HYDRIDE AND RELATED COMPLEXES OF RHODIUM(III) WITH NITROGEN DONOR LIGANDS

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Abstract—The reactions of the two isomeric series (α and β) of complex hydrides of rhodium of the type RhHX₂L₃ (X is a halogen and L is a tertiary arsine or phosphine) with the nitrogen donor ligands 2,2'-dipyridyl (N-N) and 1,10-phenanthroline (N-N) have been investigated. In presence of perchloric acid the α hydrides give new hydrido compounds of the type [RhHX(N-N)L₂]ClO₄, while the β hydrides give complexes of the type [RhX₂(N-N)L₂]ClO₄. Under similar conditions trihalo compounds of rhodium of the type RhX₃L₃ also give compounds similar to those obtained from the corresponding β hydrides. Configurations for the cationic species have been assigned on the basis of results of i.r. and NMR measurements.

INTRODUCTION

COMPLEX hydrides of rhodium of the type $RhHX_2L_3$, where X is a halogen and L is a tertiary arsine[1-4] or phosphine[5, 6] have been made in two isomeric forms α and β and configurations I and II respectively have



been assigned to them. The properties of these hydrido compounds have not been studied in detail, except for the fact that the methyl diphenyl arsine [7] and ethyl diphenyl phosphine [5] compounds have been found to react with mercuric halides to give compounds containing Rh-Hg bonds. We report, here, the reactions of the two isomeric series of complex hydrides containing the ligands methyl diphenyl arsine (Ph₂MeAs), ethyl diphenyl arsine (Ph₂EtAs) and ethyl diphenyl phosphine (Ph₂EtP) with the nitrogen donor ligands 2,2'dipyridyl (dipy), 1,10-phenanthroline (phen) and pyridine (py).

RESULTS

The hydrido compounds of the type $RhHX_2L_3$ dissolve in boiling alcohol in presence of equimolar amounts of dipy or phen and perchloric acid (in excess) to give orange yellow solutions. Evaporation of these solutions under reduced pressure produces yellow crystalline new hydrido compounds of the type [RhHX(N-N)L₂]ClO₄ (A) [(N-N) is dipy or phen] [8-10] in the case of the α hydrides and compounds of the type [RhX₂(N-N)L₂]ClO₄ (B) in the case of the β hydrides in good yields. It is further found that the corresponding trihalo tertiary arsine complexes of rhodium of the type RhX₃L₃ which have configuration III[11] also react with dipy or phen and give mixed ligand complexes of the type (B) identical with those produced by the β hydrides. It is found that substitution reactions involving dipy are always faster than those with phen probably due to the rigid planar geometry of phen when compared to that of the more flexible one of dipy[12].

In some cases the complex ion $[RhCl_2(N-N)L_2]^+$ may also be obtained as its chloride if the reaction is carried out in presence of hydrochloric acid (added in excess) in place of perchloric acid. However, such compounds are always associated with two water molecules[13] which cannot be removed even after keeping the compounds under vacuum at the temperature of boiling water for 2 hr. These chlorides react with sodium tetraphenyl boron in acetone to give compounds of the type [RhCl_2(N-N)L_2]BPh₄.

Under similar conditions pyridine does not get substituted into the α and β hydrides; the α hydrides remain unchanged while the β hydrides get converted to the α hydrides [3-5]. Even in the absence of pyridine, the β hydrides isomerize to the more stable α hydrides [3-5, 14]. However, if the trihalo compounds of the type Rh X_3L_3 are refluxed with pyridine in alcoholic medium in the absence of perchloric acid, in the case of compounds containing Ph₂MeAs the corresponding α hydrides are produced, while in the case of the Ph₂EtAs

