Fluorophosphine Complexes of Ruthenium and Osmium. Part 1. Syntheses and Stereochemistry of Dihydrido-complexes of Ruthenium(II) and Osmium(II) †

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The preparation of dihydrido-complexes of the type [$MH_2L(PPh_3)_3$] and [$MH_2L_2(PPh_3)_2$] [L = PF_3, M = Ru or Os; L = $PF_2(NMe_2)$, M = Ru] and [$RuH_2(PF_3)\{PF_2(NMe_2)\}(PPh_3)_2$] is described. Hydrogen-1, ^{19}F, and ^{31}P n.m.r. spectroscopy has established that all these complexes are *cis*-dihydrides with PPh_3 ligands occupying mutually *trans* positions. No evidence has been found for either inter- or intra-molecular ligand-exchange processes in these systems.

PREVIOUSLY we have described the high catalytic activity and specificity of [$RhH(PF_3)(PPh_3)_3$] and [$RhH(PF_3)_2(PPh_3)_2$] in olefin-hydrogenation and -isomerisation reactions [1] and it was of interest to synthesise a variety of fluorophosphine-containing dihydrido-complexes of ruthenium and osmium to see whether their catalytic activity was related to the type of fluorophosphine ligand involved and/or the degree of fluorophosphine substitution.

At the beginning of this present work the only reported hydrido-complexes of Ru and Os containing trifluorophosphine were [$MH_2(PF_3)_4$] and [$MH(PF_3)_4$]$^-$ (M = Ru or Os) prepared by Kruck and Prasch.[2] The former complexes were obtained by high-temperature reduction of the metal halide with copper under a high pressure of dihydrogen and PF_3. Dihydrido-ruthenium(II) and -osmium(II) complexes containing tertiary phosphines and carbonyl ligands have been reviewed,[3,4] and synthetic routes involve reduction of dihalogeno- or halogenohydrido-metal complexes with Li[AlH_4] or Na[BH_4],[5-8] addition of hydrogen to metal(0) complexes,[9-11] or ligand substitution reactions of di- or tetra-hydrido-derivatives.[12-26] Robinson and his coworkers [27,28] have developed single-step syntheses of *cis*-[MH_2L_4] and *cis*-[$MH_2(CO)_2L_2$] (M = Ru or Os; L = PPh_3) using Na[BH_4] and aqueous formaldehyde as the source of the hydrido- and carbonyl ligands respectively.

RESULTS AND DISCUSSION

The tetrahydrido-complex [$RuH_4(PPh_3)_3$] [13,27] reacts readily with an equimolar amount of trifluorophosphine or (dimethylamino)difluorophosphine at room temperature to give high yields of the dihydrido-complexes [$RuH_2(L)(PPh_3)_3$] [L = PF_3 (1) or $PF_2(NMe_2)$ (2)]. Lower yields of (1) and (2) are obtained using the dihydrido-complex [$RuH_2(PPh_3)_4$]. Complex (1) is also the major product from the reaction of [$RuH_2(CO)(PPh_3)_3$] [28] with PF_3. The complex [$OsH_4(PPh_3)_3$] reacts with an excess of PF_3 at 60 °C to give [$OsH_2(PF_3)(PPh_3)_3$] (3), no reaction occurring at room temperature even after several days. Complexes (1)—(3) readily undergo further phosphine substitution under more vigorous conditions (100 °C) and/or with an excess of fluorophosphine to give the disubstituted products [$RuH_2(PF_3)_2(PPh_3)_2$] (4), [$RuH_2\{PF_2(NMe_2)\}_2(PPh_3)_2$] (5), and [$OsH_2(PF_3)_2(PPh_3)_2$] (6) respectively which do not undergo further substitution reactions. The mixed fluorophosphine complex [$RuH_2(PF_3)\{PF_2(NMe_2)\}(PPh_3)_2$] (7) is obtained either by treating (1) with $PF_2(NMe_2)$ or by treating (2) with PF_3. Attempts to obtain (4) and (6) by reduction of the dichloro-analogues with sodium tetrahydroborate were unsuccessful.

