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Oxidative addition of triphenylstannyl aryltellurols to platinum(0) and palladium(0) complexes

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Abstract

Triphenylstannyl aryltellurols, $Ph_3SnTeAr$ (Ar = Ph, C_6H_4Me-4, C_6H_4OMe-4 and C_6H_4OEt-4), react with $Pt(PPh_3)_4$ or $Pt(PPh_3)_2(C_2H_4)$ to form *cis*-$[Pt(PPh_3)_2(TeAr)(SnPh_3)]$ (**Ia**) which with more $Ph_3SnTeAr$ gives *cis*-$[Pt(PPh_3)_2(Ph)(SnPh_2TeAr)]$ (**IIa**). The formation of **IIa** from **Ia** is explained by a mechanism involving a six coordinate platinum(IV) intermediate. A similar reaction between $Pd(PPh_3)_4$ and $Ph_3SnTeAr$ resulted in the formation of two *cis* palladium(II) complexes (**Ib** and **IIb**) in solution. Complexes **Ia** and **IIa** (Ar = C_6H_4OEt-4) have been isolated and characterized and ^1H and $^{31}P\{^1H\}$ NMR parameters are reported for all complexes.

Introduction

The diorganyl dichalcogenides, R_2E_2 (E = S, Se, Te), are known to undergo oxidative addition reactions with Pd(0) and Pt(0) by cleavage of the E–E bond [1–4]. Although Ph_2Te_2 has been found to be unreactive towards $[Pt(PPh_3)_2(C_2H_4)]$ [5], related oxidative addition reactions of $[Pt(PPh_3)_4]$ with $Te_2(C_6H_4Me\text{-}4)_2$ and *p*-$MeOC_6H_4TeCN$ [6] as well as of $[Pd(PPh_3)_4]$ with Ar_2Te_2 (Ar = *p*-$EtOC_6H_4$ or 2-C_4H_3S) [3] have been reported. Recently Jennings *et al.* [7, 8] have studied the oxidative addition of thiols and selenols to a coordinatively unsaturated triplatinum cluster. Platinum(0) complexes, $[Pt(PPh_3)_4]$ and $[Pt(PPh_3)_2(C_2H_4)]$, are known to react with organotin reagents such as $SnPh_4$, $SnPh_2Cl_2$, $SnMe_3Cl$ and $SnMe_3Ar$ to give complexes which are the products of insertion of platinum into the Sn–C bond [9–12]. The reactions of $Ph_3SnTeAr$ with the platinum(0) or palladium(0) complexes have not yet been reported. Hence, in order to investigate the behaviour of $Ph_3SnTeAr$ and to know whether M(Pt, Pd) inserts into the Sn–C or Sn–Te bond or both when treated with $Ph_3SnTeAr$, the present study is undertaken.

Experimental

All the reactions were carried out using the Schlenk tube technique under dry oxygen free nitrogen atmosphere. The ^1H and $^{31}P\{^1H\}$ NMR spectra were recorded on a Bruker AM 500 MHz FT NMR instrument in C_6D_6 using TMS as internal and H_3PO_4 as external references. The ^{31}P resonances downfield with respect to H_3PO_4 are given as positive values (ppm).

^1H NMR study in solution

A typical procedure for the ^1H NMR study is as follows. $Pt(PPh_3)_4$ or $Pt(PPh_3)_2(C_2H_4)$ (0.025 g) was dissolved in 1 cm^3 of C_6D_6 in a Schlenk tube under oxygen free dry nitrogen atmosphere. To this solution was added solid $Ph_3SnTeAr$ (Ar = C_6H_4Me-4, C_6H_4OMe-4 and C_6H_4OEt-4) in 1:0.5 (or 1:2) molar ratio and the spectrum of the resulting solution was recorded after 2–4 h at room temperature.

Reactions between $Pd(PPh_3)_4$ and $Ph_3SnTeAr$ were carried out following a similar procedure and the spectra of the resulting solutions were recorded.

^{31}P NMR study in solution

$Pt(PPh_3)_4$ or $Pt(PPh_3)_2(C_2H_4)$ (0.05 g) was dissolved in C_6D_6 (3 cm^3) and then treated with $Ph_3SnTeAr$ (Ar = Ph, C_6H_4Me-4, C_6H_4OMe-4 and C_6H_4OEt-4) in 1:0.5 (or 1:1 or 1:2) molar ratio under oxygen free dry nitrogen atmosphere. The $^{31}P\{^1H\}$ NMR spectrum of the resulting solution was recorded after 2–4 h of mixing the reagents.

