\{OC(NMePF_2)_2\}_2(^{t}BuCP)$] (X)

A solution of [$RhCl(OC(NMePF_2)_2)]_2$ (0.075 g, 0.01 mmol) in CH_2Cl_2 (10 cm^{3}) was treated with I (0.11 g, 0.021 mmol) in CH_2Cl_2 (2 cm^{3}) at room temperature. After 24 h the solution was filtered, volatiles removed in vacuo and the residue washed with hexane (3 × 5 cm^{3}) to afford brick-red crystals of X (0.16 g, 88%), m.p. > 240°C (CH_2Cl_2/hexane). Found: C, 29.41; H, 3.05; N, 3.00. $C_{22}H_{25}ClF_4Mo_2N_2O_5P_3Rh$ calcd.: C, 29.45; H, 2.79; N, 3.12%. IR ν(CO)(nujol): 1990m, 1965s, 1910s, 1690 cm^{-1}. ^{1}H ($CDCl_3$) δ 1.2 (s, 9H, ^{t}Bu) 3.07 (s, 6H, $2CH_3$) 5.2 ppm (s, 10H, $2C_5H_5$). ^{19}F ($CDCl_3$): δ(F) −41.7 ppm, ^{1}J(PF) 1212 Hz (35°C); $\delta(F^2)$ −33.2 ppm, ^{1}J(PF) 1176 Hz, $\delta(F^1)$ −50.3 ppm, ^{1}J(PF) 1226 Hz (−50°C).

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