Table 1. Physical 1	properties and anal	vtical data of cationic hy	vdrido and related com	oounds of Rh(III)
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	M.P. or		Analytical data (%)‡		
Compound*	(°C)	Λ†	Carbon	Hydrogen	Nitrogen
[RhHCldipy(Ph ₂ MeAs) ₂]ClO ₄	212-215	26.8	48.29(48.96)	3.96(3.99)	3.30(3.17)
[RhHBrdipy(Ph ₂ MeAs) ₂]ClO ₄	250-252	25.2	46-29(46-60)	3.51(3.80)	2.71(3.02)
[RhHCldipy(Ph2EtAs)2]ClO4	170-174	24.6	49-58(50-07)	4.19(4.31)	3.04(3.08)
[RhHBrdipy(Ph ₂ EtAs) ₂]ClO ₄	178-180	24.8	47.46(47.74)	3.95(4.11)	2.62(2.93)
[RhHCldipy(Ph2EtP)2]ClO4	184–186	24.1	55-35(55-42)	4.77(4.77)	3.58(3.40)
[RhHBrdipy(Ph ₂ EtP) ₂]ClO ₄	179–182	24.5	52.54(52.58)	4.39(4.53)	3.29(3.23)
[RhHClphen(Ph ₂ MeAs) ₂]ClO ₄	165-168	24.8	49.96(50.30)	3.86(3.89)	3.14(3.09)
[RhHBrphen(Ph ₂ MeAs) ₂]ClO ₄	172-175	25.3	48.07(47.94)	3.64(3.71)	2.64(2.94)
[RhHClphen(Ph2EtAs)2]ClO4	170-171	26-2	51.50(51.36)	4.08(4.20)	3.37(3.00)
[RhHBrphen(Ph ₂ EtAs) ₂]ClO ₄	174–177	24.1	49-19(49-03)	4.10(4.01)	2.66(2.86)
[RhHClphen(Ph ₂ EtP) ₂]ClO ₄	170-172	25.1	56-23(56-67)	4.62(4.64)	3.42(3.31)
[RhHBrphen(Ph ₂ EtP) ₂]ClO ₄	168-170	24.5	53-15(53-87)	4-36(4-41)	3.10(3.14)
[RhCl ₂ dipy(Ph ₂ MeAs) ₂]ClO ₄	269-273	24.9	46-50(47-11)	3.77(3.73)	3.05(3.05)
[RhBr ₂ dipy(Ph ₂ MeAs) ₂]ClO ₄	263-265	25.7	42.66(42.95)	3-45(3-40)	2.88(2.78)
[RhCl ₂ dipy(Ph ₂ EtAs) ₂]ClO ₄	223-226	25.2	47.73(48.26)	4.10(4.05)	3.03(2.96)
[RhBr ₂ dipy(Ph ₂ EtAs) ₂]ClO ₄	231-233	27.4	43.11(44.11)	3.60(3.70)	2.85(2.71)
[RhCl ₂ dipy(Ph ₂ EtP) ₂]ClO ₄	173-175	25.6	53-13(53-19)	4.55(4.46)	3-47(3-27)
[RhCl ₂ phen(Ph ₂ MeAs) ₂]ClO ₄	169-172	25.1	47.91(48.46)	3.84(3.64)	2.88(2.98)
[RhBr ₂ phen(Ph ₂ MeAs) ₂]ClO ₄	174–176	24.5	44.68(44.28)	3.58(3.32)	2.53(2.72)
[RhCl ₂ phen(Ph ₂ EtAs) ₂]ClO ₄	215-220	25-3	49.09(49.53)	3.98(3.95)	3.16(2.89)
[RhBr ₂ phen(Ph ₂ EtAs) ₂]ClO ₄	218-221	26.5	45-39(45-36)	3.62(3.62)	2.77(2.65)
[RhCl ₂ phen(Ph ₂ EtP) ₂]ClO ₄	219-221	26.5	54-46(54-48)	4.54(4.34)	3-42(3-18)
[RhCl ₂ dipy(Ph ₂ EtAs) ₂]Cl. 2H ₂ O	>280	25.6	49.46(49.72)	4.79(4.61)	2.98(3.05)
$[RhCl_2phen(Ph_2EtAs)_2]Cl. 2H_2O$	136-138	25-2	50.85(51.00)	4.77(4.49)	3.07(2.98)
[RhCl ₂ dipy(Ph ₂ EtAs) ₂]BPh ₄	212-214	14.9	64.10(63.84)	5.10(5.01)	2.50(2.40)
[RhCl ₂ phen(Ph ₂ EtAs) ₂]BPh ₄	222-225	14-9	64-60(64-60)	4.70(4.91)	2.20(2.36)
$RhCl_3(C_5H_5N)(Ph_2EtAs)_2$	238-239	low	48.88(49.23)	4.47(4.38)	2.18(1.74)
$RhBr_3(C_5H_5N)(Ph_2EtAs)_2$	255-256	low	42.31(42.25)	3.70(3.76)	1-49(1-49)

* Compounds are yellow or orange vellow in colour and show γClO_{4}^{-} ca. 1100 cm⁻¹ in the i.r. spectra.