A similar methodology was adopted for recording the $^{31}P\{^1H\}$ NMR spectrum of the reaction products in solution resulting from $Pd(PPh_3)_4$ (0.1 g) and

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$Ph_3SnTeAr$ in 1:2 molar ratio after 4 h of mixing the reagents.

*Synthesis of [Pt(PPh$_3$)$_2$(TeC$_6$H$_4$OEt-4)(SnPh$_3$)] (**Ia**)*

The complex was prepared by treatment of $Pt(PPh_3)_2(C_2H_4)$ (0.2 g, 0.26 mmol) in benzene or dichloromethane with $Ph_3SnTeC_6H_4OEt$-4 in 1:0.5 molar ratio. After *c.* 2 h at room temperature, the reaction mixture was concentrated under reduced pressure and mixed with a large volume of light petroleum and diethyl ether (1:1, vol./vol.) when a light yellow solid separated out. The product after washing with the same solvent system, was recrystallized from CH_2Cl_2/hexane. Yield, 53%. Decomposition temperature 125 °C. *Anal.* Calc. for $C_{62}H_{54}OP_2TeSnPt$: C, 56.47; H, 4.09; Te, 9.68%. Found: C, 55.80; H, 3.95; Te, 10.34%. Molecular weight (C_6H_6): found 1280, calc. 1317. λ_M (10^{-3} M, CH_3CN) 16 ohm^{-1} cm^2 mol^{-1}. $^{31}P\{^1H\}$ NMR: δ 23.9 ppm, ^1J(Pt–P) 2348 Hz, ^2J(PP) 14 Hz; δ 19.4 ppm, ^1J(Pt–P) 2106 Hz.

*Synthesis of [Pt(PPh$_3$)$_2$(Ph)(SnPh$_2$TeC$_6$H$_4$OEt-4)] (**IIa**)*

It was obtained as a light orange solid on addition of an excess of $Ph_3SnTeC_6H_4OEt$-4 to $Pt(PPh_3)_2(C_2H_4)$ in the same manner as mentioned above. Yield, 59%. Decomposition temperature, 114 °C. *Anal.* Calc. for $C_{62}H_{54}OP_2TeSnPt$: C, 56.47; H, 4.09; Te, 9.68%. Found: C, 55.01; H, 3.91; Te, 10.46%. Molecular weight (C_6H_6): found 1268, calc. 1317. λ_M (10^{-3} M, CH_3CN) 12 ohm^{-1} cm^2 mol^{-1}. $^{31}P\{^1H\}$ NMR: δ 24.8 ppm, ^1J(Pt–P) 1552 Hz, ^2J(PP) 18 Hz; δ 18.1 ppm, ^1J(Pt–P) obscured.

Results and discussion

The formation of the reaction products of the types $[M(PPh_3)_2(SnPh_3)(TeAr)]$ (**Ia** and **Ib**) and $[M(PPh_3)_2(Ph)(SnPh_2TeAr)]$ (**IIa** and **IIb**) has been examined *in situ* by ^1H and ^{31}P NMR from the reactions between $M(PPh_3)_4$ (M = Pt, Pd) or $Pt(PPh_3)_2(C_2H_4)$ with $Ph_3SnTeAr$ in C_6D_6 at room temperature. The ^1H and ^{31}P NMR parameters are compiled in Tables 1–3. Complexes **Ia** and **IIa** (M = Pt, Ar = C_6H_4OEt-4) have been isolated and characterized.

^1H NMR study

Reactions of $Pt(PPh_3)_4$, $Pt(PPh_3)_2(C_2H_4)$ and $Pd(PPh_3)_4$ with $Ph_3SnTeAr$ (Ar = C_6H_4Me-4, C_6H_4OMe-4 and C_6H_4OEt-4) in 1:0.5 and 1:2 molar ratios have been examined by ^1H NMR in C_6D_6 solution. In each case, three methyl, methoxy or ethoxy signals are observed, one of which is identified for the parent tellurol. The other two signals can be considered to originate from the reaction products. The intensity of these two signals varies with the concentration of the tin reagent suggesting that the signals are arising out of the two different complexes. The signals in the aromatic region could not give much useful information as all appeared in the same region. Also the detection of the free ethylene signal in the reaction between $Pt(PPh_3)_2(C_2H_4)$ and $Ph_3SnTeAr$ has proved to be elusive in the ^1H NMR spectrum. All the ^1H NMR parameters of methyl, methoxy and ethoxy resonances have been tabulated in Table 1.

