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PROTONATION OF TERTIARY PHOSPHINE PLATINUM(0) COMPLEXES BY AMMONIUM ION. CHARACTERIZATION AND SOME REACTIONS OF THE RESULTING HYDRIDES

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Summary

The platinum(0) complexes $Pt(PR_3)_2$ ($R_3 = (t-Bu)_3$ and $(t-Bu)_2Ph$) react readily with the ammonium salts NH_4X ($X = PF_6$ and ClO_4) to give the cationic ammine hydrides *trans*-[PtH(NH_3)(PR_3)_2]X. Reaction of Pt(PPh_3)_3 with NH_4PF_6 affords [PtH(PPh_3)_3]PF_6. The ammine hydrides *trans*-[PtH(NH_3)L_2]X are deprotonated and give the Pt(PR_3)_2 complexes when NH_3 is passed through their solutions in acetone; facile deprotonation of the ammine hydrides also occurs upon passing CO and the trinuclear complexes $Pt_3(PR_3)_3(CO)_3$ are obtained. Dichloromethane solutions of the ammine hydrides are unaffected by NH_3 but CO reacts with these solutions to give the carbonyl hydrides *trans*-[PtH(CO)(PR_3)_2]X. The hydride [PtH(PPh_3)_3]PF_6 does not react with NH_3 or CO in either acetone or dichloromethene. Proton and ³¹P-(¹H) NMR spectral parameters as well as the characteristic infrared frequencies for all the complexes are reported.

Introduction

We have recently reported [1] the first formally three-coordinate platinum(II) hydride $[PtH{P(t-Bu)_3}_2]^+$. This three-coordinate species readily reacts with small neutral ligands, including H₂O, to give four coordinate complexes of the type, *trans*-[PtH(ligand)(P(t-Bu)_3)_2]^+. Attempts to prepare the ammine hydride, *trans*-[PtH(NH₃)(P(t-Bu)_3)_2]X, by passing NH₃ through an acetone solution of the three-coordinate complex, however, failed; the platinum(0) complex, Pt(P(t-Bu)_3)_2 was obtained instead [1]. In the course of our continuing investigations [2] on the chemical reactivity of Pt(P(t-Bu)_3)_2 it was discovered that the platinum(0) complex

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reacts with ammonium salts to give the ammine hydride, trans-[PtH(NH₃){P(t-Bu)₃)₂]⁺. Due to lack of information on platinum(II) ammine hydrides, reactions of other platinum(0)-phosphine complexes such as Pt{P(t-Bu)₂Ph)₂ and Pt(PPh₃)₃ with the NH₄⁺ ion were also examined. Results of these investigations as well as characterization and some reactions of the resulting hydrides are reported herein.

Results and discussion

Platinum(0) complexes, $Pt(PR_3)_2$ ($R_3 = (t-Bu)_3$ and $(t-Bu)_2Ph$), react readily, at room temperature, with NH_4X ($X = PF_6$ and ClO_4) in acetone or tetrahydrofuran (THF) according to eq. 1 to give the ammine hydrides *trans*-[PtH(NH_3)(PR₃)₂]X

$$Pt(PR_3)_2 + NH_4 X \rightarrow trans \left[PtH(NH_3)(PR_3)_2 \right] X$$

$$(1)$$

$$(R_3 = (t-Bu)_3; X = PF_6 (1a) \text{ or } ClO_4 (1b);$$

$$R_3 = (t-Bu)_2 Ph; X = PF_6 (2))$$

(1a), (1b) and (2) in almost quantitative yields. Reaction 1 provides the first example of the preparation of an ammine hydride by protonation of a metal complex by ammonium ions. The recently reported [3] protonation of $Fe(P(OMe)_3)_5$ by NH_4^+ gives the hydride $[FeH\{P(OMe)_3\}_5]^+$ instead of an ammine hydride.