The new dihydrido-ruthenium and -osmium complexes are colourless solids which are quite sensitive to oxygen and light even in the solid state and are best stored under nitrogen in a refrigerator. Solutions are less stable and the thermal and air stability of the

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† No reprints available.

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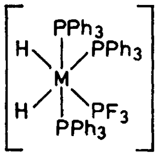
complexes increases with the degree of fluorophosphine substitution (Os > Ru).

Stereochemistry.—The presence of hydrogen, fluorine, and phosphorus nuclei in the above complexes enables unambiguous assignment of their stereochemistry using ^{1}H, ^{19}F, and ^{31}P n.m.r. spectroscopy.

(*a*) *Monofluorophosphine complexes* (1)—(3). The ^{1}H n.m.r. spectra of (1) and (3) are similar, exhibiting two



(1) M = Ru
(3) M = Os

separate high-field hydride resonances, indicating that they have the structure shown above. Decomposition of (2) in solution precluded observation of the hydride resonance. The lower-field hydride resonance of (1) shown in Figure 1, which appears as a widely spaced doublet, is assigned as arising from coupling to the *trans*-trifluorophosphine ligand, and the magnitude of the coupling constant is comparable with data reported for the complexes *cis*-$[MH_2(PF_3)_4]$.[2] The high-field resonance assigned to the hydride ligand *trans* to a PPh_3 ligand exhibits a similar coupling constant to the analogous carbonyl complex.[27] A complete analysis of the ^{1}H n.m.r. spectrum of (1) is facilitated by fluorine- and phosphorus-decoupling experiments, the results of which are summarised in Table 1.

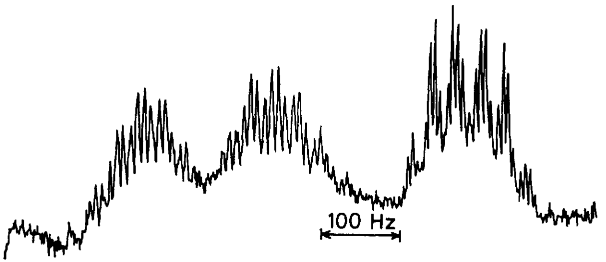


FIGURE 1 Hydrogen-1 n.m.r. hydride resonances of *cis*-$[RuH_2(PF_3)(PPh_3)_3]$ (1)

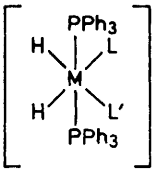
TABLE 1

High-field ^{1}H n.m.r. chemical-shift and coupling-constant data [a]

	Complex	τ	$^{2}J(HMP_{trans})$ [b]	$^{2}J(HMPPh_{3(ax)})$ [b]	$^{2}J(HMH')$ [b]	$^{3}J(HMPF)$ [b]
(1)	*cis*-$[RuH_2(PF_3)(PPh_3)_3]$	15.6 (*trans* PF_3) 19.1 (*trans* PPh_3)	177.0 66.0	27.1 23.5	6.5	36.0 (*trans*) 5.5 (*cis*)
(4)	*cis*-$[RuH_2(PF_3)_2(PPh_3)_2]$	16.6	145.3	23.2		41.5 [c]
(5)	*cis*-$[RuH_2\{PF_2(NMe_2)\}_2(PPh_3)_2]$ [e]	18.0	105.6	26.3		23.0 [c]
(7)	*cis*-$[RuH_2(PF_3)\{PF_2(NMe_2)\}(PPh_3)_2]$ [f]	16.4 (*trans* PF_3) 17.7 [*trans* $PF_2(NMe_2)$]	173.0 122.5	*ca.* 29.1 *ca.* 24.5		34.2 (*trans* PF_3) [d] 5.7 (*cis* PF_3)
(3)	*cis*-$[OsH_2(PF_3)(PPh_3)_3]$	17.6 (*trans* PF_3) 20.5 (*trans* PPh_3)	141.0 48.2	25.4 17.0	4.5	34.2 (*trans*) 4.0 (*cis*)
(6)	*cis*-$[OsH_2(PF_3)_2(PPh_3)_2]$	18.0	133.0	23.4		37.8 [c]

