Synthesis and Spectroscopic Characterization of $[ReH_5(triphos)]$ and $[ReH_6(triphos)]^+$ [triphos = PPh(CH₂CH₂PPh₂)₂]

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Reaction of *mer*-[ReCl₃(PMePh₂)₃] with PPh(CH₂CH₂PPh₂)₂ (triphos) in refluxing toluene leads to the formation of [ReCl₃(triphos)] **1**. Treatment of **1** with LiAlH₄ in refluxing Et₂O gives [ReH₅(triphos)] **2**. Protonation of **2** with HBF₄·OEt₂ gives [ReH₆(triphos)]⁺ **3**. Deprotonation of **3** with NEt₃ regenerates **2** quantitatively. The spectroscopic data for compound **3** suggest that it is near the classical/non-classical borderline but exists in solution as a single tautomer only. An estimate of 1.17 Å for the H-H distance of the η^2 -H₂ ligand in **3** was obtained from the *T*₁ data and previous structural work.

Transition-metal dihydrogen $(\eta^2 - H_2)$ complexes are now well established.¹ There are now two important directions in which the area is developing, the search for trihydrogen $(\eta^3 - H_3)$ and polyhydrogen $(\eta^n - H_n)$ complexes, and for hydride complexes in the borderline area between dihydrogen (non-classical) and dihydride (classical) behaviour. We have recently reported our finding of an $\eta^3 - H_3$ species as an intermediate in fluxional exchange of dihydrogen and hydride ligands in $[\text{ReH}_2(\eta^2 - H_2)(\text{CO})(\text{PMe}_2\text{Ph})_3]^+$.² Dynamic solution equilibria between classical and non-classical tautomers have been found for several dihydrides³ and one polyhydride.² Because both tautomers are present, we consider that these complexes are near the classical/non-classical borderline.

Are there separate ranges of $r(H \cdots H)$ values, e.g. > 1.6 and <1.1 Å, for classical and non-classical species, or are intermediate values [1.1 < r(H-H) < 1.6 Å] also possible for complexes in a borderline region? The established examples of solution equilibria between classical and non-classical forms seem to support the 'separate range' hypothesis, but the 'smooth gradation' picture may hold for other systems. Neutron diffraction is not suitable for routine application, and so spectroscopic methods must be used to select appropriate candidates for more detailed study. In this paper we use spectroscopic criteria to identify a candidate system with borderline classical/non-classical character.

Rhenium forms a wide range of polyhydride complexes that hold an important position in the area of hydride complexes by virtue of their fascinating structural chemistry^{1,2,4–10} and unusual chemical reactivity.^{11–15} The heptahydride complexes of the type [ReH₇L₂] are of particular interest in that they undergo a variety of reactions including C–H activation¹³ under mild thermal conditions. On the basis of the relatively low $T_1(\text{min.})$ values^{1b–d} of 55–67 ms (all T_1 values in this paper refer to 250 MHz), Hamilton and Crabtree⁵ questioned the accepted formulation of these compounds as classical nine-coordinate d⁰ rhenium(VII) heptahydrides and proposed that they might better be assigned as [ReH₅(η²-H₂)L₂] in solution, *i.e.* non-classical dihydrogen complexes of eight-co-ordinate d²Re^V.

The non-classical formulation remained controversial, because we^{2.6} and others^{8,16} later found classical hydrides with $T_1(\text{min.})$ values in the same range. This means that values lower than 100 ms cannot always be associated with the presence of the η^2 -H₂ ligand in polyhydrides. A neutron-diffraction study of [ReH₇{Ph₂P(CH₂)₂PPh₂}] revealed a classical nine-coordinate tricapped trigonal prismatic structure in the solid state.⁹ Subsequently, we obtained the slow-exchange-limit spectra for several heptahydrides containing chelating diphosphines.⁷ These provided clear spectroscopic evidence in favour of classical tricapped trigonal prismatic structures in solution for these species, but the structures of the heptahydrides with monodentate phosphines are still insufficiently defined.

