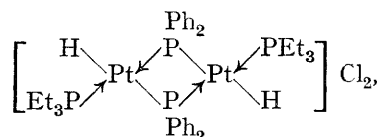


Preparation and Reactivity of Cationic Platinum(II) Hydrido-complexes

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The preparation is reported of the cationic hydrides $[\text{Pt}(\text{PEt}_3)_2 \text{diphos}(\text{H})]\text{Cl}$ (I) and $[\text{Pt}(\text{PEt}_3)_2(\text{PR}_3)\text{H}]\text{X}$ (II) ($\text{R} = \text{Et}$ or Ph ; $\text{X} = \text{ClO}_4$ or BF_4) from the reaction of 1,2-bis(diphenylphosphino)ethane (diphos) or monodentate phosphine PR_3 with *trans*- $[\text{Pt}(\text{PEt}_3)_2(\text{H})\text{Cl}]$ in benzene or dichloroethane. Conductivity and i.r. spectral data are given. The reaction of (I) with hydrogen chloride in dichloroethane leads to the complex $[\text{Pt}(\text{PEt}_3)_2 \text{diphos Cl}]\text{Cl}$ with evolution of hydrogen. (I) reacts with SnCl_2 in methanol to give the four-co-ordinate $[\text{Pt}(\text{PEt}_3)_2 \text{diphos H}]\text{SnCl}_3$.

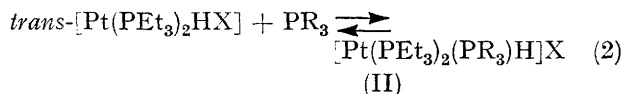
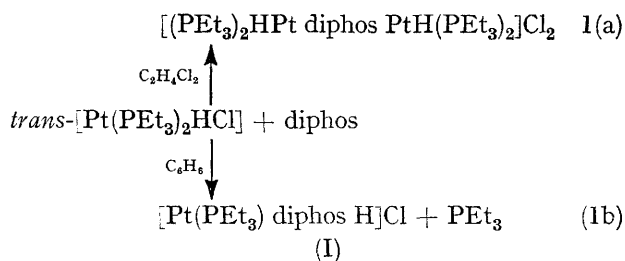
FOUR-CO-ORDINATE, positively charged platinum(II) complexes having σ metal-carbon, metal-hydride, or π metal-olefin bonds are somewhat unstable and very few have been isolated hitherto. Among the hydrides the first example¹ was a cationic, phosphido-bridged dihydrido-complex,



obtained from the reaction of *trans*- $[\text{Pt}(\text{PEt}_3)_2(\text{H})\text{Cl}]$ with PPh_2 . The complexes $[\text{Pt}(\text{PPh}_3)_3\text{H}]\text{X}$ were obtained by oxidative addition of hydrogen halides or pseudo-halides on platinum(0)-phosphine complexes.² $[\text{Pt}(\text{PEt}_3)_2(\text{diphos})\text{H}]\text{Cl}$ [diphos = 1,2-bis(diphenylphosphino)ethane] has been obtained³ by the reaction of $[\text{Pt}(\text{PEt}_3)_2 \text{diphos}(\text{GeMe}_3)]\text{Cl}$ with hydrogen chloride.

We have prepared this hydride and $[\text{Pt}(\text{PEt}_3)_2(\text{PR}_3)\text{H}]\text{X}$ ($\text{R} = \text{Et}$ or Ph ; $\text{X} = \text{ClO}_4^-$ or BF_4^-) by the reaction of *trans*- $[\text{Pt}(\text{PEt}_3)_2(\text{H})\text{Cl}]$ with 1,2-bis(diphenyl-

phosphine)ethane and with a monodentate phosphine, PR_3 . The reactions



($\text{X} = \text{Cl}$ or CN ; $\text{R}_3 = \text{Et}_3$, Bu_3 , EtPh_2 , or Ph_3)

were followed by conductometric titrations (in dichloroethane) and also by i.r. and u.v. spectroscopy where possible. Analytical data, selected i.r. frequencies, and

¹ J. Chatt and J. M. Davidson, *J. Chem. Soc.*, 1964, 2433.

² F. Cariati, R. Ugo, and F. Bonati, *Inorg. Chem.*, 1966, **5**, 1128.

³ F. Glockling and K. A. Hooton, *J. Chem. Soc. (A)*, 1968, 826.