[a] In $CDCl_3$. [b] In Hz. [c] $^{3}J(HMPF_{trans} + HMPF_{cis})$. [d] Data from ^{19}F n.m.r. spectrum [coupling to $PF_2(NMe_2)$ not resolved]. [e] $\tau(NMe_2)$ 7.91; $^{3}J(PNCH)$ 9.75, $^{4}J(FPNCH)$ 3.8 Hz. [f] $\tau(NMe_2)$ 7.80; $^{3}J(PNCH)$ 9.8, $^{4}J(FPNCH)$ 3.0 Hz.

Further confirmation of the proposed stereochemistry comes from ^{31}P n.m.r. studies on (1) and (3). The ^{31}P-$\{^{1}H\}$ n.m.r. spectra consist of a low-field trifluorophosphine resonance, relative intensity 1, and two higher-field triphenylphosphine resonances of relative intensity 2 : 1 which are assigned to the axial and equatorial ligands. Phosphorus chemical-shift and coupling-constant data are recorded in Table 2. The PF_3 resonances of (1) and (3) appear as widely spaced quartets [$^{1}J(PF)$] and each line shows a further quartet splitting from the fortuitous overlap of a doublet [$^{2}J(PMP'_{eq.})$] of triplets [$^{2}J(PMP'_{ax.})$]. The PPh_3 resonances in the ^{31}P n.m.r. spectra of (1) and (3) show a complicated pattern of lines making analysis of the spectra difficult; however, the ^{31}P-$\{^{1}H\}$ spectrum of (1) has been analysed using the L.A.M.E. computer program which is a modification of the iterative LAOCOON program developed by Castellano and Bothnerby.[29,30] The observed and computed spectra are shown in Figure 2. Fluorine-19 n.m.r. spectra (Table 3) confirm the assignment for (1) and (3), and (2) is also assigned a similar stereochemistry on the basis of its ^{31}P and ^{19}F n.m.r. spectra.



(4) M = Ru, L = L′ = PF_3
(5) M = Ru, L = L′ = $PF_2(NMe_2)$
(6) M = Os, L = L′ = PF_3
(7) M = Ru, L = PF_3, L′ = $PF_2(NMe_2)$

(*b*) *Bis*(*fluorophosphine*) *complexes* (4)—(7). The disubstituted complexes (4)—(6) are $[AMX_n]_2$ (n = 2 or 3) spin systems[31] (A = H, M = P, X = F). The high-field ^{1}H n.m.r. spectra of (4)—(6) exhibit a complicated symmetric doublet pattern indicating that both hydride ligands are chemical-shift equivalent, while the large coupling constant to phosphorus, $^{2}J(PMH)$, suggests that the fluorophosphine ligands are *trans* to the two hydrides. Irradiation at the resonance frequencies of the phosphorus and fluorine nuclei of the PF_3 ligands in (4) collapses the doublet pattern to a simple 1 : 2 : 1 triplet [$^{2}J(PRuH_{cis})$], while irradiation only at the resonance frequency of the fluorine nuclei changes the spectrum to the expected widely spaced doublet of triplets. These observations are only consistent with (4) having a *cis*-dihydride structure shown below in which the two PPh_3 ligands occupy axial positions on the octahedron. We recently established a similar type of structure for the

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TABLE 2

Phosphorus-31 n.m.r. chemical-shift and coupling-constant data

Complex	$\delta(PF_3)$ [a]	$\delta(PF_2NMe_2)$ [a]	$\delta(PPh_3)$ [a]	$^2J[P(F)MPPh_3]$ [b]
(1) [c,d]	11.4		84.2(2) 92.8(1)	28.3
(2) [c]		−50.0	69.4(2) 79.5(1)	23.3
(4) [c]	4.7		85.3	29.2
(5) [c]		−49.5	66.9	24.4
(7) [c,e]	−6.2	−48.5	69.9	27.5 (PF_3) [f] 25.2 (PF_2NMe_2) [g]
(3) [g]	50.5		122.4(2) 124.2(1)	17.1
(6) [h]	42.5		121.9	18.5

