METHYL TRANSFER REACTIONS BETWEEN PLATINUM(II) AND PLATINUM(IV) COMPLEXES AND SOME APPARENT METHYL FOR HYDRIDE EXCHANGE REACTIONS

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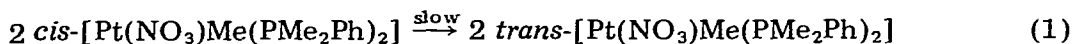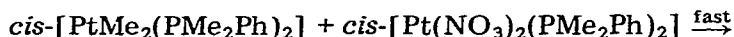*Donnan Laboratories, The University of Liverpool, Liverpool L69 3BX (Great Britain)*

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Summary

cis-$[PtMe_2(PMe_2Ph)_2]$ reacts with $[PtX_2Me_2(PMe_2Ph)_2]$ (X = I, NO_2, NO_3) to give $[PtXMe(PMe_2Ph)_2]$ and $[PtXMe_3(PMe_2Ph)_2]$. When X = I, the isomer *cis-cis-trans*-$[PtX_2Me_2(PMe_2Ph)_2]$ fails to react with *cis*-$[PtMe_2(PMe_2Ph)_2]$ though the *trans-cis-cis* isomer does react. By labelling studies, it is shown that when X = NO_3 the reaction occurs by methyl for nitrate exchange rather than by a redox mechanism, though when X = NO_2 the situation was more complex. *trans*-$[PtIHL_2]$ (L = PMe_3 or PMe_2Ph) reacted with [AuMeL] or *cis*-$[PtMe_2L_2]$ to give *trans*-$[PtIMeL_2]$, a reaction which appears to involve methyl for hydride exchange.

Introduction

Reactions in which an alkyl or aryl group is exchanged between two platinum(II) centres have been studied in considerable detail (e.g. eq. 1) [1—4].

cis-$[PtMe_2(PMe_2Ph)_2]$ + cis-$[Pt(NO_3)_2(PMe_2Ph)_2]$ $\xrightarrow{\text{fast}}$

$$2 \text{ cis-}[Pt(NO_3)Me(PMe_2Ph)_2] \xrightarrow{\text{slow}} 2 \text{ trans-}[Pt(NO_3)Me(PMe_2Ph)_2] \qquad (1)$$
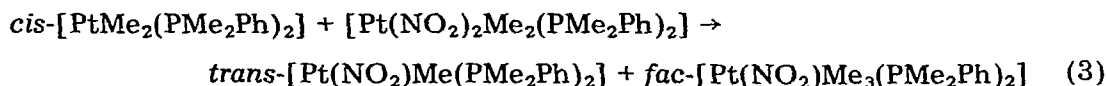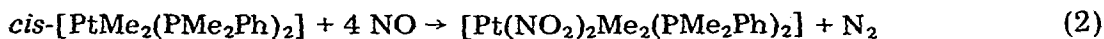
The reactions occur stereospecifically and the mechanism of reaction has been discussed.
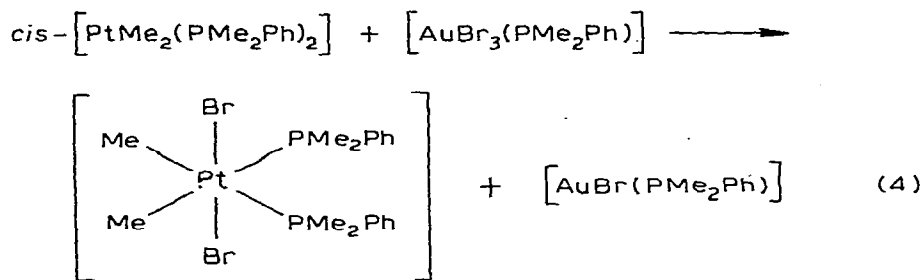
In contrast, methyl transfer reactions between platinum(II) and platinum(IV) centres have been very little studied. It was shown that the products of reaction of nitric oxide with cis-$[PtMe_2(PMe_2Ph)_2]$ arose through the sequence of

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reactions in eq. 2 and 3, involving methyl for nitro exchange between cis-$[PtMe_2(PMe_2Ph)_2]$ and $[Pt(NO_2)_2Me_2(PMe_2Ph)_2]$ [5].

$$cis\text{-}[PtMe_2(PMe_2Ph)_2] + 4\ NO \rightarrow [Pt(NO_2)_2Me_2(PMe_2Ph)_2] + N_2 \qquad (2)$$

$$cis\text{-}[PtMe_2(PMe_2Ph)_2] + [Pt(NO_2)_2Me_2(PMe_2Ph)_2] \rightarrow$$

$$trans\text{-}[Pt(NO_2)Me(PMe_2Ph)_2] + fac\text{-}[Pt(NO_2)Me_3(PMe_2Ph)_2] \qquad (3)$$