Nearly all the known η^2 -H₂ complexes are six-co-ordinate with a d⁶ configuration.^{1a-c} Do non-classical eight-co-ordinate d² rhenium(v) polyhydrides really exist? It has been demonstrated that the introduction of a net positive charge on the metal centre by protonation can decrease the M(d_π) to H₂(σ*) back donation, favouring a non-classical structure. This is best exemplified by the protonation of [IrH₅{P(C₆H₁₁)₃}₂] in which [IrH₂(η^2 -H₂)₂{P(C₆H₁₁)₃}₂]⁺, a bis(dihydrogen) complex of Ir^{III}, is formed.¹⁷ We therefore tried to make a genuinely nonclassical d² rhenium(v) polyhydride using this strategy. The complexes [ReH₅(triphos)] and [ReH₆(triphos)]⁺ [triphos = PPh(CH₂CH₂PPh₂)₂] were prepared and spectroscopically characterized. An important recent publication by Wojcicki and co-workers⁴ on the closely related species [ReH₅L] and [ReH₆L]⁺ {L = PPh[CH₂CH₂P(C₆H₁₁)₂]₂} helped us to formulate our own complexes.

Results and Discussion

Synthesis.—Reaction of $[ReCl_3(PMePh_2)_3]^{18}$ with 1 equivalent of triphos in refluxing toluene for 7 h gave $[ReCl_3(triphos)]$ 1 in 96% yield. The microanalytical data for 1 were consistent with the formulation shown, although no meaningful ¹H and ³¹P NMR spectra could be obtained due to paramagnetism. The new compound probably adopts a meridional configuration as found by X-ray crystallography for its precursor, $[ReCl_3(PMePh_2)_3]^{.8b}$

Monodentate and bidentate phosphines have been widely used as ancillary ligands in polyhydrides,¹¹ but examples supported by polydentate phosphines are quite rare. Such complexes are useful because chelation may slow down the fluxional processes and allow separate hydride resonances to be observed in the ¹H NMR spectra at low temperature.⁷ This facilitates structural studies by solution NMR spectroscopy.

We found that treatment of compound 1 with LiAlH₄ in refluxing Et₂O for 24 h followed by hydrolysis resulted in the formation of [ReH₅(triphos)] 2. Protonation of 2 with HBF₄·OEt₂ in CH₂Cl₂ at 25 °C occurred without hydrogen evolution and yielded [ReH₆(triphos)]⁺ 3. Protonation of [ReH₅L₂] (L = PPh₃ or PMe₂Ph) is known to yield [ReH₆-

Table 1 Variable-temperature ¹H NMR T_1 measurements on the hydride resonances of [ReH₅(triphos)] **2** and [ReH₆(triphos)]⁺ **3** in CD₂Cl₂ at 250 MHz

	T_1/ms	
T/K	2	3
183	117	58
193	103	48
203	91	39
213	86	32
223	83	34
233	96	38
243	110	41
253	126	46
263	154	53
273	186	62



Fig. 1 The hydride region of the 1 H NMR spectrum (298 K, 250 MHz) of [ReH₅(triphos)] 2 in CD₂Cl₂



Fig. 2 Plot of $\ln T_1 vs. 10^3/T$ for $[\text{ReH}_6(\text{triphos})]^+$ 3 in CD₂Cl₂

 L_2]^{+,15a,b} which loses hydrogen spontaneously at room temperature. In contrast, **3** is surprisingly stable toward hydrogen loss; it remains intact even after several days *in vacuo* at room temperature. Deprotonation of **3** with NEt₃ regenerated **2** quantitatively. Both compounds **2** and **3** were characterized on the basis of microanalytical, IR and NMR spectroscopic data. During the course of this work, Wojcicki and co-workers⁴ reported [ReH₆L]⁺ **4** {L = PPh[CH₂-CH₂CH₂P(C₆H₁₁)₂]₂}, a compound closely analogous to **3**.