[a] P.p.m. upfield from $P(OMe)_3$. [b] In Hz. [c] In $CDCl_3$. [d] $^2J(PPh_3RuPPh_3)_{cis}$ 17.4 Hz. [e] $^2J(FPRuP'F)$ 47.5 Hz. [f] $^3J(FPRuPF)$ 18.4 Hz. [g] $^3J(FPRuPF)$ 12.4 Hz. [h] In CH_2Cl_2.

TABLE 3

Fluorine-19 n.m.r. chemical-shift and coupling-constant data [a]

Complex	$\phi(PF_3)$ [b]	$\phi(PF_2NMe_2)$ [b]	$^1J(PF_3)$ [c]	$^1J(PF_2)$ [c]	$^3J(FPMPPh_3)$ [c]
(1)	4.8		1 285		5.8
(2)		24.5		1 094	
(4)	6.8		1 291 [d]		5.1
(5)		30.8		1 082	6.0
(7)	6.2	27.9	1 300	1 096	5.7 (PF_3)
(3)	7.4		1 248		*ca.* 2
(6)	8.3		1 296 [d]		*ca.* 2

[a] In $CDCl_3$. [b] P.p.m. upfield from $CFCl_3$. [c] In Hz. [d] $^1J(PF) + {}^3J(PMPF')$. $^2J(PMP')$ is small and could not be accurately determined.

related dichloro-complex *cis*-$[RuCl_2(PF_3)_2(PPh_3)_2]$ by *X*-ray crystallography.[32] Complexes (5) and (6) are assigned a similar stereochemistry, as is the mixed fluorophosphine complex (7) although overlap of lines has prevented a complete analysis of its ^1H n.m.r. spectrum. Hydrogen-1 chemical-shift and coupling-constant data are listed in Table 1.

Support for the stereochemical assignments comes from the ^{31}P-$\{^1H\}$ n.m.r. spectra of (4)—(6) which exhibit the expected low-field quartet [(4) and (6)] or triplet [(5)] patterns, fluorophosphine resonances [$^1J(PF)$], and a high-field overlapping doublet of doublets for the PPh_3 ligands. Chemical-shift and coupling-constant data are listed in Table 2. The proton-undecoupled spectra show a further large doublet pattern [$^2J(PMH_{trans})$] for the fluorophosphine resonance, confirming that both fluorophosphine ligands are *trans* to hydride ligands. For (4) and (6) a further smaller doublet splitting is also observed from coupling to the *cis*-hydride ligand. The ^{31}P-$\{^1H\}$ spectrum of (7) exhibits the expected resonances for the three types of phosphine ligands. There is considerable overlapping of lines but a complete analysis of the spectrum has been made using the L.A.M.E. computer program (Table 2). Fluorine-19 n.m.r. spectra provide further confirmation of the structures, and chemical-shift and coupling-constant data are summarised in Table 3.

The *cis*-dihydride stereochemistry established for complexes (1)—(6) is similar to that assigned to the complexes $[MH_2L_4]$ ($M = Ru$ or Os; $L = CO$ or PF_3) on the basis of vibrational spectroscopy.[2,17] Complexes of the type $[RuH_2(PR_3)_4]$ [$PR_3 = PPh(OMe)_2$, $PPh(OEt)_2$, or $PPh(OPr^i)_2$], exist as both *cis* and *trans* isomers,[33] while *trans* complexes are known when chelating ditertiary phosphines are used.[34,35]

Infrared spectroscopy may be inconclusive in certain cases in assigning stereochemistry in this type of complex; for example, although the i.r. spectra of (1)—(3), (6), and (7) exhibit the expected two metal–hydrogen stretching bands in the 1 800—2 150 cm^{-1} region only one band is resolved for complexes (4) and (5).