Reactions of this type could proceed in two ways. Thus, a redox mechanism is possible in which a methyl and nitro group are transferred from platinum(IV) to platinum(II), in an analogous fashion to the oxidation of methylplatinum(II) complexes by gold(III) halide complexes (eq. 4).[2].

$$cis-\left[PtMe_2(PMe_2Ph)_2\right] + \left[AuBr_3(PMe_2Ph)\right] \longrightarrow$$

$$\left[\begin{array}{c} Br \\ Me \diagdown \mid \diagup PMe_2Ph \\ Pt \\ Me \diagup \mid \diagdown PMe_2Ph \\ Br \end{array}\right] + \left[AuBr(PMe_2Ph)\right] \qquad (4)$$
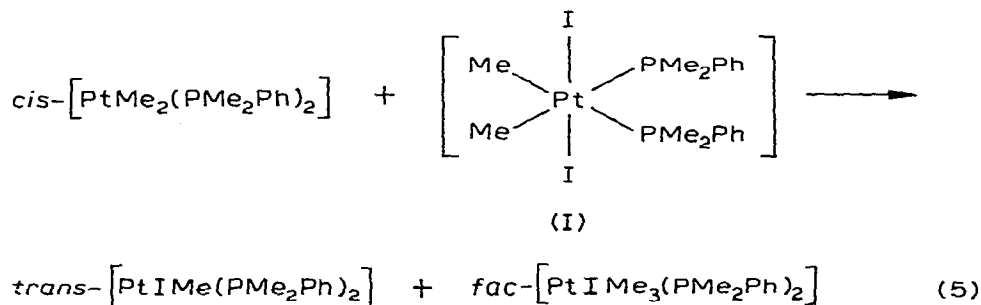
Alternatively a simple methyl for nitro exchange could occur. By analogy with the proposed mechanisms of methyl transfer reactions between platinum(II) centres and methyl transfer to octahedral ruthenium(II) complexes, it is expected that dissociation of a ligand from platinum(IV) would be necessary to create a vacant coordination site before the exchange reaction could occur [2—4,6].
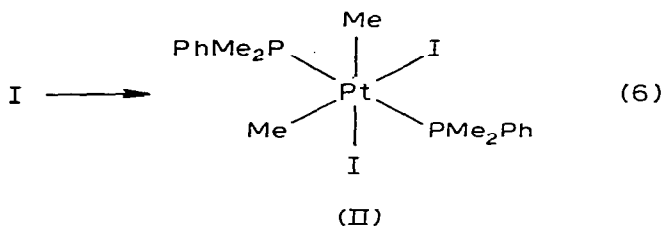
In an attempt to resolve these problems, we have studied the course of reaction between cis-$[PtMe_2(PMe_2Ph)_2]$ and some platinum(IV) complexes $[PtX_2Me_2(PMe_2Ph)_2]$ (X = I, NO_2 or NO_3). A preliminary account of some of this work has been published [3]. We also report some reactions of hydrido-platinum(II) complexes which may involve hydride for methyl exchange.

Methyl exchange between platinum(II) and platinum(IV)

The complex cis-$[PtMe_2(PMe_2Ph)_2]$ is a strong methylating agent and will react with several complexes $[PtX_2Me_2(PMe_2Ph)_2]$ (X = I, NO_2 or NO_3), though the reactions are not simple. For example, reaction occurs with the isomer of $[PtI_2Me_2(PMe_2Ph)_2]$ with structure I according to eq. 5.

$$cis-\left[PtMe_2(PMe_2Ph)_2\right] + \left[\begin{array}{c} I \\ Me \diagdown \mid \diagup PMe_2Ph \\ Pt \\ Me \diagup \mid \diagdown PMe_2Ph \\ I \end{array}\right] \longrightarrow$$

(I)

$$trans-\left[PtIMe(PMe_2Ph)_2\right] + fac-\left[PtIMe_3(PMe_2Ph)_2\right] \qquad (5)$$

However, this reaction occurred at a similar rate as the isomerisation of I to the more stable isomer II (eq. 6).

I ⟶

$$\begin{array}{c}\text{Me}\\ \text{PhMe}_2\text{P}\diagdown\ \big|\ \diagup\text{I}\\ \text{Pt}\\ \text{Me}\diagup\ \big|\ \diagdown\text{PMe}_2\text{Ph}\\ \text{I}\end{array}$$
(6)

(II)

Since II did not react with *cis*-[PtMe$_2$(PMe$_2$Ph)$_2$], the final products after 2 days were *trans*-[PtIMe(PMe$_2$Ph)$_2$], *fac*-[PtIMe$_3$(PMe$_2$Ph)$_2$], (II) and unchanged *cis*-[PtMe$_2$(PMe$_2$Ph)$_2$].