Complexes 1 and 2 are air-stable white solids and represent the first fully characterized platinum(II) ammine hydrides. The only other known complex of this kind is $(PtH(NH_3)(PPh_3)_2)ClO_4 * [4]$ which has been characterized by only microanalysis and Pt-H stretching frequency. Complexes 1-2 were characterized unequivocally by analytical, infrared, and ¹H and ³¹P-{¹H} NMR spectral data. The analytical and infrared data are given in Table 1 and the NMR data in Table 2. The $^{31}P-{^{1}H}$ NMR spectra (in dichloromethane) of 1-2 showed a single resonance accompanied by a pair of satellite resonances due to the ¹⁹⁵Pt-³¹P spin-spin coupling (¹J(Pt-P) 2870 Hz). The spectra of 1a and 2 also showed a characteristic septet at $\delta - 144.7$ ppm (¹J(P-F) 714 Hz) due to PF₆⁻. The ¹H NMR spectra of 1-2, in the t-butyl region consisted of a triplet at ca. δ 1.5 ppm (${}^{3}J(P-H) + {}^{5}J(P-H)$ 12.6 Hz) confirming the trans geometry of the phosphine ligands [5]. The hydride resonance for each complex was observed as a 1/2/1 triplet, upfield from TMS, and flanked on either side by a satellite triplet due to ¹⁹⁵Pt-H spin-spin coupling. The ¹H- as well as ³¹P-{¹H} NMR parameters for 1 and 2 are comparable to those for the complexes *trans*-[PtHL(PR₃)₂]⁺, where L = a nitrile and R₃ = (t-Bu)₃ or (t-Bu)₂Ph [1].

The infrared spectra of 1 and 2 showed a medium intensity band at 2280–2370 cm⁻¹ due to the Pt-H stretching frequency [6,7]. The observed $\nu(Pt-H)$ for 1-2 is higher than that reported for $[PtH(NH_3)(PPh_3)_2]ClO_4$ (2202 cm⁻¹) [4]. The neutral hydrides *trans*-PtHCl[P(t-Bu)_3]₂ [8] and *trans*-PtHCl[P(t-Bu)_2Ph]₂ [9] also have $\nu(Pt-H)$ values higher than that for the analogous triphenylphosphine complex [10]. The infrared spectrum for each complex also showed the characteristic frequencies for the counter anion [11]. These frequencies are listed in Table 1, and clearly show the presence of the unperturbed anion in each compound. Characteristic infrared

^{*} It has been prepared by the reaction of NH₃ with [PtH(CO)(PPh₃)₂]ClO₄ (ref. 4). We find that NH₃ does not react with either [PtH(CO)(P(t-Bu)₃)₂]⁺ or [PtH(CO)(P(t-Bu)₂Ph)₂] (vide infra).

bands [11] due to coordinated NH₃ were also observed for each ammine hydride.

The ¹H NMR parameters for 1 and 2 did not change when the spectra were measured in $(CD_3)_2CO$ instead of CD_2Cl_2 or $CDCl_3$ indicating their non-lability in acetone. However, as shown by eq. 2, the platinum(0) complexes $Pt(PR_3)_2(R_3 = (t-Bu)_3 \text{ or } (t-Bu)_2Ph)$ were obtained quantitatively when NH_3 was passed into acetone solutions of 1 and 2. In marked contrast to this facile deprotonation, no reaction

trans-
$$[PtH(NH_3)(PR_3)_2]^+ \xrightarrow{NH_3} \xrightarrow{acetone} Pt(PR_3)_2 + NH_4^+$$
 (2)
CH₂Cl₂ no reaction

occurred when dichloromethane was used as solvent.

As shown by equations 3a and 3b, reactions of 1 and 2 with CO also proceeded differently in acetone and dichloromethane. Deprotonation of the complexes 1 and 2 occurred and the trinuclear clusters, $Pt_3(PR_3)_3(CO)_3$ [2,9], were obtained as the sole

$$trans-\left[PtH(NH_3)(PR_3)_2\right]^+ \xrightarrow{CO} \frac{acetone}{CH_2Cl_2} \frac{1/3\left[Pt_3(PR_3)_3(CO)_3\right] + NH_4^+}{trans-\left[PtH(CO)(PR_3)_2\right]^+ + NH_3^+ (3b)}$$

platinum containing species in acetone (eq. 3a). Under similar conditions, reactions in dichloromethane resulted in the substitution of
$$NH_3$$
 by CO and gave the carbonyl hydrides *trans*-[PtH(CO)(PR_3)₂]⁺ (eq. 3b).

The trinuclear complexes $Pt_3(PR_3)_3(CO)_3$ were formed quantitatively when CO was bubbled through suspensions of the $Pt(PR_3)_2$ complexes in acetone. Thus, reaction 3a proceeds via formation of the $Pt(PR_3)_2$ complexes which subsequently react with CO to give the $Pt_3(PR_3)_3(CO)_3$ complexes. Reactions 2 and 3a are driven to completion due to the insolubility of the $Pt(PR_3)_2$ or $Pt_3(PR_3)_3(CO)_3$ complexes in acetone.