^{31}P NMR study

In a typical experiment $Pt(PPh_3)_4$ or $Pt(PPh_3)_2(C_2H_4)$ and $Ph_3SnTeAr$ in 2:1 molar proportion are dissolved in C_6D_6 and the clear solution is left to stand at room temperature for 2 h before recording its $^{31}P\{^1H\}$ NMR spectrum. The spectrum of the resulting complex **Ia** comprises of two doublets each with ^{195}Pt satellites (Fig. 1). The coupling constant ^2J(P–Pt–P) values (12–14 Hz; Table 2) are typical for a *cis* structure [13]. In general, the *cis* complexes have lower ^2J(PP) coupling constants than those of *trans* complexes (^2J(PP) for *cis* complexes is <40 Hz and for *trans* complexes it is >150 Hz [14]). Values of ^1J(Pt–P) are known to depend largely on the nature of the phosphine and the ligand *trans* to it in platinum(II) complexes [15, 16]. The two ^1J(Pt–P) coupling constants observed are in the range 2342–2352 and 2102–2118 Hz (Table 2); the former arises due to coupling of Pt with PPh_3 *trans* to $SnPh_3$ (since the values are of the order observed for *cis*-$[PtPh(SnPh_3)(PPh_3)_2]$ (2352 Hz) [12]) and the latter, although relatively smaller than the reported values for *cis*-$[Pt(Te_2C_6H_4)(PPh_3)_2]$ (2990 Hz) and *cis*-$[Pt(Te_2C_5H_6)(PPh_3)_2]$ (2860 Hz) [17], corresponds to coupling of Pt with PPh_3 *trans* to the TeAr group. Thus, the ^{31}P NMR strongly suggests that the complexes **Ia** have *cis* structure. Since the Pt(0) complexes are highly sensitive towards oxidative addition reactions and the Sn–Te bond in $Ph_3SnTeAr$ is also very reactive, [18, 19], formation of **Ia** from $Ph_3SnTeAr$ and $Pt(PPh_3)_4$ or $Pt(PPh_3)_2(C_2H_4)$ may be represented by eqn. (1)

$$\begin{array}{l} Pt(PPh_3)_4 \\ \text{or} \\ Pt(PPh_3)_2(C_2H_4) \end{array} \xrightarrow[-2PPh_3 \text{ or } -C_2H_4]{Ph_3SnTeAr/C_6D_6} \underset{\textbf{Ia}}{cis\text{-}[Pt(PPh_3)_2(TeAr)(SnPh_3)]} \qquad (1)$$

The free PPh_3 liberated in the reaction between $Pt(PPh_3)_4$ and $Ph_3SnTeAr$ is observed at $-\delta$ 9.3 ppm (upfield from the reference H_3PO_4).

TABLE 1. ^1H NMR parameters of reaction products of mixing $Pt(PPh_3)_4$ (or $Pt(PPh_3)_2(C_2H_4)$) (A) or $Pd(PPh_3)_4$ (B′) with $Ph_3SnTeAr$ (L) in C_6D_6

Mixture	δCH_2 (ppm)	δCH_3 (ppm)
A, L(Ar = $C_6H_4CH_3$-4)[a]		2.34(s)
		2.26(s)
A, L(Ar = $C_6H_4OCH_3$-4)[b]		3.55(s)
		3.47(s)
A, L(Ar = $C_6H_4OCH_2CH_3$-4)[c]	3.83(q, J = 7 Hz)	1.40(t, J = 7 Hz)
	3.77(q, J = 7 Hz)	1.34(t, J = 7 Hz)
B′, L(Ar = $C_6H_4CH_3$-4)		2.38(s)
		2.26(s)
B′, L(Ar = $C_6H_4OCH_3$-4)		3.56(s)
		3.46(s)
B′, L(Ar = $C_6H_4OCH_2CH_3$-4)	3.87(q, J = 7 Hz)	1.37(t, J = 7 Hz)
	3.74(q, J = 7 Hz)	1.32(t, J = 7 Hz)

For free L[a], δCH_3 2.16 ppm(s); L[b], δOCH_3, 3.37 ppm(s); L[c], δCH_2 3.63 ppm (q, J = 7Hz), δCH_3 1.28 ppm (t, J = 7 Hz); s = singlet, t = triplet, and q = quartet.