The different reactivities of isomeric platinum(IV) species towards methylation has a precedent in the methylation of isomers of [RuCl$_2$(CO)$_2$(PMe$_2$Ph)$_2$] by HgMe$_2$. These reactions were shown to occur after dissociation of a carbonyl ligand to generate a vacant coordination site at ruthenium. The rate of reaction was determined by the ease of dissociation of the carbonyl ligand which, in turn, was determined by the *trans*-influence of the ligand *trans* to carbonyl [6]. The results for the platinum(IV) complexes can be explained in a similar way if the rate-determining step is loss of a phosphine ligand from platinum(IV). In isomer I the phosphine ligands are *trans* to methyl, which has a very high *trans*-influence [7], whereas in (II) the phosphine ligands are mutually *trans* and are expected to dissociate less readily. The proposed mechanism is shown in Scheme 1. According to this scheme the reaction should be retarded by added
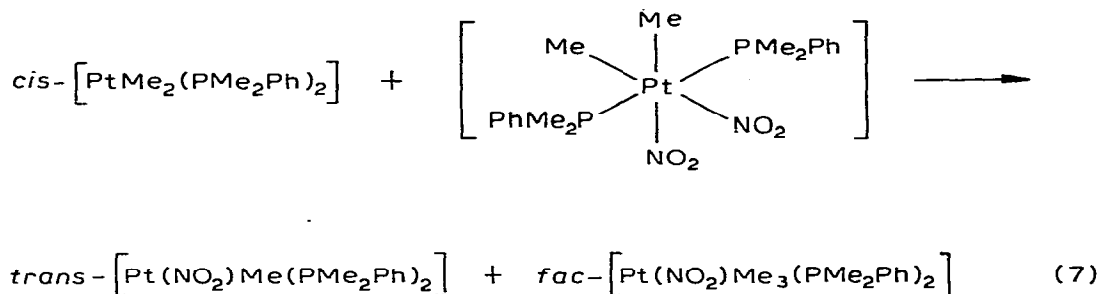
SCHEME 1
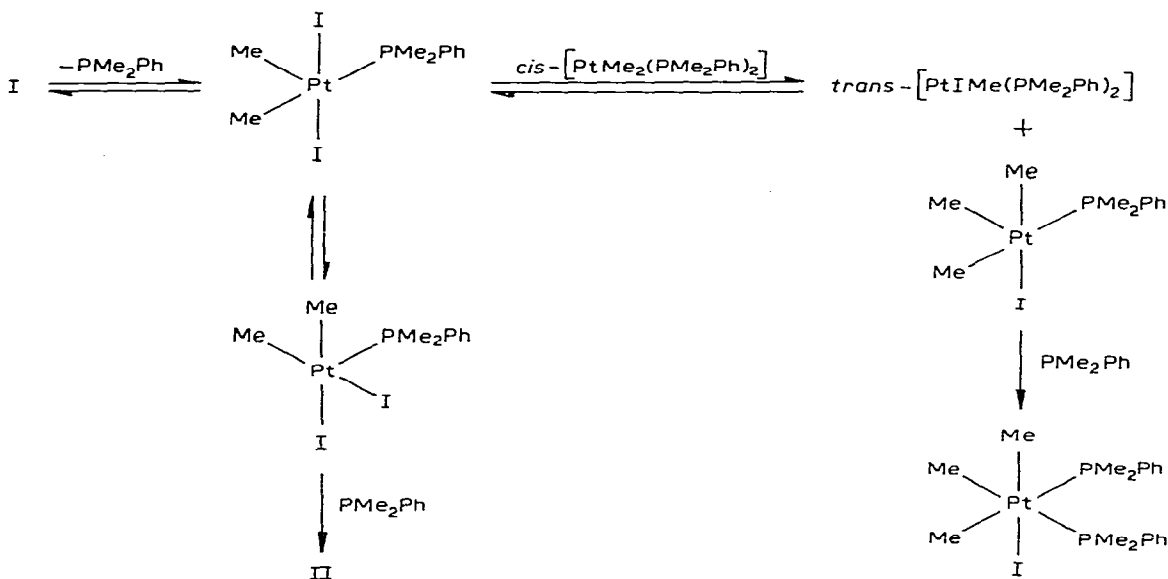
PMe$_2$Ph, since this would lower the steady state concentration of the 5 coordinate intermediates. We have attempted to check this point but, except at very