The ammine hydride trans- $[PtH(NH_3)(PPh_3)_2]^+$ has been prepared [4] by the substitution of CO in trans-[PtH(CO)(PPh₃)₂]⁺ by NH₃. Surprisingly, reverse of reaction 3b did not take place when NH₃ was passed through solutions of the carbonyl hydrides trans-[PtH(CO)(PR₃)₂]⁺ in acetone or dichloromethane; ¹H- and ³¹P-{¹H} NMR measurements showed the presence of the unchanged carbonyl hydrides. Thus, deprotonation of the ammine hydrides seems to be more facile than that of the carbonyl hydrides. As shown by the data in Tables 1 and 2, the $\nu(Pt-H)$ as well as the magnitude of the upfield hydride chemical shift for the ammine hydrides 1 and 2 are markedly higher than those for the analogous carbonyl hydrides. Both the $\nu(Pt-H)$ and the upfield hydride shifts for the complexes in the series trans-[PtHL(PEt₃)₂]⁺ have been found [12] to decrease with increasing σ -donor power of the ligand L. Thus, the facile deprotonation of the ammine hydrides 1 and 2 relative to the carbonyl hydrides seems to be consistent with a lower electron density at platinum in the former complexes. This is undoubtedly due to steric reasons. Ammonia has a much larger steric requirement than CO, and, cannot get as close to the platinum as CO due to the steric crowding caused by the two bulky phosphine ligands.

The complex $Pt(PPh_3)_3$ also reacts with ammonium salts but, unlike the $Pt(PR_3)_2$ complexes, does not afford the ammine hydride; the previously known hydride

Complex		Analyses " ('	8)	:	IR $(cm^{-1})^{b}$		
		С	Н	Z	µ(Pt−H)	<i>ν</i> (NH ₃)	δ(NH ₃)
trans-[PtH(NH ₁)(P(t-Bu) ₁),]PF ₆ ^c	(1 a)	37.66	7.60	1.80	2362m	3390m,3300m	1625s
		(37.79)	(1.68)	(1.84)			
trans-[PtH(NH ₃)(P(t-Bu) ₃),]ClO ₄ ^d	(1þ)	40.24	8.22	1.91	2360m	3340m,3265m	1620s
		(40.18)	(8.17)	(1.95)			
trans-[PtH(CO)(P(t-Bu) ₁),]PF ₆ ^{c.e}		38.91	7.00		2240m		
		(38.80)	(7.18)				
[PtH(NH ₃)(P(t-Bu) ₂ Ph) ₂]PF ₆ ^c	(2	41.75	6.40	1.72	2280s.	3360m,3285m	1615s
		(41.89)	(6.29)	(1.75)			
<i>trans</i> -[PtH(CO)(P(t-Bu) ₂ Ph ₂)]PF ₆ ^{c,J}		42.72	5.74		2210m		
		(42.80)	(5.83)				
[PtH(PPh ₁),]PF ⁶	3)	57.44	3.98				
		(57.54)	(4.08)		2150m		
" Calculated values are in parenthesis. ^b F $^{\prime}$ P(CO), 20408. ^f $_{P}$ (CO), 2045s.	or the solid sta	ate (Nujol mull).	^ε μ(PF), ~ 840(ν	s)cm ⁻¹ ;	~ 555(s) cm ⁻¹ . ^d	▶(ClO), 1190(vs)cm ⁻¹ ;	δ(ClO), 618(s) cm ⁻¹ .

TABLE I ANALYTICAL AND IR SPECTRAL DATA

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TABLE 2 ¹H AND ³¹P-(¹H) NMR SPECTRAL DATA

Complex		¹ H NMR ^a					³¹ P NMR [/]	
		&(t-Bu) (ppm)	(³ <i>J</i> (P-H)+ ⁵ <i>J</i> (P-H) (Hz)	å(Pt–H) (ppm)	¹ J(Pt-H) (Hz)	² /(Pt-H) (H2)	g (mqq)	¹ J(Pt-P) (Hz)
<i>trans</i> -[PtH(NH ₃), R(t-Bu) ₃), JPF ₆ ^c <i>trans</i> -[PtH(NH ₃), R(t-Bu) ₃), JD10 ₄ <i>trans</i> -[PtH(CO), R(t-Bu) ₃), JPF ₆ ^c <i>trans</i> -[PtH(NH ₃), RP(t-Bu) ₂ , Ph ₃ , PF ₆ ^c <i>trans</i> -[PtH(CO), R(t-Bu) ₂ , Ph ₃ , PF ₆ ^c [PtH(PPh ₁), JPF ₆ ^c	(1 a) (1b) (3) (2) (1b)	1.52t 1.51t 1.69t ^d 1.53t 1.50t	12.6 12.7 13.9 13.5	- 19.3t - 19.2t - 5.2t ^d - 17.7t - 5.1t - 4.7m ^d	955 940 142 874 778	11.6 9.6 9.9 9.9	79.0 79.0 89.4 ^d 67.6 ^d 24.9 d.8	2848 2856 2416 2881 2508 2836
	, ,					12.07	25.3 ^{d.h}	2226

