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Platinum-metal Complexes containing Four or Five Diphenylphosphine Ligands

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Convenient syntheses of the complexes *trans*- and *cis*-$[MCl_2(HPPh_2)_4]$ (M = Ru or Os) and *cis*-$[ReCl_2(PPh_2)(HPPh_2)_3]$, and of salts of the $[Rh(HPPh_2)_4]^+$, $[Ir(CO)(HPPh_2)_4]^+$, $[RuH(HPPh_2)_5]^+$, and *trans*-$[RuH(CO)(HPPh_2)_4]^+$ cations are described. The structures and properties of these complexes, including the formation of *cis*-$[OsH_2(HPPh_2)_4]$, are discussed.

ALTHOUGH the preparations of secondary phosphine complexes of the platinum metals have been carried out by many workers,[1-4] the reactions of secondary phosphines with simple salts and complexes of these metals have not been as extensively studied as have those of tertiary phosphines. This paper describes convenient syntheses of some mononuclear complexes of Group VIII metals and rhenium which each contain four or five diphenylphosphine ligands. These complexes have been prepared in order to compare their structure and chemistry with related tertiary and ditertiary phosphine complexes, and to investigate their use as homogeneous catalysts for hydrocarbon reactions.

Neutral Complexes.—Ammonium hexachloro-osmate reacts with diphenylphosphine to give *trans*-dichlorotetrakis(diphenylphosphine)osmium(II), the osmium analogue of *trans*-dichlorotetrakis(diphenylphosphine)ruthenium(II).[2] The binuclear complexes $[M_2Cl_3(PMePh_2)_6]Cl$ ($M = Ru$ or Os), react with diphenylphosphine to give *cis*-dichlorotetrakis(diphenylphosphine)-ruthenium and -osmium. This method follows the course of preparation of the ditertiary phosphine complexes *cis*-$[MCl_2(Et_2PCH_2CH_2PEt_2)_2]$.[5] Oxotrichlorobis(triphenylphosphine)rhenium(V) reacts with diphenylphosphine to give the complex *cis*-dichloro(diphenylphosphido)tris(diphenylphosphine)rhenium(III).

The complexes are monomeric as is shown by their molecular weights in chloroform and the absence of conductivity in acetone and nitrobenzene. Their i.r. spectra show bands in the 2350—2300 cm^{-1} region assignable to ν_{P-H}. They are diamagnetic; to account for this the rhenium complex is assumed to contain one diphenylphosphido (PPh_2) ligand. The ^1H n.m.r. spectrum of this complex does not contain any signal which

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[1] K. Issleib and E. Wenschuh, *Z. anorg. Chem.*, 1960, **305**, 15; K. Issleib and G. Schwager, *ibid.*, 1961, **310**, 43; 1961, **311**, 83.

[2] R. G. Hayter, *J. Amer. Chem. Soc.*, 1962, **84**, 3046; *Inorg. Chem.*, 1963, **2**, 309; 1964, **3**, 301.

[3] J. Chatt and J. M. Davidson, *J. Chem. Soc.*, 1964, 2433.

[4] W. Hieber and R. Kummer, *Ber.*, 1967, **100**, 148; W. Hieber and W. Opavsky, *ibid.*, 1968, **101**, 2966.

[5] J. Chatt and R. G. Hayter, *J. Chem. Soc.*, 1961, 896.

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can be assigned to a proton bound to rhenium, and so it appears unlikely that the complex can have the structure $[ReHCl_2(HPPh_2)_4]$.

The *trans*- and *cis*-configurations of the isomers of $[RuCl_2(HPPh_2)_4]$ may be assigned on the basis of their dipole moments, measured in benzene. The *trans*-isomer has a moment of less than 2·5 D, the *cis*-isomer one of 11—12 D. These figures are comparable to those found for complexes such as *trans*- and *cis*-$[RuCl_2(Et_2PCH_2CH_2PEt_2)_2]$.[5]

The dipole moments of the ruthenium complexes provide evidence for the configurations of their osmium analogues which have identical i.r. spectra except in the 320—250 cm^{-1} region, where bands assigned to ν_{M-Cl} are shifted to lower frequencies. The spectra of the *trans*-complexes contain one band assigned to ν_{M-Cl} in this region while those of the *cis*-complexes contain two bands.[6a] Since the spectrum of the rhenium complex also shows two bands assigned to ν_{Re-Cl} in this region, the complex is tentatively assigned a *cis*-configuration.