low phosphine concentrations where no appreciable effect on the rate of reaction was observed, the experiments were unsuccessful due to general broadening of the NMR spectra under these conditions.
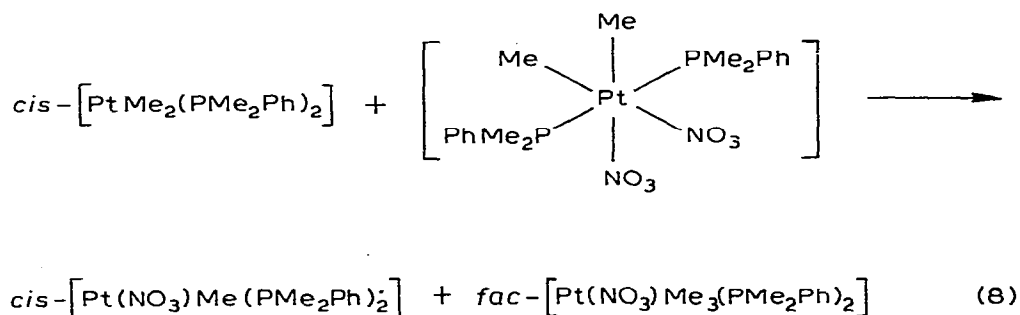
An apparently simpler reaction, which was complete in 24 h, occurred between cis-[PtMe$_2$(PMe$_2$Ph)$_2$] and [Pt(NO$_2$)$_2$Me$_2$(PMe$_2$Ph)$_2$] (eq. 7).

$$cis-\left[PtMe_2(PMe_2Ph)_2\right] \; + \; \left[\begin{array}{c} Me \\ Me \diagdown \; | \diagup PMe_2Ph \\ Pt \\ PhMe_2P \diagup \; | \diagdown NO_2 \\ NO_2 \end{array}\right] \longrightarrow$$

$$trans-\left[Pt(NO_2)Me(PMe_2Ph)_2\right] \; + \; fac-\left[Pt(NO_2)Me_3(PMe_2Ph)_2\right] \qquad (7)$$

An attempt was made to distinguish between the simple exchange mechanism and a redox mechanism by carrying out the reaction with labelled cis-[Pt(CD$_3$)$_2$-(PMe$_2$Ph)$_2$] and analysing the CD$_3$ content of the products by NMR spectroscopy. The following results are predicted: (i) Simple methyl for nitro exchange mechanism should give as products trans-[Pt(NO$_2$)(CD$_3$)(PMe$_2$Ph)$_2$] and [Pt(NO$_2$)-Me$_2$(CD$_3$)(PMe$_2$Ph)$_2$] and (ii) redox mechanism with methyl and nitro group transfer from platinum(IV) to platinum(II) should give trans-[Pt(NO$_2$)Me(PMe$_2$-Ph)$_2$] and [Pt(NO$_2$)Me(CD$_3$)$_2$(PMe$_2$Ph)$_2$].
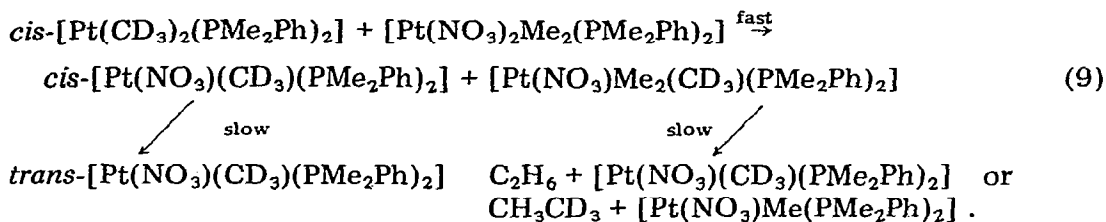
In fact, analysis of the products indicated that complete scrambling of methyl and CD$_3$ groups between platinum(II) and platinum(IV) and amongst all stereochemical positions in the platinum(IV) product had occurred. Thus it seems that either a multiple exchange occurs or else that methyl for methyl exchange occurs more rapidly than methyl for nitro exchange. The results are not useful in distinguishing between the two possible mechanisms of reaction.

It has previously been shown that nitratoplatinum(II) complexes undergo substitution reactions particularly rapidly [2,8,9] and nitratoplatinum(IV) complexes have now been shown to behave similarly. Thus reaction 8 occurred instantly at room temperature.

$$cis-\left[PtMe_2(PMe_2Ph)_2\right] \; + \; \left[\begin{array}{c} Me \\ Me \diagdown \; | \diagup PMe_2Ph \\ Pt \\ PhMe_2P \diagup \; | \diagdown NO_3 \\ NO_3 \end{array}\right] \longrightarrow$$

$$cis-\left[Pt(NO_3)Me(PMe_2Ph)_2\right] \; + \; fac-\left[Pt(NO_3)Me_3(PMe_2Ph)_2\right] \qquad (8)$$

This reaction was complicated by the decomposition of fac-[Pt(NO$_3$)Me$_3$-(PMe$_2$Ph)$_2$] to give ethane and trans-[Pt(NO$_3$)Me(PMe$_2$Ph)$_2$] by reductive elimination. This reaction was complete in ca. 1 h.