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Spectroscopic Studies.--The IR spectra of compounds 2 and 3 show characteristic Re-H stretching bands in the region 1870-1990 cm⁻¹. No bands which could be assigned to the $Re(\eta^2-H_2)$ binding were observed. The ³¹P-{¹H} NMR spectrum of **2** shows an AB₂ pattern (${}^{2}J_{PP} = 6.0$ Hz), consistent with the existence of two types of ${}^{31}P$ nuclei. The ${}^{1}H$ NMR spectrum (Fig. 1) at 298 K shows a triplet of doublets (δ – 7.34, ${}^{2}J_{\rm PH} = 15.1$ and 9.6 Hz) in the hydride region; the five hydride ligands are apparently equivalent through rapid fluxionality commonly observed for rhenium polyhydrides.¹⁹ The hydride resonance becomes a broad feature on cooling to 183 K, but the slow-exchange-limit spectrum could not be obtained. The ¹H-{³¹P} NMR spectrum of an isotopomeric mixture of $[\text{ReH}_{5-x}D_x(\text{triphos})]$ (x = 0-5) at 298 K shows a very small upfield isotope shift of -0.0054 ppm per D for the hydride resonance. This value is comparable to those observed for some classical rhenium polyhydrides.^{2,6,7}

Compound 2 gives a $T_1(\min)$ value of 83 ms at 223 K (Table 1). Although this is somewhat lower than usually found for most classical polyhydrides, it is consistent with the formulation of 2 as a classical pentahydride on the basis of the recent results on rhenium polyhydrides.⁶⁻⁹ In particular, classical structures have been established for the analogous $[\text{ReH}_5(\text{PPh}_3)_3]^{3c}$ and $[\text{ReH}_5(\text{PMePh}_2)_3]^{10}$ by X-ray and neutron diffraction studies.

The ¹H and ³¹P-{¹H} NMR spectra of compound 3 are quite similar to those of 2. The ³¹P-{¹H} NMR spectrum displays an AB₂ pattern (²J_{PP} = 5.1 Hz). The ¹H NMR spectrum at 298 K shows a triplet of doublets hydride resonance ($\delta - 5.11$, ²J_{PH} = 14.0 and 10.3 Hz) which becomes a featureless signal upon cooling to 183 K. Again, the slow-exchange-limit spectrum could not be observed. The ¹H-{³¹P} NMR spectrum of an isotopomeric mixture of [ReH_{6-x}D_x(triphos)]⁺ (x = 0-6) shows a very small upfield isotope shift of -0.0049 ppm per D. As in the case of 2, this value is comparable to those observed for classical rhenium polyhydrides.^{2,6,7}

Interestingly, compound 3 gives a rather short $T_1(\min)$ value of 32 ms at 213 K (Table 1). A comparable value of 27 ms was found for 4 at 233 K.⁴ In an ideal case, the plot of ln T_1 vs. 1/T is expected to be V-shaped in the sense that the slopes in the highand low-temperature regimes have the same values and differ only in sign. The plot for 3, shown in Fig. 2, is not as well behaved as we have seen in other cases. On the other hand, it does not resemble the grossly distorted curve observed in the case of [ReH₄(CO)(PMe₂Ph)₃]⁺,^{1c,2} where the classical and non-classical tautomers are in rapid equilibrium. This suggests that 3 is predominantly a single isomer in solution.

While it is tempting to formulate compound 3 as nonclassical on the basis of its low $T_1(\min)$ value,⁵ the small upfield isotope shift observed seems unlikely to be caused by the i.p.r. (isotopic perturbation of resonance) effect due to isotope fractionation between different sites.^{2,6,7} Nevertheless, since the low-temperature-limiting spectrum cannot be obtained, we do not know the chemical shift difference between the classical and non-classical sites, and so the absence of a large i.p.r. shift of the hydride resonance is probably not good evidence against a nonclassical structure. On the other hand, the fact that different isotopomers of 3 exhibit the same ${}^2J_{\rm PH}$ coupling constant seems to be in favour of a classical formulation.

Another possibility is that compound 3 adopts a classical/ non-classical borderline structure which contains one or more $H \cdot \cdot \cdot H$ distances intermediate between that of an η^2 - H_2 ligand and that of two classical hydride ligands. If this is the case, the i.p.r. effects are perhaps not operative because significant H/D isotopic fractionation over different sites depends on the force constants of the bonds being sufficiently different.