The complexes are yellow solids, which are stable to air and soluble in dichloromethane, moderately soluble in benzene and acetone, and insoluble in alcohols and water. *trans*-$[RuCl_2(HPPh_2)_4]$ is more soluble than its *cis*-isomer in benzene but less soluble in chloroform. It does not react in benzene with hydrogen or carbon monoxide at atmospheric pressure and room temperature or with aldehydes, acetonitrile, or triphenylphosphine at 80°.

The ^1H n.m.r. spectra of the *trans*-isomers of both ruthenium and osmium in deuteriobenzene or deuteriochloroform show only signals assignable to phenyl protons. The spectrum of *cis*-$[RuCl_2(HPPh_2)_4]$ in deuteriochloroform shows a large complex signal centred at τ 2·75 which may be assigned to phenyl protons, and three small broad signals at τ 1·55, 5·30, and 6·30. The ratio of the sum of the intensities of these broad signals to that of the large signal is approximately 3 : 40, so, if it is assumed that a further small signal overlaps the large complex signal, the small signals may be assigned to the protons on phosphorus atoms. These signals may be broadened by coupling of the protons with distant phosphorus nuclei but they are too small to be resolved satisfactorily and are of no help in establishing the configuration of the complex. Its osmium analogue has a similar spectrum.

Both *trans*- and *cis*-isomers of $[OsCl_2(HPPh_2)_4]$ react with sodium dihydridobis(2-methoxyethoxy)aluminate giving the white complex *cis*-dihydridotetrakis(diphenylphosphine)osmium(II). The i.r. spectrum of this dihydride in dichloromethane contains a band at 1929 cm^{-1} assigned to ν_{Os-H}, which is split into two bands in the solid state. This position is characteristic of ν_{M-H} in the spectra of *cis*-dihydrido-octahedral complexes; the spectra of *trans*-dihydrido-complexes contain bands assigned to ν_{M-H} in the 1750—1600 cm^{-1} region.[6b]

The ^1H n.m.r. spectrum of this dihydride in dichloromethane contains a complex signal with a centre of symmetry at τ 19·03 which may be assigned to the protons bound to osmium. Ten broad overlapping lines may be distinguished in this spectrum; their positions with approximate relative intensities are at τ 18·48 (1), 18·66 (1), 18·71 (2), 18·87 (2), 18·98 (2), 19·08 (2), 19·19 (2), 19·35 (2), 19·40 (1), 19·58 (1).

If the two central lines are each assumed to consist of two superimposed lines of relative intensity 1, it is possible to regard this signal as four overlapping 1–2–1 triplets, produced by coupling of each proton with the *trans*-phosphorus nucleus and with one unique and two equivalent *cis*-phosphorus nuclei. However the spectrum resembles the published spectrum of *cis*-$[RuH_2(PMePh_2)_4]$ where the phosphorus and hydride spins are treated as an $AA'PP'X_2$ system,[7] and the spectrum of *cis*-$[OsH_2(HPPh_2)_4]$ is better explained thus. The broadness of the lines may be due to further splitting of the signals by coupling of the protons on osmium with the protons on the phosphorus atoms.

The ruthenium analogue of this compound could not be obtained pure from a similar preparation, and no hydrido-complexes were formed when *trans*-$[RuCl_2(HPPh_2)_4]$ was treated with hydrogen in the presence of triethylamine or with hydrazine hydrate.

Ionic Complexes.—Rhodium trichloride and ammonium hexachloroiridate(IV) react with diphenylphosphine to give the complexes $[MCl_3(HPPh_2)_3]$ (M = Rh, Ir).[2,8] However, chlorotris(triphenylphosphine)rhodium(I) and *trans*-chlorocarbonylbis(triphenylphosphine)rhodium(I) react with diphenylphosphine to give the ionic complex tetrakis(diphenylphosphine)rhodium(I) chloride. The $[Rh(HPPh_2)_4]^+$ cation may be isolated as its tetrafluoroborate, hexafluorophosphate, and perchlorate salts. Similarly, *trans*-$[IrCl(CO)(PPh_3)_2]$, $[RuHCl(PPh_3)_3]$, and $[RuHCl(CO)(PPh_3)_3]$ react with diphenylphosphine to give salts of the cations $[Ir(CO)(HPPh_2)_4]^+$, $[RuH(HPPh_2)_5]^+$, and *trans*-$[RuH(CO)(HPPh_2)_4{}^+$ respectively.