The analogous reaction using labelled cis-$[Pt(CD_3)_2(PMe_2Ph)_2]$ occurred as shown in eq. 9.

$$cis\text{-}[Pt(CD_3)_2(PMe_2Ph)_2] + [Pt(NO_3)_2Me_2(PMe_2Ph)_2] \xrightarrow{fast}$$

$$cis\text{-}[Pt(NO_3)(CD_3)(PMe_2Ph)_2] + [Pt(NO_3)Me_2(CD_3)(PMe_2Ph)_2] \qquad (9)$$

<center>slow ↙   slow ↙</center>

$trans\text{-}[Pt(NO_3)(CD_3)(PMe_2Ph)_2]$ $C_2H_6 + [Pt(NO_3)(CD_3)(PMe_2Ph)_2]$ or

                                      $CH_3CD_3 + [Pt(NO_3)Me(PMe_2Ph)_2]$.

The initial platinum(II) product was cis-$[Pt(NO_3)(CD_3)(PMe_2Ph)_2]$ which could only be formed by the simple methyl for nitrate exchange mechanism. The final products after 2 h were $trans$-$[Pt(NO_3)(CD_3)(PMe_2Ph)_2]$ and $trans$-$[Pt(NO_3)Me(PMe_2Ph)_2]$ in the ratio $(2.1 \pm 0.1)/1$. Again this is only consistent with the methyl for nitrate exchange mechanism. Thus assuming that $[Pt(NO_3)Me_2(CD_3)(PMe_2Ph)_2]$ decomposes to give $[Pt(NO_3)Me(PMe_2Ph)_2]$ and $[Pt(NO_3)(CD_3)(PMe_2Ph)_2]$ in a $2/1$ ratio as predicted by statistical considerations only, the final ratio of $trans$-$[Pt(NO_3)Me(PMe_2Ph)_2]$ to $trans$-$[Pt(NO_3)(CD_3)(PMe_2Ph)_2]$ should be $1/2$ in good agreement with experiment. The redox mechanism

$$(10)$$

$$\longrightarrow fac\text{-}[Pt(NO_3)Me_3(PMe_2Ph)_2] + cis\text{-}[Pt(NO_3)Me(PMe_2Ph)_2] \qquad (11)$$
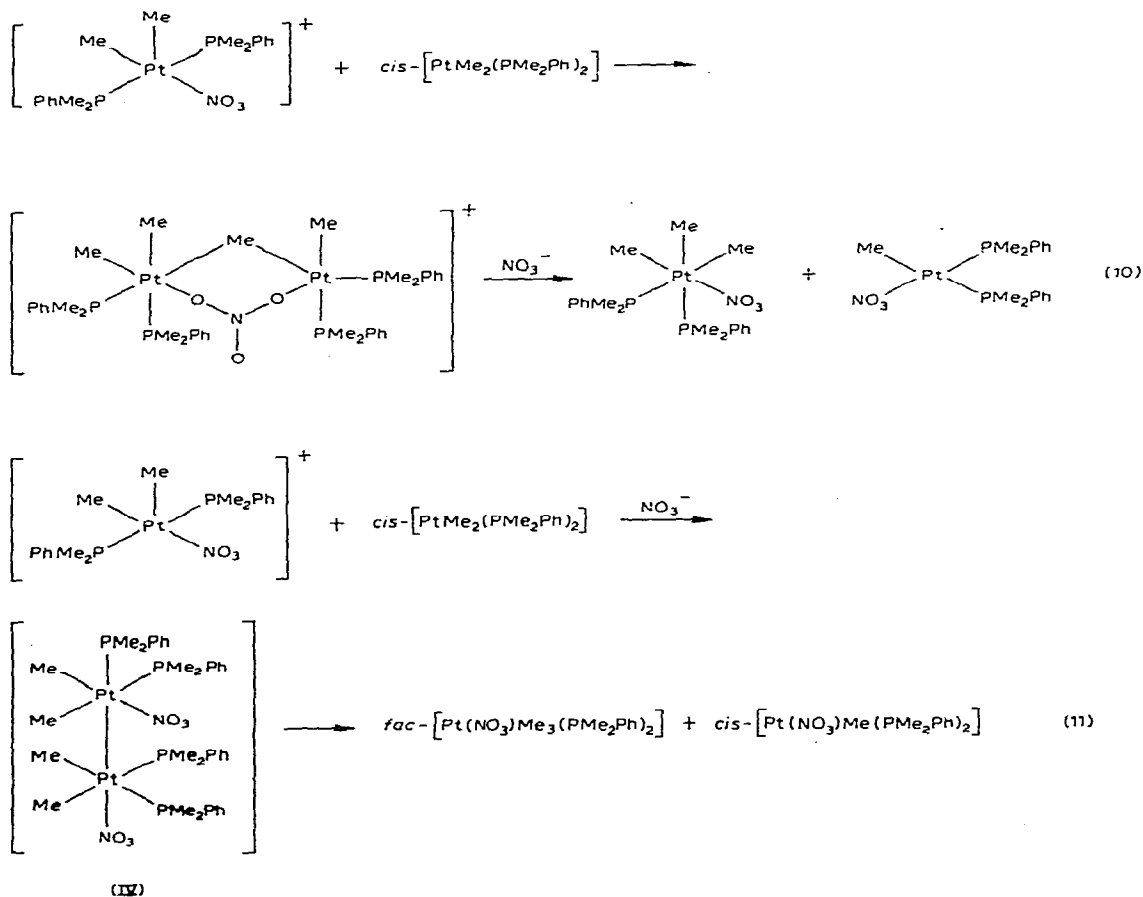
(IV)