† Molar conductance (Ω^{-1} cm² mole⁻¹) of ca. 10⁻³ M solutions in nitrobenzene at 25°C.

‡ Calculated values are in parenthesis.

containing compounds pyridine substituted compounds of the type RhX_3pyL_2 (C) are obtained. Compounds of iridium of a similar type containing pyridine have been reported [15, 16]. We have also found that all the trihalo compounds of the type RhX_3L_3 containing Ph_2MeAs and Ph_2EtAs when refluxed with alcoholic piperidine (which is more basic than pyridine) give only the α hydrides [17].

Compounds of both the types (A) and (B) behave as 1:1 electrolytes in nitrobenzene (Table 1) and show bands due to perchlorate and coordinated dipy or phen in their i.r. spectra. The new hydrido compounds show a rhodium-hydrogen stretching band in the 2060 cm⁻¹ region of the i.r. and the value of vRh-H of each of the hydrido compound of type (A) is lower by 10-50 cm⁻¹ (spectra taken in nujol) when compared to that of the corresponding parent hydride[1-5]. The proton NMR spectra of these compounds (A) have the characteristic resonance peak around $\tau 23.5$ (Table 2) due to the hydridic hydrogen[8, 18]. Complexes of the type (B) formed from the β hydrides or the trihalo compounds do not show any metal-hydrogen bands in the i.r.

DISCUSSION

Three stereoisomeric configurations IV, V and VI are possible for these cationic complexes. The proton NMR spectra of the Ph₂EtP containing compounds have



been very helpful in deciding the stereochemistry of these cations. Compounds of type (A) containing Ph_2EtP show a hydridic proton resonance which appears as a 1:3:3:1 quartet around $\tau 23.5$ ($J_{P-H} \simeq J_{Rh-H}$ which is equal to 10-12 Hz). The quartet reson-

ance pattern suggests that there are two phosphorus nuclei at positions *cis* to the hydrogen which would hence give a 1:2:1 triplet resonance. This couples with the rhodium giving two triplets which overlap [18] to give a 1:3:3:1 quartet. The methyl protons of the ethyl group show a 1:4:6:4:1 quintet around $\tau 9.2$ (Table 2). Such a resonance pattern would arise from "virtual" coupling of the methyl proton resonance (triplet) with two trans phosphines (configuration IV) resulting in three 1:2:1 triplets of total relative intensity 1:2:1. These overlap to give a 1:4:6:4:1 quintet. The methylene band is complex and is not well resolved. Similar methyl and methylene patterns are also observed in the case of the cationic complexes of the type (B) containing Ph₂EtP, suggesting an identical trans geometry of the phosphine ligands. Several other workers have made similar observations for complexes containing ethyl diphenyl phosphine[19], diethyl phenyl phosphine[20] and triethyl phosphine[21-23]. The above results suggest that in the cations of both the (A) and (B) type of compounds the phosphine ligands take up trans positions and hence the cations should have configuration IV. It is reasonable to expect that the corresponding methyl diphenyl arsine and ethyl diphenyl arsine complexes also have configuration IV.

It is of interest to mention here that Birnbaum [24] has examined the $\tau_{\rm H}$ values of a large number of octahedral complex hydrides and has concluded that for octahedral iridium and rhodium hydrides, $\tau_{\rm H}$ of the hydridic hydrogen increases in the order I < Br < Clwhen hydrogen is trans to the halogen as well as when hydrogen is simultaneously cis to a halogen and trans to a halogen and decreases in the order I > Br > Clwhen hydrogen is cis to halogen(s). Similar trends in the variation of $\tau_{\rm H}$ values are also observed in the two series of complex hydrides of iridium (α and β) of configuration I and II that we have reported earlier [25, 26]. Thus

the way in which the $\tau_{\rm H}$ values vary in a series of octahedral hydrido complexes of rhodium could be diagnostic in deciding whether a halogen is trans or cis to the hydridic hydrogen. When the $\tau_{\rm H}$ values of the complexes of the type (A) are examined it is seen that the $\tau_{\rm u}$ of a bromo hydride is always higher than the $\tau_{\rm u}$ of the corresponding chloro hydride supporting the conclusion that the halogen is cis to the hydrogen in these hydrido complexes.