The i.r. spectra of these salts show bands assignable to ν_{P-H} in the 2400—2300 cm^{-1} region. The position of these bands in solid-state spectra varies only slightly with variation of the anion in the salt. The ^1H n.m.r. spectrum in dichloromethane of $[RuH(HPPh_2)_5]^+PF_6{}^-$ contains a signal centred at τ 17·89 consisting of two overlapping 1 : 4 : 6 : 4 : 1 quintets. This is assigned to the proton bound to ruthenium which is coupled to the *trans*-phosphorus nucleus (J_{P-H} 72 Hz) and to the four equivalent *cis*-phosphorus nuclei (J_{P-H} 19 Hz). A saturated solution of *trans*-$[RuH(CO)(HPPh_2)_4]^+BF_4{}^-$ in dichloromethane gives a spectrum containing a quintet signal centred at τ 16·79. This is assigned to the proton bound to ruthenium which is coupled to four *cis*-phosphorus nuclei (J_{P-H} 20 Hz).

These salts are yellow (Rh and Ir) or white (Ru) air-stable solids which are soluble in acetone or chlorinated solvents but insoluble in benzene or alcohols. The

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[6] (*a*) D. M. Adams, 'Metal–Ligand and Related Vibrations,' Arnold, London, 1967, p. 44; (*b*) p. 6.

[7] K. C. Dewhirst, W. Keim, and C. A. Reilly, *Inorg. Chem.*, 1968, **7**, 546.

[8] J. R. Sanders, unpublished observations.

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complex $[Rh(HPPh_2)_4]^+BF_4^-$ in ethylene dichloride does not react with hydrogen or carbon monoxide at room temperature and atmospheric pressure or with acetonitrile or aldehydes at 80°. It reacts with hydrogen chloride and hydrogen bromide to give white substances which are completely insoluble in all organic solvents. These are tentatively formulated as the salts $[Rh_2HX_2(HPPh_2)_6]^+BF_4^-$ (X = Cl or Br).

If the complex $[Rh(HPPh_2)_4]^+BF_4^-$ is treated with bases such as triethylamine or methoxide ion, a red substance is obtained. This reacts reversibly with hydrogen in various solvents and catalyses the hydrogenation of pent-1-ene in benzene. It has not yet been characterized because it is extremely sensitive to oxygen even as a solid.

DISCUSSION

The complexes reported here are obtained from the reactions of diphenylphosphine with various platinum-metal compounds. Their structures are very similar to those of complexes formed by the reactions between the same platinum-metal compounds and ditertiary phosphines like bisdiphenylphosphinoethane.[5,9,10] Models show that one bisdiphenylphosphinoethane ligand has similar steric requirements to two *cis*-diphenylphosphine ligands, and that these are much smaller than those of two *cis*-triphenylphosphine ligands. Tetrakisdiphenylphosphine complexes, like bis(ditertiary phosphine) complexes therefore do not dissociate in solution, and the phosphine ligands are not readily replaced by ligands such as CO. In contrast, tetrakistriphenylphosphine complexes like $[RuCl_2(PPh_3)_4]$ dissociate in solution,[11] and even in some tristriphenylphosphine complexes like $[RhCl(PPh_3)_3]$ one phosphine ligand is readily replaced by CO or H_2.

It is theoretically possible for the 'unsaturated' cation $[Rh(HPPh_2)_4]^+$ to react with CO or H_2 without displacement of a phosphine ligand giving the 'saturated' cations $[RhCO(HPPh_2)_4]^+$ and $[RhH_2(HPPh_2)_4]^+$. The failure of these reactions to occur is comparable with the failure of $[Rh(Ph_2PCH_2CH_2PPh_2)_2]^+$ to undergo similar reactions.[12] However hydrogen and carbon monoxide undergo reversible addition reactions with the $[Rh(Me_2PCH_2CH_2PMe_2)_2]^+$ cation,[13] and the $[Rh(HPMe_2)_4]^+$ cation would be expected to behave similarly.