A low-temperature X-ray diffraction study of compound 4 revealed an η^2 -H₂ ligand with an r(H-H) value of 1.08 Å.⁴ If this is reliable (it has not yet been confirmed by neutron-diffraction data), the H–H distance seems to fall at the lower end of the range of intermediate r(H-H) values [1.1 <

r(H-H) < 1.6 Å] in the borderline region. As noted by Wojcicki and co-workers,⁴ the hydride positions given in the X-ray crystal structure do correctly predict the observed $T_1(min.)$ using the method we suggest ^{1b-d,7b} and assuming there is slow rotation²⁰ of the η^2 -H₂ ligand (calc., 23.4; obs., 27 ms). If we assume that 3 has the same structure as 4 except for the H-H distance of the η^2 -H₂ ligand, and that there is slow H₂ rotation, we can make a crude estimate of r(H-H) in 3. We calculate that an H-H distance of 1.17 Å would account for the observed T_1 (min.) of 32 ms for 3 at 250 MHz. This r(H-H) value would also provide a reasonable explanation of the spectroscopic properties of 3; this distance may be long enough so isotopic fractionation does not occur to a sufficient extent to give easily detectable i.p.r. effects. If we make the extreme assumption that the classical hydrides are not relaxing at all and ascribe all the relaxation to one freely rotating η^2 -H₂ ligand, we can set a lower limit of 1.04 Å for the H \cdots H distance in compound 3. We are attempting to obtain large crystals of 3 and related species for a neutron-diffraction study.

Conclusion

We hoped to prepare a d² rhenium(v) dihydrogen complex $[\text{ReH}_6\text{L}_3]^+$ by protonating $[\text{ReH}_5\text{L}_3]$. Like Wojcicki, we find that use of a chelating tridentate phosphine confers remarkable stability on the cationic rhenium hexahydride, which loses H_2 when monodentate phosphines are used. The unusual properties of $[\text{ReH}_6\text{L}]^+$ 4 {L = PPh[CH₂CH₂CH₂P- $(C_6H_{11})_2]_2$, are however not attributed to any unusual steric and/or electronic effects of the cyclohexyl groups on the ligand that Wojcicki used, because triphos gives very similar results. The solution spectroscopic data for $[\text{ReH}_6(\text{triphos})]^+$ 3 do not allow a definitive structural assignment regarding the presence of the η^2 -H₂ ligand, but the compound probably exists as a single tautomer in solution. The low $T_1(\min)$ value of 32 ms suggests that the molecule is something other than a classical hydride with normal van der Waals H · · · H contacts $[r(H \cdots H) > 1.6 \text{ Å}]$. Compound 3 appears to be near the classical/non-classical borderline, and an η^2 -H₂ complex with a long H-H distance seems to be the most likely structure despite the fact that other systems show equilibria between classical and non-classical tautomers.^{2,3} If 3, like 4, is indeed an η^2 -H₂ complex, then we can say that the i.p.r. method may not always be a sensitive indicator for H ••• H interactions of this type.

Experimental

General Procedures.—All manipulations were performed under a dry nitrogen atmosphere by standard Schlenk-tube techniques. Proton ¹³C and ³¹P NMR spectra were recorded on Bruker WM 250 or WM 500 spectrometers with CD_2Cl_2 as the solvent; ¹H and ¹³C chemical shifts were measured with the residual solvent resonance as reference, ³¹P chemical shifts with external 85% H₃PO₄ as reference. Infrared spectra were recorded on a Nicolet 5-SX FT-IR spectrometer. The T_1 measurements were carried out by the inversion-recovery method using a standard 180°– τ –90° pulse sequence.

Diethyl ether, hexane and tetrahydrofuran were distilled from Na-Ph₂CO, dichloromethane from CaH₂. All solvents were stored under N₂ over 4 Å molecular sieves. The compound [ReCl₃(PMePh₂)₃] was prepared according to the literature method.¹⁸

[ReCl₃(triphos)] 1.—A solution of [ReCl₃(PMePh₂)₃] (1.00 g, 1.12 mmol) and triphos (0.60 g, 1.12 mmol) in toluene (40 cm³) was refluxed for 7 h. The solvent was removed to dryness. The residue was extracted with CH_2Cl_2 (4 × 10 cm³). A small amount of insoluble impurity was filtered off. The filtrate was concentrated to *ca.* 3 cm³ and Et₂O (40 cm³) added. The resulting yellow precipitate was collected on a frit, washed with Et₂O (4 × 10 cm³), and dried *in vacuo.* Yield: 0.89 g,

96% (Found: C, 50.05; H, 4.25. Calc. for $C_{34}H_{33}Cl_3P_3Re: C$, 49.35; H, 4.00%).