TABLE 1

¹H NMR SPECTRA OF METHYLPLATINUM COMPLEXES ᵃ

Complex ᵇ	δ(MePt) (ppm) ᶜ	³J(PH) (Hz)	²J(PtH) (Hz)	δ(MeP) (ppm) ᶜ	²J + ⁴J(PH) (Hz)	³J(PtH) (Hz)
cis-[PtMe₂(PMe₂Ph)₂]	0.39m	1.9ᵈ	67.2	1.44d	7.8	20.1
cis-[PtMe₂(PMe₃)₂]	0.23m	2.0ᵈ	68.0	1.36d	8.0	20.7
t-[PtIMe(PMe₂Ph)₂]	0.26t	7.0	80.6	1.90t	7.1	30.3
t-[Pt(NO₂)Me(PMe₂Ph)₂]	−0.02t	7.5	70.9	1.66t	7.0	32.5
t-[Pt(NO₃)Me(PMe₂Ph)₂]	0.33t	7.2	85.2	1.71t	7.2	28.8
c-[Pt(NO₃)Me(PMe₂Ph)₂]	0.52dd	7.4 / 7.6	47.2	1.53ᵉ / 1.43ᶠ	11 / 8.8	52 / 15
t-c-c-[PtI₂Me₂(PMe₂Ph)₂]	1.64m	1.2ᵈ	57.7	1.82d	9.3	11.7
c-c-t-[PtI₂Me₂(PMe₂Ph)₂]	0.18t	6.0	66	2.43t	7.8	19.2
c-c-t-[Pt(NO₂)₂Me₂(PMe₂Ph)₂]	0.30t	6.0	60.6	1.90t	7.8	17.8
c-c-t-[Pt(NO₃)₂Me₂(PMe₂Ph)₂]	0.79t	5.8	70.6	1.90t	8.1	16.7
fac-[PtIMe₃(PMe₂Ph)₂]	0.60tᵍ / 0.92cʰ	7.6 / 1.7ᵈ	69.7 / 56	1.66d / 1.57d	8.8 / 9.2	10.0 / 11.8
fac-[Pt(NO₂)Me₃(PMe₂Ph)₂]	0.13tⁱ / 0.75cʰ	8.1 / 1.6ᵈ	64.1 / 55.6	1.49d / 1.47d	9.0 / 9.1	18.1 / 18
fac-[Pt(NO₃)Me₃(PMe₂Ph)₂]	0.39tʲ	7.2	69.6	k	h	k

ᵃ In CH₂Cl₂. ᵇ c = cis, t = trans, ᶜ d = doublet, t = triplet, m = multiplet. ᵈ J(PH) + J(P'H), ᵉ trans to NO₃. ᶠ trans to Me. ᵍ Me trans to L. ʰ Me trans to L. ⁱ Me trans to P. ʲ Me trans to NO₂. ʲ Me trans to NO₃. ᵏ Obscured.

would predict a ratio of 2/1 *trans*-[Pt(NO$_3$)Me(PMe$_2$Ph)$_2$]/*trans*-[Pt(NO$_3$)(CD$_3$)-(PMe$_2$Ph)$_2$] by similar arguments.