It is sometimes found that in the formation of diphenylphosphine complexes of transition metals a halide ligand is replaced by a diphenylphosphido-ligand.[1,2] Such behaviour has never given a complex with more than four phosphorus-containing groups bound to one metal atom, and it seems possible that the large size of these groups prevents further displacement of halide by diphenylphosphido-ligands. In the present work the complex *cis*-$[ReCl_2(PPh_2)(HPPh_2)_3]$ is formed by reaction of diphenylphosphine with a complex containing three rhenium–chlorine bonds; no further chloride ligands are displaced, and in the complexes $[MCl_2(HPPh_2)_4]$ (M = Ru or Os) there is no tendency for the two chloride ligands to be replaced.

EXPERIMENTAL

Preparations were carried out under nitrogen in dry solvents. Diphenylphosphine was prepared by the reaction of triphenylphosphine and lithium in tetrahydrofuran.[14] The complexes were dried and melting points determined *in vacuo*. Microanalyses were by Yarsley Testing Laboratories, Ashtead, Surrey. Molecular weights were determined, in chloroform, unless stated otherwise, on a Hitachi–Perkin-Elmer model 115 osmometer, and conductivity measurements on the salts were made on 5×10^{-4}M solutions in acetone at 25° on a Pye conductivity bridge. N.m.r. spectra were recorded on a JEOL JNM-4H-100 instrument at 100 MHz, and calibrated against tetramethylsilane. I.r. spectra in the 4000—250 cm^{-1} region were for Nujol mulls and, in some cases, in the 2400—1800 cm^{-1} region for dichloromethane solutions, using a Perkin-Elmer model 457 spectrometer. The Table gives the positions of bands assigned to ν_{P-H}, ν_{M-H}, ν_{C-O}, and ν_{M-Cl} in the spectra of the complexes.

trans-*Dichlorotetrakis*(*diphenylphosphine*)*ruthenium*(II).—Yellow crystals, m.p. 245—255° (decomp.),[2] were obtained immediately (90% yield) on addition of ruthenium trichloride (1 g) in ethanol to diphenylphosphine (5 g) in boiling ethanol.

trans-*Dichlorotetrakis*(*diphenylphosphine*)*osmium*(II).—Ammonium hexachloro-osmate (1·4 g) and diphenylphosphine (3·0 g) were heated under reflux for 15 h in a mixture of water (60 ml), ethanol (70 ml), and benzene (10 ml). A yellow precipitate was obtained which was filtered off after cooling the reaction mixture. It was recrystallized from dichloromethane–methanol (90%) and dried at 100°, m.p. 215° (Found: C, 57·6; H, 4·3; Cl, 7·6. $C_{48}H_{44}Cl_2OsP_4$ requires C, 57·3; H, 4·4; Cl, 7·1%). The compound was too insoluble in chloroform for its molecular weight to be measured.

cis-*Dichlorotetrakis*(*diphenylphosphine*)*ruthenium*(II).— μ-Trichlorohexakis(methyldiphenylphosphine)-diruthenium-(II) chloride (5·0 g) and diphenylphosphine (5·0 g) were heated under reflux for 24 h in ethanol (200 ml). A light yellow precipitate was obtained which was filtered off from the cool reaction mixture. It crystallized as needles from dichloromethane–methanol (68%) and was dried at 125°, m.p. 270—280° (decomp.) (Found: C, 63·1; H, 5·5; Cl, 8·1%; *M*, 856. $C_{48}H_{44}Cl_2P_4Ru$ requires C, 62·9; H, 4·9; Cl, 7·8%; *M*, 916).

cis-*Dichloro*(*tetrakisdiphenylphosphine*)*osmium*(II).— This compound was similarly prepared (78%) by the reaction of μ-trichlorohexakis(methyldiphenylphosphine)-diosmium(II) chloride with diphenylphosphine for 48 h in propanol at 100°, m.p. 245° (Found: C, 57·6; H, 4·8; Cl, 7·9%; *M*, 1000. $C_{48}H_{44}Cl_2OsP_4$ requires C, 57·3; H, 4·4; Cl, 7·1%; *M*, 1005).

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[12] A. Sacco, M. Rossi, and C. F. Nobile, *Chem. Comm.*, 1966, 589.

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[14] D. Wittenberg and H. Gilman, *J. Org. Chem.*, 1958, **23**, 1063.