No evidence was found for intramolecular phosphine exchange or hydrogen ' tunnelling ' in these complexes, and the ^{19}F n.m.r. spectra of (1), (4), and (5) remained unchanged over the temperature range −50 to 80 °C. This contrasts with the fluxional behaviour established previously for the structurally related complexes $[FeH_2L_4]$ [$L = PPh(OEt)_2$, PPh_2H, and $P(OEt)_3$],[5,36-38] $[FeH_2(CO)(PR_3)_3]$,[38] and $[RuH_2(PMePh_2)_4]$ at 100 °C,[16] although $[RuH_2(CO)(PMePh_2)_3]$ is stereochemically rigid.[5]

Unlike certain rhodium(I) trifluorophosphine complexes,[39,40] no intermolecular ligand exchange occurs in the ruthenium complexes as evidenced by ^{19}F and ^{31}P n.m.r. spectroscopy.

EXPERIMENTAL

Reactions were carried out in sealed evacuated Pyrex-glass tubes using a high-vacuum system. Solvents were rigorously dried and redistilled before use. Elemental

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analyses were by Mr. and Mrs. A. G. Olney of this department. Trifluorophosphine was obtained from Ozark-Mahoning Co., carefully fractionated *in vacuo* before use, and its purity checked by molecular-weight and i.r. measurements. Hydrogen-1, ^{31}P, and ^{19}F n.m.r. spectra were recorded on a JEOL PFT 100 spectrometer, i.r. spectra on a Perkin-Elmer PE 157 spectrometer.

Although $[RuH_4(PPh_3)_3]$ has been prepared by the reaction of $[RuH_2(N_2)(PPh_3)_3]$ with molecular hydrogen [10] or by reduction of $RuCl_3$ in the presence of PPh_3,[7] we find that treatment of $[RuCl_2(PPh_3)_3]$ with $Na[BH_4]$ in ethanol gives high yields of an analytically pure product. Other workers [41,42] have also recently utilised this synthetic route with methanol as solvent.

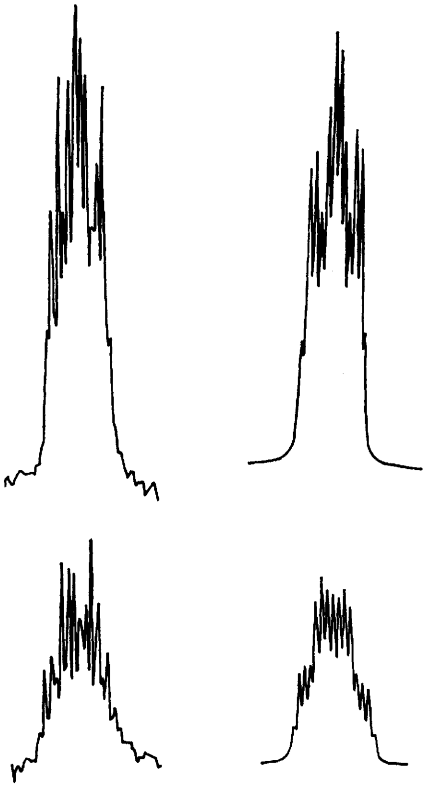
(*a*) (*b*)

FIGURE 2 Comparison of recorded (*a*) and computed (*b*) ^{31}P n.m.r. triphenylphosphine resonances of *cis*-$[RuH_2(PF_3)(PPh_3)_3]$ (1)

Preparations.— *Tetrahydridotris*(*triphenylphosphine*)*ruthenium*(IV). A solution of $Na[BH_4]$ (0.20 g, 5.3 mmol) in ethanol (20 cm^3) was added to a suspension of $[RuCl_2(PPh_3)_3]$ (1.27 g, 1.32 mmol) in ethanol (120 cm^3) at room temperature. The resulting precipitate was filtered off, washed with methanol (50 cm^3), water (100 cm^3), and methanol (100 cm^3), and dried *in vacuo* to give the white microcrystalline *complex* (0.610 g, 1.19 mmol, 90%), m.p. 130 °C (decomp.) (Found: C, 71.5; H, 5.6. Calc. for $C_{54}H_{49}P_4Ru$: C, 72.8; H, 5.5%). Infrared spectrum in Nujol mull: 1 950 cm^{-1} [ν(Ru–H)] (lit.,[28] 1 950 cm^{-1}).