The far-i.r. spectra of both the (A) and (B) type of compounds containing Ph₂EtP have been taken in the region $160-400 \text{ cm}^{-1}$. The hydrido compounds (A) containing chloride show a single rhodium-chlorine stretching band around 330 cm⁻¹ which is absent in the corresponding bromo complexes. Further these values are of the same order as those reported by other workers [27-30] for vRh-Cl with the chloride trans to a nitrogen indicating configuration IV for the hydrido cations. The chloro complexes of the type (B) on the other hand show two bands around 350 and 340 cm⁻¹ (the corresponding bromo compounds could not be made pure). These vRh-Cl values are also of the same order of magnitude as those reported [13, 31] for similar rhodium compounds with the two chlorines cis to each other. These results though not conclusive are consistent with the assignment of configuration IV to the cations of the (B) type of compounds.

The above results show that the hydridic hydrogen trans to a halogen of weak trans effect in the α hydrides is not replaced during substitution by dipy or phen. whereas the hydrogen trans to an arsine of strong trans effect in the β hydrides is evidently lost. It is seen that pyridine does not displace an arsine ligand either in the α hydrides or in the β hydrides and also converts the trihalo compounds containing Ph₂MeAs into the α hydrides. This latter reaction is similar to the preparation of hydride complexes in the presence of amines[32].

	Rh-H stretch (cm ^{-1})		Chemical shift $(\tau)^*$		
Compound	Nujol	Chloroform	Rh–H	CH3	CH ₂
RhHCl ₂ (Ph ₂ EtP) ₃ (α)	2122		25-33qt		
$RhHBr_{2}(Ph_{2}EtP)_{3}(\alpha)$	2115		24.64qt		
[RhHCldipy(Ph, MeAs),]ClO4	2050	2045	23.24d	8-29s	
RhHBrdipy(Ph, MeAs), ClO	2053	2047	23.65d	8-26s	
RhHCldipy(Ph_EtAs), ClO	2045	2039	23-29d	8-97t	7.75m
[RhHBrdipy(Ph2EtAs)2]ClO4	2056	2043	23-80d	8-87t	7.67m
[RhHCldipy(Ph2EtP)2]ClO4	2070	2063	23.38g	9-15qn	7.78m,br
[RhHBrdipy(Ph2EtP)2]ClO4	2073	2071	23.81g	9.19qn	7.81m,br
[RhHClphen(Ph, MeAs),]ClO4	2060	2050	23·12d	8·26s	
[RhHBrphen(Ph, MeAs),]ClO4	2060	2054	23.60d	8·24s	
[RhHClphen(Ph2EtAs)2]ClO4	2065	2045	23·24d	8.98t	7.71m
[RhHBrphen(Ph2EtAs)2]ClO4	2070	2050	23.64d	8-89t	7.65m
[RhHClphen(Ph2EtP)2]ClO4	2080	2075	23.33q	9-17qn	7.73m,br
RhHBrphen(Ph_EtP)_ ClO	2077	2075	23.63a	9.17an	7.72m.br

*NMR spectra taken in CDCl₃ using TMS as internal standard; s = singlet, d = doublet, t = triplet, q = quartet, qn = quintet, qt = overlapped quartets, m = multiplet, br = broad.

Pyridine however, replaces an arsine ligand in the trihalo compounds (configuration III) containing Ph_2EtAs . It is likely that the lower basicity of Ph_2EtAs when compared to Ph_2MeAs is responsible for this difference. In support of this suggestion we have found that similar replacement of an arsine ligand by pyridine takes place fairly readily in the trihalo compounds of the type RhX_3L_3 (configuration III) containing *n*-propyl diphenyl arsine and *n*-butyl diphenyl arsine[33]. Since the pyridine containing compounds have been obtained from the trihalo complexes of configuration III by the displacement of an arsine, they should have configuration VII or VIII (assuming that there is no



ligand migration during the reaction). The less symmetric configuration VIII where each of the two cis arsines has a different ligand at the trans position may be expected to show two ethyl patterns (as the two arsines may be considered to be inequivalent) in its proton NMR spectrum, while the more symmetric configuration VII where the two arsines are trans to each other (hence are equivalent) would normally show a single ethyl pattern. The proton NMR spectrum of the compound RhCl₃(C₅H₅N)(Ph₂EtAs)₂ has a single ethyl pattern suggesting configuration VII for the pyridine containing compounds [34, 35] (the corresponding pyridine containing Ph_EtP complexes could not be isolated). Shaw and coworkers [16], however favour (without supporting evidence) configuration VIII for a similar pyridine containing compound of iridium.