^a In CDCl₃ except stated otherwise. ^b In CH₂Cl₂ containing 10% CDCl₃ except stated otherwise. ^c δ (PF₆), -145 ppm (¹)(P-F) ~ 714 Hz). ^d In (CD₃)₂CO. ^c ²J(P-H)_{trans}, ^{f2}J(P-H)_{cis}, ^g PPh₃ cis to the hydride, ²J(P-H)_{cis}, ^g PPh₃ cis to the hydride.

 $[PtH(PPh_{3})_{3}]^{+} [13-14] \text{ was obtained instead (eq. 4).}$ $Pt(PPh_{3})_{3} + NH_{4}PF_{6} \rightarrow [PtH(PPh_{3})_{3}]PF_{6} (3) + NH_{3}$ (4)

Thus, reaction of $Pt(PPh_3)_3$ with NH_4^+ appears to be similar to the reported [3] protonation of $Fe[P(OMe)_3]_5$ by NH_4^+ .

The infrared spectrum of $[PtH(PPh_3)_3]PF_6$ showed a medium band at 2150 cm⁻¹ due to $\nu(Pt-H)$ [6], and characteristic bands at 830 and 555 cm⁻¹ due to the PF₆⁻ ion [11]. The $\nu(Pt-H)$ is in good agreement with the $\nu(Pt-H)$ value of 2145 cm⁻¹ reported [13] for $[PtH(PPh_3)_3][(CF_3CO_2)H]$. Previous workers [13,14] have reported that the hydride resonance for $[PtH(PPh_3)_3]^+$ at 60 MHz as well as at 100 MHz consist of two centre double multiplets flanked on either side by the ¹⁹⁵Pt satellites which appear as double triplets. The spectrum has been interpreted [14] in terms of an MXAB₂ spin system and the observation of the complex centre band has been attributed to second-order effects. We also observed a similar ¹H NMR spectrum for $[PtH(PPh_3)_3]PF_6$ at 60 as well as at 400 MHz.

The ³¹P-{¹H} NMR spectrum of [PtH(PPh₃)₃]PF₆ at 24.4 MHz showed a strong resonance at δ 24.9 and a weaker resonance at 25.3 ppm. The δ 24.9 resonance was flanked on each side by a satellite doublet showing ¹⁹⁵Pt-³¹P as well as P-P spin-spin coupling (¹J(Pt-P) 2836 Hz; ²J(P-P) 18 Hz). The spectrum also showed a characteristic septet due to the PF₆⁻ ion. For a first-order MXAB₂ type spectrum, the two *trans* phosphines are expected to give rise to a centre doublet and a pair of satellite doublets; the phosphine *trans* to the hydride is expected to give rise to a centre resonance is, thus, attributable to the *trans* phosphines and the δ 25.3 resonance to the phosphine *trans* to the hydride. This was confirmed by ³¹P-{¹H} NMR measurements at 161.9 MHz which gave a first order spectrum exhibiting a centre doublet and a singlet. The doublet was accompanied by a pair of satellite doublets (¹J(Pt-P) 2836 Hz; ²J(P-P) 18 Hz). The singlet was broad and was accompanied by a pair of satellite singlets which were also broad (¹J(Pt-P) 2226 Hz).

The hydride 3, unlike the ammine hydrides 1 and 2, does not react with CO, in either dichloromethane or acetone. The ¹H- and ³¹P-(¹H) NMR spectra of acetone as well as dichloromethane solutions of 3 did not show any change when CO was passed through the solution for 2 h; the hydride 3 was recovered unchanged. In marked contrast to the facile deprotonation of the ammine hydrides 1 and 2, no reaction also occurred when NH₃ was passed through an acetone solution of 3.

Experimental

General. Tri-t-butylphosphine [15] $P(t-Bu)_2Ph$ [16], $Pt[P(t-Bu)_3]_2$ [2] $Pt[P(t-Bu)_2-Ph]_2$ [17], and $Pt(PPh_3)_3$ [18] were prepared following published procedures. The solvents were reagent grade and were dried prior to use. Reagent grade ammonium salts were used as received. All operations were carried out under an atmosphere of oxygen-free dry argon using a Vacuum Atmosphere Corporation drybox. Standard vacuum line techniques were used for removing solvents and volatile materials.