cis-*Dihydrido*(*trifluorophosphine*)*tris*(*triphenylphosphine*)*ruthenium*(II) (1). (*i*) *From* $[RuH_4(PPh_3)_3]$. A mixture of $[RuH_4(PPh_3)_3]$ (0.500 g, 0.56 mmol), PF_3 (0.0524 g, 0.595 mol), and benzene (25 cm^3) was sealed off in an ampoule and shaken at room temperature for 16 h. After removal of solvent the resulting oil was washed with hexane (10 cm^3) and recrystallisation from benzene–hexane gave colourless crystals of the *complex* isolated as the benzene solvate (0.512 g, 0.49 mmol, 86%), m.p. 171 °C (decomp.) (Found: C, 68.8; H, 5.5. $C_{60}H_{53}P_4Ru$ requires C, 68.3; H, 5.0%). Infrared spectrum in Nujol: 3 060w, 1 970m, 1 944w, 1 586w, 1 480w, 1 432m, 1 187w, 1 160w, 1 090s, 1 032w, 1 002w, 904w, 834vs, 801vs, 758w, 745vs, 700vs,br, 627w, 611w, 525vs,br, 492w, 462w, 447w, 430w, and 420vw cm^{-1}.

(*ii*) *From* $[RuH_2(PPh_3)_4]$. Similarly $[RuH_2(PPh_3)_4]$ (0.963 g, 0.92 mmol) and PF_3 (0.087 g, 0.99 mmol) in benzene (20 cm^3) gave (1) after repeated recrystallisation from dichloromethane–hexane (0.300 g, 0.31 mmol, 33.7%), m.p. 170 °C (decomp.) (Found: C, 65.0; H, 4.9. $C_{54}H_{47}F_3P_4Ru$ requires C, 66.3; H, 4.8%).

(*iii*) *From* $[RuH_2(CO)(PPh_3)_3]$. Similarly, $[RuH_2(CO)(PPh_3)_3]$ and PF_3 in benzene (5 cm^3) gave (1) as the major product (by ^{19}F n.m.r. spectroscopy).

The following *complexes* were obtained by a similar procedure from $[RuH_4(PPh_3)_3]$ or $[OsH_4(PPh_3)_3]$.

cis-$[RuH_2\{PF_2(NMe_2)\}(PPh_3)_3]$, (2), colourless crystals from benzene–hexane, 86%, m.p. 121 °C (decomp.) (Found: C, 66.5; H, 5.1; N, 1.4. $C_{55}H_{53}F_2NP_4Ru$ requires C, 67.1; H, 5.3; N, 1.4%). Infrared spectrum in Nujol: 3 050w, 1 972w, 1 880w, 1 585w, 1 570vw, 1 480w, 1 435m, 1 300w, 1 189m,br, 1 155vw, 1 086s, 1 030w, 1 000w, 948s, 769s, 755w, 749s, 740w, 724w, 708w, 695vs, 680vs, 635w, 620w, 544w, 520vs,br, 459w, 450w, 440w, and 424w cm^{-1}.

cis-$[RuH_2(PF_3)_2(PPh_3)_2]$ (4), colourless crystals from CH_2Cl_2–hexane, 92%, m.p. 169—170 °C (decomp.) (Found: C, 53.8; H, 3.5. $C_{36}H_{32}F_6P_4Ru$ requires C, 53.8; H, 4.0%). Infrared spectrum in Nujol: 3 060w, 1 884w, 1 589vw, 1 572vw, 1 483w, 1 436m, 1 190w, 1 163w, 1 093m, 1 077vw, 1 031w, 1 001w, 876s, 846s,br, 827vs,br, 812s,br, 800s, 755m, 748m, 720w, 699s,br, 621w, 556w, 548w, 520vs,br, 447w, 430w, 413w, 408w, and 388w cm^{-1}; a sample recrystallised from benzene–hexane exhibited two Ru–H stretching modes at 1 965w and 1 889w cm^{-1}.