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cis-*Dichloro*(*diphenylphosphido*)*tris*(*diphenylphosphine*)-*rhenium*(III).— Oxotrichlorobis(triphenylphosphine)rhenium(V) (0·5 g) and diphenylphosphine (0·7 g) were heated under reflux in benzene (20 ml) for 2 h. The colour of the solution gradually changed from blue-green to yellow. Benzene was then removed *in vacuo* leaving an oily residue. This was washed several times with boiling hexane and crystallized from benzene–hexane (75%) and dried at 100°, m.p. 160—170° (decomp.) (Found: C, 57·1; H, 4·5; Cl, 7·1%; *M*, 900. $C_{48}H_{43}Cl_2P_4Re$ requires C, 57·6; H, 4·3; Cl, 7·1%; *M*, 1000).

cis-*Dihydridotetrakis*(*diphenylphosphine*)*osmium*(II).—*trans*-Dichlorotetrakis(diphenylphosphine)osmium(II) (1 g) and sodium dihydrobis(2-methoxyethoxy)aluminate (70% in benzene, 1 ml) were heated under reflux in benzene (10 ml) for 30 min. A clear light yellow solution was obtained. Methanol (1 ml) was added and the solution became colourless, a gas was evolved and a white precipitate was formed. The hot solution was filtered and solvent was removed from the filtrate *in vacuo* leaving a white residue. This was recrystallized from dichloromethane–methanol (65%) and dried at room temperature, m.p. 160—162° (decomp.) [Found: C, 61·6; H, 5·1%; *M* (in benzene), 857. $C_{48}H_{46}OsP_4$ requires C, 62·3; H, 5·4%; *M*, 934].

Tetrakis(*diphenylphosphine*)*rhodium*(I) *Tetrafluoroborate*.—Chlorotris(triphenylphosphine)rhodium(I) (9·0 g) and diphenylphosphine (9·0 g) were warmed to 50° for 5 min in methanol (50 ml). A clear red solution was obtained. Solid ammonium tetrafluoroborate (3·0 g) was added and the reaction mixture was cooled. A yellow precipitate formed which was filtered off, recrystallized from dichloromethane–methanol (75%) and dried at 100°, m.p. 210—220° (decomp.), molar conductivity 84 Ω^{-1} (Found: C, 61·4; H, 5·2. $C_{48}H_{44}BF_4P_4Rh$ requires C, 61·6; H, 4·7%).

The corresponding *hexafluorophosphate* was made using potassium hexafluorophosphate as precipitating agent, m.p. 180—185° (decomp.) (Found: C, 57·5; H, 4·4. $C_{48}H_{44}F_6P_5Rh$ requires C, 58·1; H, 4·4%).

The *perchlorate*, made using lithium perchlorate, exploded *in vacuo* at 150°.

Carbonyltetrakis(*diphenylphosphine*)*iridium*(I) *Perchlorate*.—*trans*-Chlorocarbonylbis(triphenylphosphine)iridium(I) (1·45 g) and diphenylphosphine (3·0 g) were stirred in methanol (20 ml) until a clear yellow solution was obtained (1 h). Lithium perchlorate (1·0 g) was added and a light yellow precipitate formed. This was filtered off, recrystallized from dichloromethane–methanol (60%) and dried at 40°, m.p. 170—175° (decomp.) (Found: C, 55·9; H, 4·3; P, 12·8. $C_{49}H_{44}ClIrO_5P_4$ requires C, 55·8; H, 4·5; P, 12·2%).

The *tetrafluoroborate*, molar conductivity, 111 Ω^{-1}, m.p. 190—195° (decomp.) (Found: C, 55·1; H, 4·4. $C_{49}H_{44}BF_4IrOP_4$ requires C, 56·0; H, 4·2%) and the *tetraphenylborate* (Found: C, 67·5; H, 5·4. $C_{73}H_{64}BIrOP_4$ requires C, 68·2; H, 5·0%) were obtained similarly.

Hydridopentakis(*diphenylphosphine*)*ruthenium*(II) *Hexafluorophosphate*.— Hydridochlorotris(triphenylphosphine)ruthenium(II) (2·0 g) and diphenylphosphine (3·0 g) were warmed to 50° in methanol (20 ml). A light yellow solution with a slight light yellow precipitate was obtained. The solution was filtered and potassium hexafluorophosphate (1·0 g) added to the filtrate which was then cooled. A white precipitate formed. This was filtered off, recrystallized from dichloromethane–methanol (60%) and dried at 100°, molar conductivity, 147 Ω^{-1}, m.p. 255—260° (decomp.) (Found: C, 60·5; H, 4·6. $C_{60}H_{56}F_6P_6Ru$ requires C, 61·2; H, 4·8%).