TABLE 2. ^{31}P NMR data of reaction products of mixing $Pt(PPh_3)_2(C_2H_4)$ or $Pt(PPh_3)_4$ with $Ph_3SnTeAr$ in C_6D_6

Ar	δP (ppm)	^1J(Pt–P) (Hz)	^2J(PP) (Hz)
cis-[$Pt(PPh_3)_2(SnPh_3)(TeAr)$] (**Ia**)			
Ph	23.81(d)	2342(*trans* $SnPh_3$)	14
	19.37(d)	2102(*trans* TePh)	
C_6H_4Me-4	23.88(d)	2352(*trans* $SnPh_3$)	14
	19.34(d)	2118(*trans* TeAr)	
C_6H_4OMe-4	23.89(d)	2344(*trans* $SnPh_3$)	14
	19.49(d)	2106(*trans* TeAr)	
C_6H_4OEt-4	28.40(d)	2348(*trans* $SnPh_3$)	12
	24.00(d)	2108(*trans* TeAr)	
cis-[$Pt(PPh_3)_2(Ph)(SnPh_2TeAr)$] (**IIa**)			
Ph	20.8(d)	1590(*trans* Ph)	15
	16.1(d)	2350(*trans* Sn)	
C_6H_4Me-4	21.0(d)	1544(*trans* Ph)	18
	15.9(d)	2370(*trans* Sn)	
C_6H_4OMe-4	22.7(d)	1550(*trans* Ph)	15
	18.3(d)	2414(*trans* Sn)	
C_6H_4OEt-4	25.2(d)	1540(*trans* Ph)	18
	20.5(d)	obscured	

TABLE 3. ^{31}P NMR data of reaction products of mixing $Pd(PPh_3)_4$ with $Ph_3SnTeAr$ in C_6D_6

Ar	δP (ppm)	^2J(PP) (Hz)
cis-[$Pd(PPh_3)_2(SnPh_3)(TeAr)$] (**Ib**)		
Ph	9.84(d)	25
	9.54(d)	
C_6H_4Me-4	9.69(d)	22
	9.41(d)	
C_6H_4OMe-4	9.58(d)	22
	9.47(d)	
C_6H_4OEt-4	9.81(d)	24
	9.50(d)	
cis-[$Pd(PPh_3)_2(Ph)(SnPh_2TeAr)$] (**IIb**)		
Ph	7.08(d)	17
	6.78(d)	
C_6H_4Me-4	7.15(d)	16
	6.87(d)	
C_6H_4OMe-4	7.17(d)	15
	6.81(d)	
C_6H_4OEt-4	7.12(d)	17
	6.82(d)	

In view of substantive dissociation of $Pt(PPh_3)_4$ in solution to give $Pt(PPh_3)_3$ and even $Pt(PPh_3)_2$ [20, 21], a five coordinate or a tetrahedral intermediate of Pt species with $Ph_3SnTeAr$ can be proposed to be the most plausible one for the formation of *cis* complex **Ia** (Scheme 1). The intermediate is, of course, not present in sufficient quantity at any time to be observed in the $^{31}P\{^1H\}$ NMR spectrum.

In another experiment, when the platinum(0) complex in C_6D_6 is treated with $Ph_3SnTeAr$ in 1:2 molar ratio, the $^{31}P\{^1H\}$ NMR spectrum of the resulting solution after 4 h does not show the presence of