The precise mechanism by which the methyl for nitrate exchange occurs is not yet known. Since the rate of reaction of the nitratoplatinum(IV) complex was very much greater than with analogous nitro or iodo complexes, it is very likely that dissociation of a nitrate ligand rather than a neutral phosphine ligand from platinum(IV) to create a vacant coordination site occurs [2,8,9]. The methyl transfer from *cis*-[PtMe$_2$(PMe$_2$Ph)$_2$] could then involve a cyclic transition state (III, eq. 10), or an intermediate Pt—Pt bonded species (IV, eq. 11). Note that in either mechanism the change of configuration of the two phosphine ligands from *trans* to *cis* on platinum(IV) is readily explained since the initial five coordinate [Pt(NO$_3$)Me$_2$(PMe$_2$Ph)$_2$]$^+$ is expected to be stereochemically non-rigid. The mechanism of eq. 10 rationalizes the stereochemistry of the initial products most readily [3], though there is evidence that methyl transfer reactions from methyl(tertiary phosphine)—platinum(II) complexes often proceed by the oxidative addition-reduction elimination sequence shown in eq. 11 [4,10]. Note that formation of an intermediate analogous to IV could explain the scrambling of CH$_3$ and CD$_3$ groups in the reaction of *cis*-[Pt(CD$_3$)$_2$(PMe$_2$Ph)$_2$] with [Pt(NO$_2$)$_2$Me$_2$(PMe$_2$Ph)$_2$].

Hydride exchange reactions

Methylplatinum(II) and methylgold(I) complexes undergo exchange reactions with *trans*-[PtIHL$_2$] (L = PMe$_3$ or PMe$_2$Ph), but in each case only one of the two products was stable. For example, reaction between [AuMe(PMe$_3$)] and *trans*-[PtIH(PMe$_3$)$_2$] gave a deep red solution from which *trans*-[PtIMe(PMe$_3$)$_2$] could be isolated. The initial reaction therefore appears to involve a methyl for hydride exchange (eq. 12)

$$\textit{trans}\text{-[PtIH(PMe}_3)_2] + [\text{AuMe(PMe}_3)] \rightarrow \textit{trans}\text{-[PtIMe(PMe}_3)_2] + [\text{AuH(PMe}_3)]$$

$$(12)$$
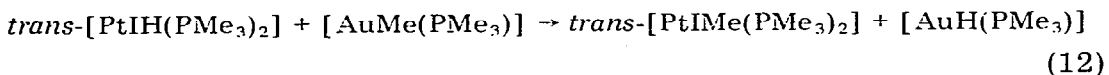
The presumed hydridogold(I) intermediate evidently decomposes perhaps to a gold cluster complex [11], many of which are highly coloured. The red material formed an oil which could not be purified and which decomposed to gold metal on heating.

Similarly, reaction of *cis*-[PtMe$_2$(PMe$_2$Ph)$_2$] with *trans*-[PtIH(PMe$_2$Ph)$_2$] gave, after 2 days in acetone at 60°C, *trans*-[PtIMe(PMe$_2$Ph)$_2$] and CH$_4$ as the only identified products. A red oil was also formed. The reaction probably proceeds according to eq. 13.

$$\textit{cis}\text{-[PtMe}_2(\text{PMe}_2\text{Ph})_2] + \textit{trans}\text{-[PtIH(PMe}_2\text{Ph})_2] \rightarrow$$

$$\textit{trans}\text{-[PtIMe(PMe}_2\text{Ph})_2] + [\text{PtHMe(PMe}_2\text{Ph})_2] \qquad (13)$$
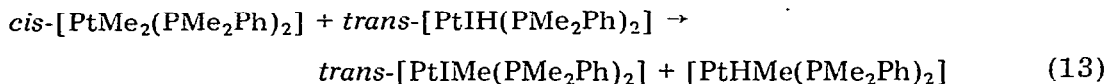
The presumed [PtHMe(PMe$_2$Ph)$_2$] is expected to decompose to methane and [Pt(PMe$_2$Ph)$_2$], which is itself unstable and gives various polymeric products [12]. Hydrido(alkyl)platinum(II) complexes are known to be stable only when the alkyl group carried electronegative substituents [13].

Experimental

cis-[PtMe$_2$(PMe$_2$Ph)$_2$], cis-cis-trans-[PtI$_2$Me$_2$(PMe$_2$Ph)$_2$], cis-cis-trans-[Pt(NO$_2$)$_2$-Me$_2$(PMe$_2$Ph)$_2$], cis-cis-trans-[Pt(NO$_3$)$_2$Me$_2$(PMe$_2$Ph)$_2$] and [AuMe(PMe$_3$)] were prepared by literature methods [5,14,15]. trans-cis-cis-[PtI$_2$Me$_2$(PMe$_2$Ph)$_2$] was prepared by reaction of cis-[PtMe$_2$(PMe$_2$Ph)$_2$] (0.11 g, 0.22 mmol) in ether (5 cm^3) with iodine (0.058 g, 0.23 mmol) in ether (5 cm^3). Bright orange crystals precipitated. The product was filtered off, washed with ether and dried under vacuum. m.p. 154—158°C (decomp.) Yield 0.13 g.