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molar conductivities, of the new complexes are listed in Table 1. The complexes are soluble in dichloromethane, dichloroethane, and alcohol, insoluble in benzene and ether. The molar conductivities ($10^{-3}M$) in dichloroethane are in the range $35\text{--}45\text{ ohm}^{-1}\text{ cm}^2\text{ mole}^{-1}$ and are therefore indicative of uni-univalent electrolytes. The

tendency to form cationic hydrides is in the order: $\text{PEt}_3 \approx \text{PBU}_3 > \text{PEtPh}_2 > \text{PPh}_3$.

Reaction (1) was carried out also in benzene, dichloromethane, and ethanol. The nature of the solvent affects both the course and the rate of the reaction. Thus, the reaction proceeds according to equation (1a)

TABLE 1

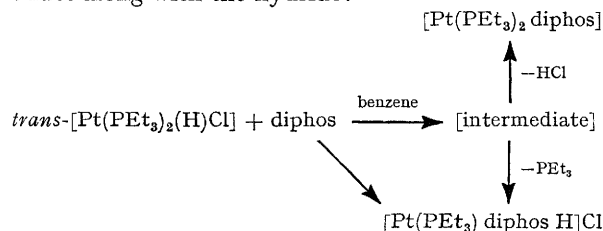
Analytical data, spectra, and molar conductivities of hydrido- and phosphino-platinum(II) complexes

Complex	Colour	M.p. (°)	Found (%)			Required (%)			$\nu(\text{Pt-H})$ (cm^{-1})	Molar conductivity * at 25° ($\text{ohm}^{-1}\text{ cm}^2\text{ mole}^{-1}$)
			C	H	Cl	C	H	Cl		
$[\text{Pt}(\text{PEt}_3)_2\text{diphos H}]\text{Cl}$	White	107—109 †	51.79	5.43	4.85	51.39	5.35	4.85	2012	35
$[\text{Pt}(\text{PEt}_3)_2(\text{PPh}_3)\text{H}]\text{BF}_4$	White	164—166	46.46	5.88	—	46.12	5.89	—	2072	42
$[\text{Pt}(\text{PEt}_3)_2\text{H}]\text{ClO}_4$	White	158—160	—	—	—	33.27	7.08	—	2066	37
$[\text{Pt}(\text{PEt}_3)_2\text{diphos H}](\text{SnCl}_3)$	Pale yellow	164—166	39.85	4.08	11.15	41.00	4.26	11.34	2050	
<i>trans</i> - $[\text{Pt}(\text{PEt}_3)_2(\text{H})\text{Cl}]$	White	—	—	—	—	—	—	—	2222 ‡	
$[\text{Pt}(\text{PEt}_3)_2\text{diphos Cl}]\text{Cl}$	White	271 †	48.90	4.90	9.10	49.13	4.98	9.06	—	36.2

* At 25° in dichloroethane. † Decomp. ‡ I.r. spectrum in Nujol mull from ref. 4.

plot of the conductometric titration of *trans*- $[\text{Pt}(\text{PEt}_3)_2(\text{H})\text{Cl}]$ with diphos [reaction (1a)] shows one equivalence point corresponding to addition of *ca.* one half mole of diphos per platinum atom, after which the conductivity remains constant. In dichloroethane a binuclear complex is probably formed as the main product, together with (I). With monodentate phosphines, PR_3 , the conductivity plot was slightly curved and did not reach a constant value until *ca.* 2 moles of ligand per metal atom has been added (Figure). This dependence of the

in $\text{C}_2\text{H}_4\text{Cl}_2$, whereas in benzene the platinum(0) complex $[\text{Pt}(\text{PEt}_3)_2\text{diphos}]$ is probably formed as a side product along with the hydride:



SCHEME 1

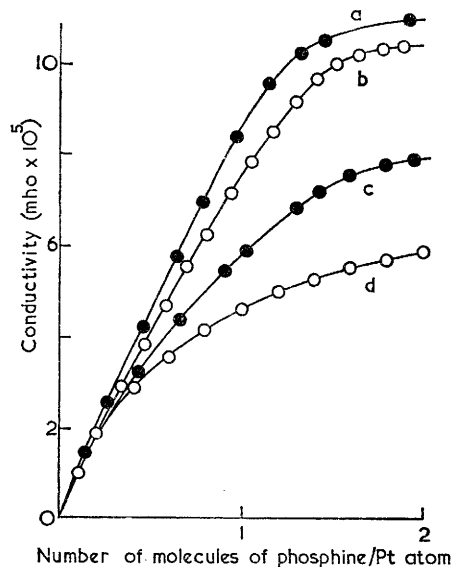


FIGURE Changes in conductivity of a solution of *trans*- $[\text{Pt}(\text{PEt}_3)_2\text{HCl}]$ in dichloroethane when a monodentate phosphine is added in increasing amounts; a, PEt_3 ; b, PBU_3 ; c, PEtPh_2 ; and d, PPh_3 .

shape of the conductometric curves on the nature of the phosphine indicates that a balanced equilibrium is involved depending on the entering ligand. The

Reaction (1) is instantaneous in dichloroethane, and much slower in benzene. In the latter medium the ionic hydride begins to precipitate after a few minutes. From the equilibrium reaction (2) in dichloroethane and extracting with aqueous sodium tetrafluoroborate we were able to isolate in good yield the hydride $[\text{Pt}(\text{PEt}_3)_2(\text{PPh}_3)\text{H}]^+$. Its i.r. spectrum (Nujol mull) shows a strong band at 2072 cm^{-1} attributable to $\nu(\text{Pt-H})$. The analogous derivative with triethylphosphine was extracted with sodium perchlorate [$\nu(\text{Pt-H}) = 2066\text{ cm}^{-1}$].

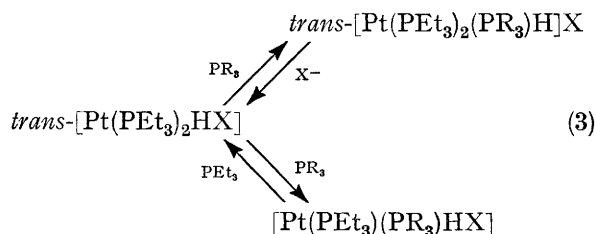
Trans- $[\text{Pt}(\text{PEt}_3)_2(\text{H})\text{Cl}]$ reacts much faster than the corresponding cyano-complex with monodentate phosphines in dichloroethane and the equilibrium lies farther to the right, reflecting the greater tendency of CN^- over Cl^- to bond with platinum.⁵ This is confirmed by comparing the conductivity of the equilibrated solutions with those of equimolar solutions of the ionic hydrides (II) in the same solvent. The fact that the reactions of type (2) lead to equilibria would seem to indicate some reluctance of four-co-ordinate Pt^{II} complexes to assume a positive charge.

In the reaction of *trans*- $[\text{Pt}(\text{PEt}_3)_2(\text{H})\text{X}]$ with PBU_3 and PPh_3 in dichloroethane, the replacement of X is accompanied by a slower, partial displacement of the triethylphosphine originally bonded in the platinum hydride. In fact, an equilibrated reaction mixture ($\text{Pt}:\text{PPh}_3 = 1$) smelled strongly of triethyl phosphine

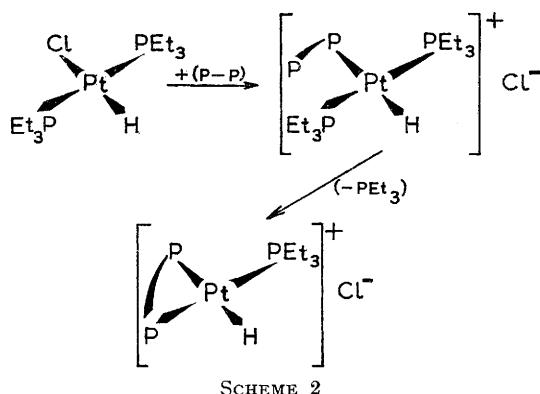
⁴ J. Chatt, *Proc. Chem. Soc.*, 1962, 318.

⁵ F. Basolo and R. G. Pearson, 'Mechanism of Inorganic Reactions,' Wiley, New York, 1967.

and displayed a progressive decrease in conductivity, with time, to a limiting value, this change being accompanied by a slight change in the absorption band due to $\nu(\text{Pt-H})$. Therefore, the equilibria which take place in the reaction mixture is outlined as reaction (3).