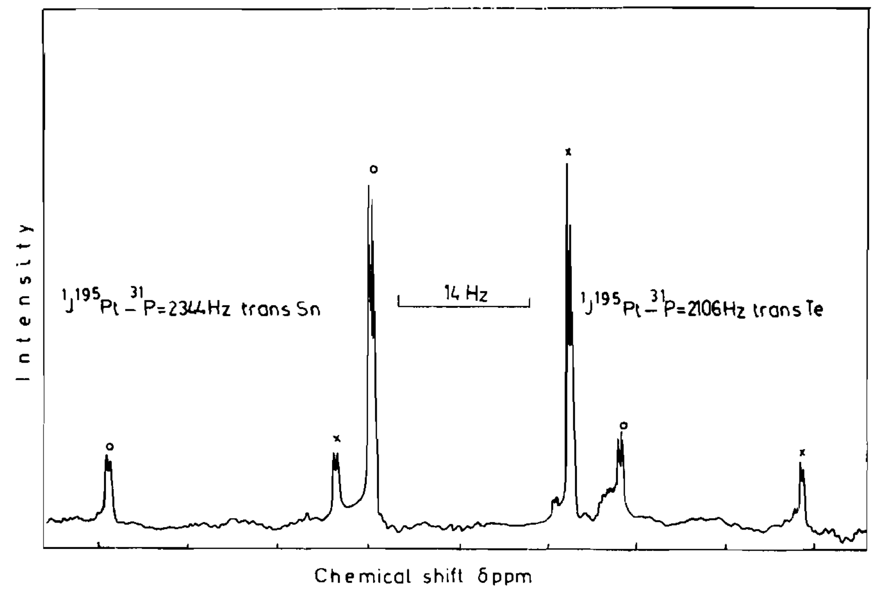
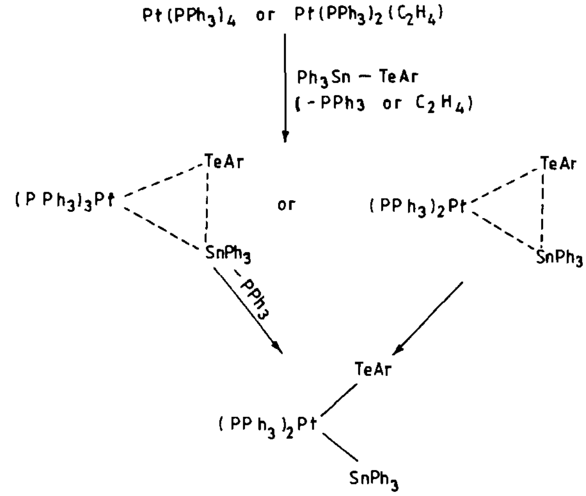


Fig. 1. ^{31}P NMR spectrum of *cis*-$[Pt(PPh_3)_2(SnPh)_3(TeC_6H_4OMe\text{-}4)]$ in C_6D_6 at 202.4 MHz.



Ia

Scheme 1. Suggested mechanism for the formation of **Ia** from Pt(0) and $Ph_3SnTeAr$.

any detectable amount of **Ia**. However, it does indicate the existence of a new complex **IIa**. The spectrum consists of two doublets of equal intensities both having platinum satellites (Fig. 2). Using arguments similar to those given for **Ia**, it follows that **IIa** is also a *cis* complex with two non-equivalent phosphorus atoms attached to platinum (^2J(PP) 15–18 Hz, Table 2). The values of 1540–1590 and 2350–2414 Hz for ^1J(Pt–P) (Table 2) are of the order expected for PPh_3 *trans* to Ph [12, 22] and $SnPh_2TeAr$ [12] groups, respectively.

The $^{31}P\{^1H\}$ NMR spectrum of the C_6D_6 solution containing the tin reagent and the platinum(0) complex in 1:1 molar ratio shows the coexistence of both the complexes **Ia** and **IIa** even after 4 h although **IIa** is the major product.

The $^{31}P\{^1H\}$ NMR experiment clearly indicates that, of the two products in the reaction of Pt(0) and $Ph_3SnTeAr$, complex **Ia** is initially formed which then reacts with more tin reagent to give another complex **IIa**. The formation of **IIa** from **Ia** and $Ph_3SnTeAr$ may be satisfactorily explained by Scheme 2 (a mechanism similar to that for the formation of *cis*-$[PtMe(SnMe_2Cl)(PPh_3)_2]$ from $[Pt(PPh_3)_2(C_2H_4)]$ and $SnMe_3Cl$ [23]) in which it is suggested that a second molecule of $Ph_3SnTeAr$ adds oxidatively to complex **Ia** to give a six coordinate platinum(IV) complex. One molecule of $Ph_3SnTeAr$ is then reductively eliminated to give the new complex **IIa**. Since no $SnPh_2(TeAr)_2$ is detected during the reaction by ^1H NMR, it is considered that the elimination of $Ph_3SnTeAr$ is probably faster than the sterically hindered elimination of $SnPh_2(TeAr)_2$. It is important to note that the proposed mechanism does not require the insertion of **Ia** into the Sn–Ph bond to be faster than its insertion into the Sn–TeAr bond. Indeed the reverse is likely to be true. But insertion of **Ia** into Sn–TeAr is followed by elimination of $Ph_3SnTeAr$ to reform **Ia**, whereas insertion of **Ia** into the Sn–Ph bond, although slower, can result in the formation of the **IIa** by elimination of $Ph_3SnTeAr$.