Physical measurements. Elemental analyses were performed by Guelph Chemical Laboratory, Guelph. Infrared spectra were recorded with a Perkin-Elmer 180 spectrometer using samples prepared as mulls in Nujol and pressed between KBr plates. Proton and ³¹P- $\{^{1}H\}$ spectra were recorded in the Fourier transform mode

with Bruker WP-60 and WH-400 spectrometers using internal TMS and external 85% H₃PO₄ as references; positive chemical shifts are downfield from the reference.

Reactions of $Pt[P(t-Bu)_3]_2$ and $Pt[P(t-Bu)_2Ph]_2$ with ammonium salts

A mixture of $Pt[P(t-Bu)_3]_2$ or $Pt[P(t-Bu)_2Ph]_2$ (0.5 mmol) and $NH_4X(PF_6$ or ClO_4) (0.5 mmol) was stirred in ~ 10 ml tetrahydrofuran (THF) for ~ 48 h. The solvent was removed and dichloromethane (~ 4 ml) was added to the residue. The resulting suspension was passed through a small florisil column to give a clear solution which was diluted with pentane, and then cooled to give colourless crystals of *trans*-[PtH(NH₃)(PR₃)₂]X (R₃ = (t-Bu)₃ or (t-Bu)₂Ph and X = PF₆ or ClO₄) in ~ 90% yield.

Deprotonation of the hydridoamminebis(phosphine)platinum(II) complexes by NH_3

(a) Ammonia was bubbled through a solution of *trans*-[PtH(NH₃)(PR₃)₂]X (R₃ = (t-Bu)₃ or (t-Bu)₂Ph; X = PF₆ or ClO₄) (0.3 mmol) in acetone (5 ml) at room temperature. A white solid gradually precipitated which was filtered, washed with acetone and dried. The ¹H and ³¹P NMR spectra of these solids were identical to those of authentic samples of Pt[P(t-Bu)₃]₂ or Pt[P(t-Bu)₂Ph]₂. Yield: ~90%.

(b) The complexes *trans*-[PtH(NH₃)(PR₃)₂]X (R₃ = (t-Bu)₃ or (t-Bu)₂Ph; X = PF₆ or ClO₄) were recovered unchanged when ammonia was passed into their solutions in dichloromethane at room temperature.

Reactions of the hydridoamminebis(phosphine)platinum(II) complexes with CO

(a) Carbon monoxide was slowly bubbled into solutions of *trans*-[PtH(NH₃)-(PR₃)₂]X (R₃ = (t-Bu)₃ or (t-Bu)₂Ph) (0.2 g) in acetone (5 ml) at room temperature. An orange-red solid gradually separated which was filtered and washed with acetone. The IR and ³¹P NMR spectral data for the orange-red solid were similar to an authentic sample of [Pt(PR₃)(CO)]₃. Yield: > 75%.

(b) When dichloromethane was used as the solvent for the above reaction, corresponding carbonyl complexes *trans*-[PtH(CO)(PR₃)₂]X were obtained in > 90% yield.

Reaction of $Pt(PPh_3)_3$ with NH_4PF_6

(a) A mixture of $Pt(PPh_3)_3$ (0.5 mmol) and NH_4PF_6 (0.6 mmol) in THF (10 ml) was stirred at room temperature for 48 h and then at ~ 40°C for 12 h. The solvent was removed and the residue was dissolved in dichloromethane (5 ml). The resulting solution was passed through a small Florisil column, diluted with pentane and cooled to give colourless crystals of $[PtH(PPh_3)_3]PF_6$ in 90% yield.

(b) $(Ph_3P)_3Pt$ and NH_4PF_6 were allowed to react in THF as in a. Pentane was added to the THF solution and the resulting precipitate was filtered, washed with pentane and benzene and then extracted with dichloromethane. The ³¹P-(¹H) NMR spectrum of the filtrate was identical to that of [(PPh_3)_3PtH]PF_6.

Attempted reactions of $[PtH(PPh_3)_3]PF_6$ with NH_3 and CO

(a) Ammonia was passed through solutions of $[PtH(PPh_3)_3]PF_6$ (100 mg) in dichloromethane or acetone (4 ml) for ~ 2 h. The ³¹P NMR spectrum of the solution was identical to that of $[PtH(PPh_3)_3]PF_6$ showing no reaction.

(b) Carbon monoxide was bubbled through a solution of $[PtH(PPh_3)_3]PF_6$ (80

mg) in dichloromethane or acetone (5 ml) for 2 h at room temperature. The ${}^{31}P$ NMR spectrum of the resulting solution was identical to that of $[PtH(PPh_3)_3]PF_6$.

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