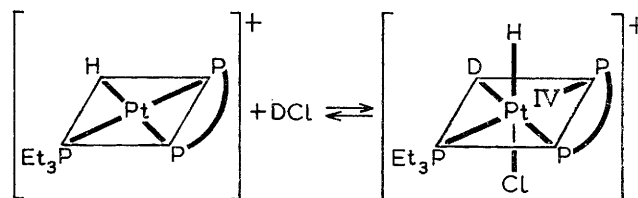
By analogy with reaction (2), in which the neutral hydride is converted into a cationic one with a monodentate phosphine, the corresponding reaction path with diphos in dichloroethane might be inferred to involve first the substitution of chloride *trans* to the hydride, followed by closure of the chelate ring (Scheme 2).



The reaction of *trans*-[Pt(PEt₃)₂(H)Cl] with diphos in benzene probably involves formation of the same intermediate as in Scheme 2 in which the diphosphine is bonded to platinum via a single phosphorous atom. This intermediate may eliminate either the hydride ligand as hydrogen chloride leading to the platinum(0) complex, [Pt(PEt₃)₂ diphos], or the monodentate phosphine affording [Pt(PEt₃) diphos H]Cl as shown in Scheme 1.

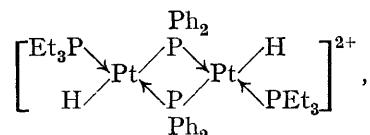
The assignment of the band at 2012 cm⁻¹ in the complex [Pt(PEt₃) diphos H]Cl as $\nu(\text{Pt-H})$ was based on the expected shift of such band in the corresponding deuteriated complex [$\nu(\text{Pt-H})/\nu(\text{Pt-D}) = 1.39$]. The deuteriocomplex [Pt(PEt₃) diphos D]Cl could not be prepared by deuteration of the parent, ionic hydrido-complex with DCl in CH₃OD (10⁻²M), *i.e.*, following the procedure of Halpern *et al.*⁶ for non ionic hydrides. We prepared first *trans*-[Pt(PEt₃)₂(D)Cl]⁷ and then treated it with the diphosphine in benzene to obtain [Pt(PEt₃) diphos D]Cl. This deuteriocomplex shows

(Pt-D) at 1441 cm⁻¹ and $\delta(\text{Pt-D})$ at 554 cm⁻¹ (halogenocarbon mull). The failure to obtain the deuterio-complex from [Pt(PEt₃) diphos H]Cl by Halpern's method is probably due to the positive charge in this complex. In fact the suggested mechanism for these H-D exchange reactions involves formation of a labile six-co-ordinate Pt^{IV} intermediate through oxidative addition of DCl on the initial four-co-ordinate Pt^{II} complex (Scheme 3). The presence of a positive charge on this latter would render such a process less feasible.



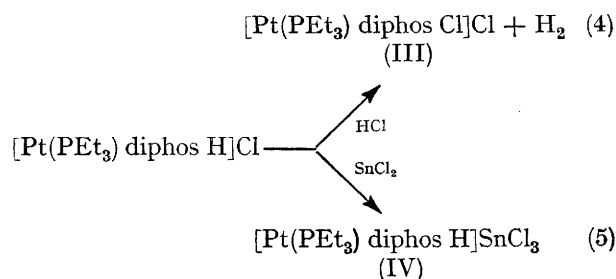
SCHEME 3

The Pt-H stretching frequencies of cationic platinum hydrides (Table 2) call for some comments. The values of $\nu(\text{Pt-H})$ in *trans*-[Pt(PEt₃)₂(PR₃)H]⁺ are rather low, differing very little from one another, and are close to that in *trans*-[Pt(PEt₃)₂H(CN)] (2072 cm⁻¹).⁸ Since $\nu(\text{Pt-H})$ bears an inverse relationship to the *trans*-effect of the ligand *trans* to the hydride,⁹ such low values are in keeping with the high *trans*-effect of the phosphines. At present we cannot suggest any interpretation of the observed decrease of $\nu(\text{Pt-H})$ (*ca.* 50 cm⁻¹) in [Pt(PEt₃) diphos H]⁺ relative to cationic hydrides having monodentate phosphines. In this regard it should be noted that the complex



wherein the bridging phosphido-group is *trans* to the hydride, has a strikingly low $\nu(\text{Pt-H})$ (2005 cm⁻¹). The rather high value for $\nu(\text{Pt-H})$ in the recently reported *trans*-[Pt(PEt₃)₂(CO)H]⁺ (2167 cm⁻¹),¹⁰ is hardly related to the high *trans*-effect of CO even in the light of a strong coupling of the CO and Pt-H vibrations.¹⁰

Chemical Properties of [Pt(PEt₃) diphos H]Cl, (I).—The complex undergoes the reactions (4) and (5).