Hydridocarbonyltetrakis(*diphenylphosphine*)*ruthenium*(II) *Tetrafluoroborate*.— Hydridochlorocarbonyltris(triphenylphosphine)ruthenium(II) (1·0 g) and diphenylphosphine (1·5 g) were warmed to 50° in methanol (10 ml). A clear light yellow solution was obtained to which ammonium tetrafluoroborate (1·0 g) was added; the reaction mixture was cooled. A white precipitate formed. This was filtered off, recrystallized from dichloromethane–methanol (50%) and dried at 100°, molar conductivity, 175 Ω^{-1}, m.p. 240—250° (decomp.) (Found: C, 61·4; H, 5·0. $C_{49}H_{45}BF_4OP_4Ru$ requires C, 61·1; H, 4·7%).

Reactions of $[Rh(HPPh_2)_4]^+BF_4^-$ *with Hydrogen Chloride and Hydrogen Bromide*.—The complex $[Rh(HPPh_2)_4]^+BF_4^-$ (1·0 g) in dichloromethane (30 ml) was treated with dry hydrogen chloride for 5 s. The solution became colourless immediately and a white precipitate slowly formed; 0·7 g of this white substance were obtained after the reaction mixture was left to stand overnight (Found: C, 57·9; H,

Positions and assignments of some bands in the i.r. spectra of diphenylphosphine complexes

Complex	Conditions	ν_{P-H}/cm^{-1}	Other bands/cm^{-1}
trans-$[RuCl_2(HPPh_2)_4]$	Nujol	2341m	311s (ν_{Ru-Cl})
	CH_2Cl_2	2332m, 2305sh	
trans-$[OsCl_2(HPPh_2)_4]$	Nujol	2330m, 2310sh	292s (ν_{Os-Cl})
cis-$[RuCl_2(HPPh_2)_4]$	Nujol	2350w, 2339sh, 2331m	301m, 267m (ν_{Ru-Cl})
	CH_2Cl_2	2342m, 2305sh	
cis-$[OsCl_2(HPPh_2)_4]$	Nujol	2350w, 2322sh, 2316m	289m, 260m (ν_{Os-Cl})
cis-$[ReCl_2(PPh_2)(HPPh_2)_3]$	Nujol	2345m	286m, 251m (ν_{Re-Cl})
	CH_2Cl_2	2336m, 2304sh	
cis-$[OsH_2(HPPh_2)_4]$	Nujol	2297m, 2268m	1976m, 1940s (ν_{Os-H})
	CH_2Cl_2	2305sh, 2280s	1929s (ν_{Os-H})
$[Rh(HPPh_2)_4]^+BF_4^-$	Nujol	2318m	
$[Rh(HPPh_2)_4]^+PF_6^-$	Nujol	2320m	
$[Rh(HPPh_2)_4]^+ClO_4^-$	Nujol	2319m	
$[Ir(CO)(HPPh_2)_4]^+BF_4^-$	Nujol	2315w	1932s (ν_{C-O})
$[Ir(CO)(HPPh_2)_4]^+BPh_4^-$	Nujol	2340w, 2320w, 2300w	1939s (ν_{C-O})
$[Ir(CO)(HPPh_2)_4]^+ClO_4^-$	Nujol	2340w, 2315w	1932s (ν_{C-O})
$[RhH(HPPh_2)_5]^+PF_6^-$	Nujol	2317m, 2290sh	1961m (ν_{Ru-H})
$[RhH(CO)(HPPh_2)_4]^+BF_4^-$	Nujol	2316m	2021 (ν_{C-O}) 1971w (ν_{Ru-H})
$[Rh_2HCl_2(HPPh_2)_6]^+BF_4^-$	Nujol	2373m, 2360sh, 2340w	2083m (ν_{Rh-H})
$[Rh_2HBr_2(HPPh_2)_6]^+BF_4^-$	Nujol	2370m, 2359sh, 2330w	2074m (ν_{Rh-H})

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4·5; Cl, 4·6. $C_{72}H_{67}BCl_2F_4P_6Rh_2$ requires C, 58·2; H, 4·6; Cl, 4·8%).

A similar white substance was obtained when the complex $[Rh(HPPh_2)_4]^+BF_4^-$ was treated with dry hydrogen bromide (Found: C, 54·8; H, 4·2; Br, 11·3. $C_{72}H_{67}BBr_2F_4P_6Rh_2$ requires C, 54·9; H, 4·3; Br, 10·2%).

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