[ReH₅(triphos)] **2**.—A suspension of [ReCl₃(triphos)] **1** (1.00 g, 1.12 mmol) and LiAlH₄ (0.43 g, 11.2 mmol) in Et₂O (40 cm³) was refluxed for 24 h. The mixture was filtered through Celite and the filtrate evaporated to dryness *in vacuo*. The residue was dissolved in tetrahydrofuran (thf) (20 cm³), cooled to 0 °C, and hydrolysed by dropwise addition of water (0.8 cm³) in thf (20 cm³). The mixture was dried with anhydrous Na₂SO₄ (5 g) an filtered through Celite. The yellow filtrate was concentrated to *ca*. 1 cm³ *in vacuo*. Addition of hexane (40 cm³) and cooling to 0 °C resulted in the precipitation of a pale yellow solid which was filtered off, washed with hexane (4 × 10 cm³), and dried *in vacuo*. Yield: 0.37 g, 45%. (Found: C, 56.40; H, 5.20. Calc. for C₃₄H₃₈P₃Re: C, 56.25; H, 5.30%). IR (Nujol): v(Re–H) 1949, 1914 and 1872 cm⁻¹. NMR (CD₂Cl₂, 298 K): ¹H, δ 7.9–7.3 (c, 25 H, Ph), 3.2–3.1 (c, 2 H, CH₂), 2.6–2.5 (c, 2 H, CH₂), 1.9–1.7 (c, 4 H, CH₂) and -7.34 (td, ²J_{PP} = 6.0, 1 P) and 49.1 (d, ²J_{PP} = 6.0 Hz, 2 P).

[ReH₆(triphos)]BF₄ **3**.—To a solution of [ReH₅(triphos)] **2** (0.12 g, 0.17 mmol), in CH₂Cl₂ (5 cm³) was added HBF₄•OEt₂ (85% in Et₂O, 20 μl, *ca*. 0.17 mmol) *via* a microsyringe. The reaction mixture was stirred for 10 min and then concentrated to 1 cm³. Addition of Et₂O (20 cm³) resulted in the precipitation of a beige solid which was collected on a frit and washed with Et₂O. The product was recrystallized from CH₂Cl₂–Et₂O. Yield: 0.090 g, 65% (Found: C, 49.85; H, 4.80. Calc. for C₃₄H₃₉BF₄P₃Re: C, 50.20; H, 4.85%). IR (Nujol): v(Re-H) 1985 and 1937 cm⁻¹. NMR (CD₂Cl₂, 298 K): ¹H δ 8.0–7.4 (c, 25 H, Ph), 3.6–3.1 (c, 4 H, CH₂), 2.3–2.1 (c, 4 H, CH₂) and –5.11 (td, ²J_{PH} = 14.0 and 10.3 Hz, 6 H, Re–H); ³¹P-{¹H}, δ 77.3 (t, ²J_{PP} = 5.1, 1 P) and 38.5 (d, ²J_{PP} = 5.1 Hz, 2 P); ¹³C-{¹H}: δ 135.3 (c, C¹ of PPh), 134.6 (c, C¹ of PPh₂), 133.7 (d, ²J_{PC} = 11.1, C² of PPh), 132.9 (s, C⁴ of PPh), 132.4 (c, C² of PPh₂), 131.8 (d, ³J_{PC} = 7.4, C³ of PPh₂), 35.2 (c, CH₂PPhCH₂) and 31.8 (c, CH₂PPh₂).

Calculation of T_1 .—The positional parameters of the hydride ligands in compound 4 were obtained from the supplementary data for ref. 4, and the methods of refs. 1*d* and 7*b* were used. For the estimate of $r(H \cdots H)$ in 3 the classical hydride relaxation rate of 10.52 s⁻¹ per H found for 4 was assumed. This number includes relaxation among the classical hydrides and the relaxation between the η^2 -H₂ ligand and the other hydrides.

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