⁶ J. Halpern and C. D. Falk, 'Exchange Reactions,' International Atomic Energy Agency, Vienna, 1965, 191; *J. Amer. Chem. Soc.*, 1965, **87**, 3523.

⁷ P. Uguagliati and W. H. Baddley, *J. Amer. Chem. Soc.*, 1968, **90**, 5446.

⁸ J. Chatt and B. L. Shaw, *J. Chem. Soc.*, 1962, 5075.

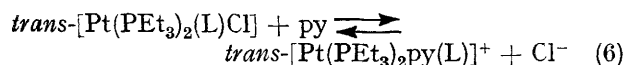
⁹ J. Chatt, L. A. Duncanson, and B. L. Shaw, *Chem. and Ind.*, 1958, 859.

¹⁰ M. J. Church and M. J. Mays, *Chem. Comm.*, 1968, 435.

The far-i.r. spectrum of the product (III) shows a band at 306 cm.⁻¹ attributable to $\nu(\text{Pt}-\text{Cl})$. The molar conductivity in dichloroethane (36.4 ohm⁻¹ cm.² mole⁻¹) corresponds to that of a uni-univalent electrolyte. The formulation was confirmed by elemental analysis. Reaction (4) proceeds very slowly in methanol, appreciably faster in dichloroethane, possibly owing to ionization-solvation phenomena. Compound (I) reacts with tin(II) chloride in methanol to give a crystalline yellow product of stoichiometry $[\text{Pt}(\text{PET}_3)_2\text{diphos}(\text{H})\text{ClSnCl}_2]$. Its i.r. spectrum shows $\nu(\text{Pt}-\text{H})$ at 2050 cm.⁻¹ (Nujol mull), and it may be formulated either as a five-co-ordinate complex, $[\text{Pt}(\text{PET}_3)_2\text{diphos}(\text{H})\text{Cl}(\text{SnCl}_3)]$ or as a four-co-ordinate cationic platinum hydride, $[\text{Pt}(\text{PET}_3)_2\text{diphos}(\text{H})\text{SnCl}_3]$. The product shows two i.r. bands at 276 and 260 cm.⁻¹ which can be attributed to $\nu(\text{Sn}-\text{Cl})$ and are very close to those (289 and 252 cm.⁻¹) found for $[(\text{PPh}_3)_4\text{As}][\text{SnCl}_3]$.¹¹ In the complex $[\text{Pt}(\text{PET}_3)_2\text{H}(\text{SnCl}_3)]$ $\nu(\text{Sn}-\text{Cl})$ is found at a higher wavelength (330 cm.⁻¹, doublet).¹² Thus, we think that the complex is best formulated as $[\text{Pt}(\text{PET}_3)_2\text{diphos}(\text{H})\text{SnCl}_3]$ (IV). Accordingly, a dichloroethane solution of (IV) is conducting and its i.r. spectrum shows the $\nu(\text{Pt}-\text{H})$ band at 2038 cm.⁻¹, which is identical with that of $[\text{Pt}(\text{PET}_3)_2\text{diphos}(\text{H})\text{Cl}]$. $\nu(\text{Pt}-\text{H})$ in the complex (IV) (2050 cm.⁻¹) is higher than the corresponding value found for the initial compound (I) (2012 cm.⁻¹), and this may be due to the effect of the different counter-anion.

Unlike *trans*- $[\text{Pt}(\text{PET}_3)_2(\text{H})\text{Cl}]$,¹⁰ $[\text{Pt}(\text{PET}_3)_3\text{H}]\text{X}$ does not react with carbon monoxide under ambient conditions.