EXPERIMENTAL

The rhodium salts used were from Johnson Matthey Chemicals Limited, London. Hydride complexes of the type $RhHX_2L_3$ and the trihalo compounds of the type RhX_3L_3 were prepared as reported elsewhere[3–5]. I.R. measurements were made with a Carl Zeiss UR 10 I.R. Spectrophotometer. Far-i.r. spectra were taken with a Perkin-Elmer Model 180 Spectrophotometer. NMR spectra were taken in CDCl₃ with Varian 100 MHz and Bruker 90 MHz Spectrometers. Conductivity measurements were carried out using Toshniwal Instruments-Conductivity bridge type CL01.02. Micro-analyses were carried out for most of the compounds at University of Sussex and University of Surrey, England, and for some of them at CIBA Research Laboratories, Bombay. Halo hydrido 2,2'-dipyridyl (or 1,10-phenanthroline) bistertiary arsine or phosphine) rhodium(III) perchlorate compounds: $[RhHX(N-N)L_2]ClO_4$

0.2 m-mole of RhH X_2L_3 (α), 0.4 ml of perchloric acid (60 per cent) and 0.21 m-mole of dipy or phen in ethanol (30-40 ml) were refluxed on a water bath when the solid dissolved turning the solution yellow or orange. Refluxing was continued for about 10 hr and the solution was filtered and concentrated under reduced pressure to get yellow shining crystals. The solid was washed successively with ether, water and dilute alcohol and dried *in vacuo*.

Dihalo 2,2'-dipyridyl (or 1,10-phenanthroline) bis(tertiary arsine or phosphine) rhodium(III) perchlorate compounds: $[RhX_2(N-N)L_2]ClO_4$

These compounds were made in the same way as the compounds of the type $[RhHX(N-N)L_2]ClO_4$ by using the β hydrides of the type $RhHX_2L_3$ in place of the α hydrides. They are however more easily prepared by replacing the β hydrides with the corresponding trihalo compounds of the type RhX_3L_3 .

Dichloro 2,2'-dipyridyl (or 1,10-phenanthroline) bis(ethyl diphenyl arsine) rhodium(III) chloride dihydrate compounds: $[RhCl_2(N-N)(Ph_2EtAs)_2]Cl. 2H_2O$

0.2 m-mole of RhCl₃(Ph₂EtAs)₃, 1 ml of concentrated hydrochloric acid and 0.21 m-mole of dipy or phen in ethanol (30-40 ml) were refluxed till all the solid dissolved turning the solution yellow. Solution was filtered and concentrated under reduced pressure to get yellow shining crystals. The solid was washed successively with ether and water and dried *in vacuo*.

Dichloro 2,2'-dipyridyl (or 1,10-phenanthroline) bis(ethyl diphenyl arsine) rhodium(III) tetraphenyl boron compounds: $[RhCl_2(N-N)(Ph_2EtAs)_2]BPh_4$

0.1 m-mole of $[RhCl_2(N-N)(Ph_2EtAs)_2]Cl. 2H_2O$ was suspended in acetone (25 ml) and 0.15 m-mole of sodium tetraphenyl boron was added. The solution was stirred for 3-4 hr and the white solid (NaCl) that separated was filtered off. The filtrate was concentrated to a small volume to get yellow shining crystals. The solid was washed with water and ether and dried *in vacuo*.

Trihalo pyridine bis(ethyl diphenyl arsine) rhodium(III) compounds: $RhX_3(C_5H_5N)(Ph_2EtAs)_2$

0.1 m-mole of $RhX_3(Ph_2EtAs)_3$ and 0.12 m-mole of pyridine were taken in alcohol (20 ml) and refluxed for 1 hr when a shining yellow solid was obtained. The solid was washed with water and alcohol and dried in vacuo.

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