Exchange reactions

The general experimental technique was to mix solutions of the reagents in an NMR tube and to follow the course of the reaction by recording NMR spectra periodically. The solvent was dichloromethane unless otherwise specified. In many cases reaction products were subsequently isolated, while in other cases products were identified by their characteristic ^{1}H NMR spectra by direct comparison with spectra of authentic samples [2]. Only representative experiments are described below.

cis-[PtMe$_2$(PMe$_2$Ph)$_2$] with trans-cis-cis-[PtI$_2$Me$_2$(PMe$_2$Ph)$_2$]. A mixture of cis-[PtMe$_2$(PMe$_2$Ph)$_2$] (0.036 g, 0.072 mmol) and trans-cis-cis-[PtI$_2$Me$_2$-(PMe$_2$Ph)$_2$] (0.055 g, 0.073 mmol) in dichloromethane (0.8 cm^3) was sealed in an NMR tube after first degassing the solution by 3 freeze-pump-thaw cycles.

After 10 h, 50% of the cis-(PtMe$_2$(PMe$_2$Ph)$_2$] has reacted to give fac-[PtIMe$_3$-(PMe$_2$Ph)$_2$] and trans-[PtIMe(PMe$_2$Ph)$_2$] and a small quantity of cis-cis-trans-[PtI$_2$Me$_2$(PMe$_2$Ph)$_2$] was detected. As the reaction proceeded the proportion of the latter isomerised product increased. In a separate experiment it was shown that isomerisation of trans-cis-cis to cis-cis-trans-[PtI$_2$Me$_2$(PMe$_2$Ph)$_2$] in dichloromethane solution was complete in 2 days.

cis-[Pt(CD$_3$)$_2$(PMe$_2$Ph)$_2$] with cis-cis-trans-[Pt(NO$_3$)$_2$Me$_2$(PMe$_2$Ph)$_2$]. cis-[Pt-(CD$_3$)$_2$(PMe$_2$Ph)$_2$] (0.035 g, 0.069 mmol) and cis-cis-trans-[Pt(NO$_3$)$_2$Me$_2$-(PMe$_2$Ph)$_2$] (0.043 g, 0.069 mmol) were mixed in dichloromethane (0.7 cm^3) in an NMR tube. An immediate reaction occurred to give cis-[Pt(NO$_3$)(CD$_3$)-(PMe$_2$Ph)$_2$] and [Pt(NO$_3$)Me$_2$(CD$_3$)(PMe$_2$Ph)$_2$], which decomposed in 2 h to give C$_2$H$_6$ and CH$_3$CD$_3$ (δ 0.80 ppm) and trans-[Pt(NO$_3$)Me(PMe$_2$Ph)$_2$] and trans-[Pt(NO$_3$)(CD$_3$)(PMe$_2$Ph)$_2$]. After 1 day the only products were trans-[Pt(NO$_3$)-(CD$_3$)(PMe$_2$Ph)$_2$] and trans-[Pt(NO$_3$)Me(PMe$_2$Ph)$_2$] in the ratio (2.1 ± 0.1)/1, as deduced by comparing the ratio of methylplatinum/methylphosphorus signal peak heights with that for a pure sample of trans-[Pt(NO$_3$)Me(PMe$_2$Ph)$_2$] [2].

trans-[PtIH(PMe$_3$)$_2$] with [AuMe(PMe$_3$)]. trans-[PtIH(PMe$_3$)$_2$] (0.33 g, 0.70 mmol) was dissolved in toluene (10 cm^3) and cooled to —78°C. To this solution was added [AuMe(PMe$_3$)] (0.2 g, 0.70 mmol) in toluene (5 cm^3). On allowing the mixture to warm to room temperature, the solution became darker until it was an intense red colour. The toluene was evaporated and the residue crystallised from dichloromethane/petroleum ether to give first trans-[PtIMe(PMe$_3$)$_2$] (0.25 g), m.p. 157—158°C (lit. [16], 158—160°C), and then an oily red precipitate which could not be induced to crystallise. An attempt was made to purify the product by vacuum sublimation, but decomposition to gold occurred and only a trace of trans-[PtI$_2$(PMe$_3$)$_2$] sublimed.

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