It is a general feature of the chemistry of platinum complexes that the most stable σ -bonded platinum hydride and platinum-carbon complexes occur when the central metal is linked to π -accepting groups^{4,13} which are able to remove electron charge from the metal. Therefore, one would expect that positively charged, σ -bonded, platinum hydride and platinum-carbon complexes would be more stable than neutral ones. Reactions of type (2) as well as of type (6) ($\text{L} = \text{H}$,



Me, or Ph) are reversible and the equilibria lie far over to the left and are established very rapidly.¹⁴ This thermodynamic instability is at variance with what would have been expected from the preceding reasoning, and renders more difficult a full understanding of the factors affecting the stability of Pt-H and Pt-C bonds. We surmise that the formal positive charge in such cationic complexes as $[\text{Pt}(\text{PET}_3)_3\text{H}]^+$ is partially delocalized over the phosphine groups owing to the σ donor ability of these ligands. That this is so is suggested by a comparison of $\nu(\text{Pt}-\text{H})$ for complexes *trans*- $[\text{Pt}(\text{PR}_3)_2(\text{PPh}_3)\text{H}]^+$ and *trans*-

$[\text{Pt}(\text{PR}_3)_2(\text{H})\text{Cl}]$ (Table 2) which shows that substitution of PET_3 with PPh_3 in the phosphine PR_3 *cis* to hydride brings about much the same increase in $\nu(\text{Pt}-\text{H})$ for both types of complexes ($\Delta\nu$ ca. 40 cm.⁻¹). If one assumes that such increase is due to an increased H→Pt bond-strength caused by a larger electron shift from platinum

TABLE 2
Platinum-hydrogen stretching frequencies (cm.⁻¹) of cationic hydridoplatinum(II) complexes

Complex	$\nu(\text{Pt}-\text{H})$ ^a	$\nu(\text{Pt}-\text{H})$ ^b
$[\text{Pt}(\text{PET}_3)_2(\text{PET}_3)\text{H}]^+$	2066	2090
$[\text{Pt}(\text{PET}_3)_2(\text{PBu}_3)\text{H}]^+$		2090
$[\text{Pt}(\text{PET}_3)_2(\text{PPh}_3)\text{H}]^+$	2072	2098
$[\text{Pt}(\text{PPh}_3)_2(\text{PPh}_3)\text{H}]^+$	2112 ^c	
$[\text{Pt}(\text{PET}_3)_2(\text{CO})\text{H}]^+$	2167 ^d	
$[\text{Pt}(\text{PET}_3)_2\text{diphos}(\text{H})]^+$	2012	2038
<i>trans</i> - $[\text{Pt}(\text{PET}_3)_2\text{H}(\text{CN})]$	2072	
<i>trans</i> - $[\text{Pt}(\text{PET}_3)_2\text{HCl}]$		2183 ^e
<i>trans</i> - $[\text{Pt}(\text{PPh}_3)_2\text{HCl}]$		2224 ^e

^a Nujol mulls. ^b In dichloroethane solution. ^c Ref. 2.
^d Ref. 10. ^e Data for hexane solution, taken from refs. 8 and 9.

toward the more electronegative phosphine PPh_3 ,¹⁵ one would conclude that the positive charge does not actually affect the donor ability of the metal, *i.e.*, the metal should meet the electronic requirements of electronegative ligands to an extent independent of the charge on the complex. This in turn would indicate that such charge is largely delocalized.

EXPERIMENTAL

Materials.—Triethylphosphine,¹⁶ *cis*-dichloro-[1,2-bis(diphenylphosphino)ethane]platinum(II),¹⁷ *trans*-hydrido-chlorobis(triethylphosphine)platinum(II),⁸ *trans*-hydrido-cyanobis(triethylphosphine)platinum(II),⁸ and *trans*-deuteriochlorobis(triethylphosphine)platinum(II)⁷ were prepared according to literature methods. DCl in D₂O (37%), D₂O, and CH₃OD were from Fluka. Technical grade tri-*n*-butylphosphine and ethyldiphenylphosphine (Strem) were purified by distillation under nitrogen. All other materials were reagent grade chemicals and were used without further purification.

I.r. spectra were recorded on a Perkin-Elmer 621 double-beam grating spectrophotometer. Conductivities were measured by means of a conductivity bridge LKB 3216 B at 25°.

Preparation of Complexes.—(a) *Hydrido*-[1,2-bis(diphenylphosphino)ethane](triethylphosphine)platinum(II) Chloride, $[\text{Pt}(\text{PET}_3)_2\text{diphos}(\text{H})\text{Cl}]$. To *trans*- $[\text{Pt}(\text{PET}_3)_2(\text{H})\text{Cl}]$ (0.93 g.; 2 mmoles) in benzene (20 ml.) was added dropwise with stirring a saturated solution of diphos (0.79 g.) in benzene to give a yellow solution immediately. The white crystalline solid which precipitated after a few min. was stirred for $\frac{1}{2}$ hr. when $[\text{Pt}(\text{PET}_3)_2\text{diphos}(\text{H})\text{Cl}]$ was filtered off, repeatedly washed with ether, and dried *in vacuo* (1.35 g., 90%).