cis-$[RuH_2\{PF_2(NMe_2)\}_2(PPh_3)_2]$ (5), colourless crystals from benzene–hexane, 91%, m.p. 184—185 °C (decomp.) (Found: C, 57.0; H, 5.2; N, 3.2. $C_{40}H_{44}F_4N_2P_4Ru$ requires C, 56.3; H, 5.2; N, 3.2%). Infrared spectrum in Nujol: 3 059w, 1 855m,br, 1 588w, 1 571w, 1 480w, 1 435w, 1 309m, 1 190s, 1 159vw, 1 090s, 1 074w, 1 029w, 1 000w (sh), 990vs,br, 819vs, 783vs, 750vs, 722w, 710vs, 700vs, 687w, 621w, 549w, 525vs,br, 460w, 447w, 430w, and 420vw cm^{-1}.

cis-$[OsH_2(PF_3)(PPh_3)_3]$ (3), colourless crystals from CH_2Cl_2–hexane, 71%, m.p. 198 °C (decomp.) (Found: C, 61.2; H, 4.6. $C_{54}H_{47}F_3OsP_4$ requires C, 60.8; H, 4.4%). Infrared spectrum in Nujol: 3 059w, 2 051m, 2 030w, 1 589w, 1 571vw, 1 482w, 1 435m, 1 189w, 1 160vw, 1 093m, 1 080vw, 1 033w, 1 004w, 890vw, 856m, 843vs, 824s, 810s, 801s, 758m, 751m, 747vs, 740w, 735w, 704vs, 697s, 690w, 630w, 617w, 530vs, 517m, 494w, 471w, 465w, 448w, 432w, and 389w cm^{-1}.

cis-$[OsH_2(PF_3)_2(PPh_3)_2]$ (6), colourless crystals from CH_2Cl_2–hexane gave the 2 : 1 dichloromethane solvate, 94%, m.p. 185—187 °C (Found: C, 46.9; H, 3.6. $C_{36}H_{33}ClF_6OsP_4$ requires C, 46.95; H, 3.6%). Infrared spectrum in Nujol: 3 055w, 2 070w, 1 975w, 1 583vw,

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1 570vw, 1 480w, 1 435m, 1 187w, 1 161w, 1 092m, 1 073w, 1 029w, 1 000m, 920vw, 893vw, 882vw, 851s, 830vs,br, 753s, 744m, 702m, 697s, 618vw, 561w, 553w, 528vs, 516m, 453w, 441w, 435w, 386w, and 257w cm^{-1}.

cis-[*(Dimethylamino)difluorophosphine*]*dihydrido*(*trifluorophosphine*)*bis*(*triphenylphosphine*)*ruthenium*(II) (7). Complex (1) (0.444 g, 0.45 mmol) and $PF_2(NMe_2)$ (0.056 g, 0.49 mmol) in benzene (10 cm^3) at room temperature for several hours gave, following removal of solvent, an oil which after washing with hexane (10 cm^3) and recrystallisation from benzene–hexane gave colourless *crystals* (0.306 g, 0.37 mmol, 82%), m.p. 175—176 °C (decomp.) (Found: C, 55.8; H, 4.7; N, 1.7. $C_{38}H_{38}F_5NP_4Ru$ requires C, 55.1; H, 4.6; N, 1.7%). Infrared spectrum in Nujol: 3 079vw, 3 060w, 1 925w, 1 885w, 1 587w, 1 573vw, 1 482w, 1 437m, 1 313m, 1 190s, 1 161w, 1 095s, 1 075w, 1 030w, 998vs, 923w, 850vs, 830vs, 823vs, 783w, 760w, 752m, 744m, 732wm, 720s, 700vs, 640w, 620w, 550w, 525vs, 515vs, 465w, 450w, 529w, 391w, and 328w cm^{-1}.

In a similar way, (7) was obtained in 60% yield from the reaction of (2) and an equimolar amount of PF_3.

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