The yellow benzene filtrate was evaporated to dryness leaving a crude yellow material which could not be recrystallized, and showed no i.r. band in the $\nu(\text{Pt}-\text{H})$ region.

¹⁴ F. Basolo, J. Chatt, H. B. Gray, R. G. Pearson, and B. L. Shaw, *J. Chem. Soc.*, 1961, 2207.

¹⁵ J. Chatt, L. A. Duncanson, B. L. Shaw, and L. M. Venanzi, *Discuss. Faraday Soc.*, 1958, 26, 131.

¹⁶ H. Hibbert, *Ber.*, 1906, 39, 160.

¹⁷ A. D. Westland, *J. Chem. Soc.*, 1965, 3060.

¹¹ D. F. Shriver and M. P. Johnson, *Inorg. Chem.*, 1967, 6, 1265.

¹² R. V. Lindsay, G. W. Parshall, V. G. Stolberg, *J. Amer. Chem. Soc.*, 1965, 87, 658.

¹³ J. Chatt and B. L. Shaw, *J. Chem. Soc.*, 1959, 705.

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It was dissolved in benzene and treated with hydrogen chloride to give *cis*-dichloro-[1,2-bis(diphenylphosphino)ethane]platinum(II) identified by comparison with an authentic sample.¹⁷

(b) *Hydridotris(triethylphosphine)platinum(II) Perchlorate*, $[\text{Pt}(\text{PEt}_3)_3\text{H}]\text{ClO}_4$. To *trans*- $[\text{Pt}(\text{PEt}_3)_2(\text{H})\text{Cl}]$ (0.93 g., 2 mmoles) in 1,2-dichloroethane (10 ml.) was added with stirring a slight excess of triethylphosphine (0.4 ml.) under nitrogen. After 10 min. a cold saturated aqueous solution of sodium perchlorate was added. The mixture was shaken vigorously for *ca.* 0.5 hr. and set aside when the organic layer separated and was removed and evaporated under reduced pressure. The colourless oil which remained was dissolved in the minimum of dichloromethane and stirred with hexane (10 ml.). Removal of most of the organic liquid gave the white crystalline product $[\text{Pt}(\text{PEt}_3)_3\text{H}]\text{ClO}_4$ (0.908 g., 85%).

(c) *trans-Hydridobistriethylphosphine(triphenylphosphine)platinum(II) Tetrafluoroborate*, *trans*- $[\text{Pt}(\text{PEt}_3)_2(\text{PPh}_3)\text{H}]\text{BF}_4$. This was prepared similarly (87%).

(d) *Deuteriotriethylphosphine[1,2-bis(diphenylphosphino)ethane]platinum(II) Chloride*, $[\text{Pt}(\text{PEt}_3)_2\text{D}]\text{Cl}$. Preparation (a) was carried out using *trans*- $[\text{Pt}(\text{PEt}_3)_2(\text{D})\text{Cl}]$ as the starting material (85%).

Reactions of $[\text{Pt}(\text{PEt}_3)_2\text{H}]\text{Cl}$, (I).—(a) *With hydrogen chloride*. Dry hydrogen chloride was bubbled for 2 hr. into a solution of (I) (0.2 g.) in 1,2-dichloroethane (10 ml.). The solution was concentrated under reduced pressure, leaving $[\text{Pt}(\text{PEt}_3)_2\text{H}]\text{Cl}$ (0.195 g.).

(b) *With tin(II) chloride*. To a solution of (I) (0.75 g.) in methanol (10 ml.) was added dropwise with stirring a concentrated solution of SnCl_2 (0.19 g.) in methanol (10 ml.). The solution was then stirred for 1 hr. and the pale yellow product, $[\text{Pt}(\text{PEt}_3)_2\text{H}](\text{SnCl}_3)$, which precipitated was filtered off and recrystallized from methanol (0.55 g.).

This work was supported by the Italian Research National Council (C.N.R., Rome).

[8/1911 Received, December 23rd, 1968]