The complexes **Ia** and **IIa** (Ar = C_6H_4OEt-4) have been isolated as light yellow and light orange solids, respectively (see 'Experimental'). The elemental analyses and molecular weight measurements in benzene are consistent with the above formulations. They are found to be non-electrolytes in CH_3CN solution. The

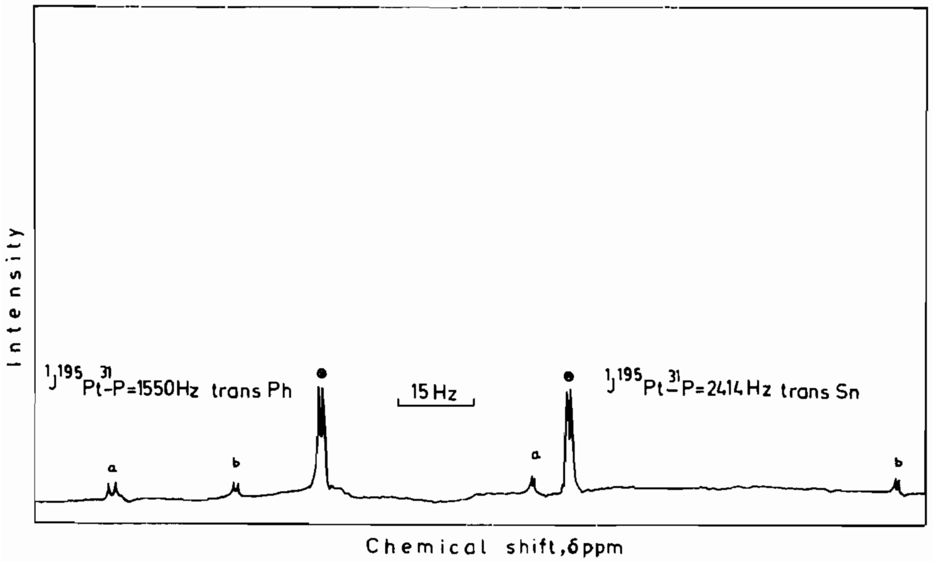
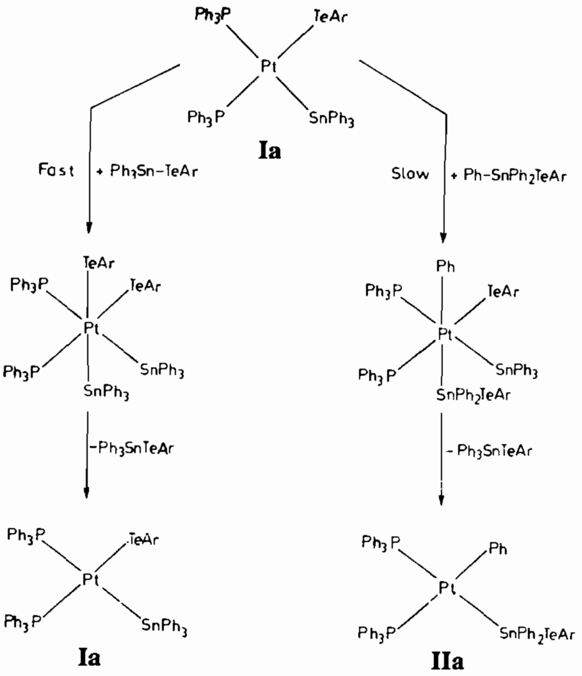


Fig. 2. ^{31}P NMR spectrum of *cis*-[Pt(PPh$_3$)$_2$(Ph)(SnPh$_2$TeC$_6$H$_4$OMe-4)] in C_6D_6 at 202.4 MHz.



Scheme 2. Proposed mechanism for the formation of **IIa** from **Ia** and Ph$_3$SnTeAr.

^1H NMR spectra show the equivalence of all aryl groups in the complexes. The $^{31}P\{^1H\}$ NMR spectra suggest their *cis* configurations in solution. The ^{31}P NMR parameters of the reaction products obtained *in situ* are in good agreement with the authentic samples. Although the ν(Pt–C) band in the Pt–aryl complexes have been reported to lie in the range 480–600 cm^{-1}, an unambiguous identification in the IR spectrum of **IIa** is difficult due to its association with aromatic ring vibrations.

The reaction between palladium(0) complexes and Ph$_3$SnTeAr (Ar = Ph, C$_6$H$_4$Me-4, C$_6$H$_4$OMe-4 and C$_6$H$_4$OEt-4) has also been examined *in situ* by ^{31}P NMR spectroscopy. The spectrum recorded with the resulting solution after 4 h consists of two doublets of equal intensities. This clearly indicates the non-equivalence of the two phosphorus atoms in the complex. The coupling constant ^2J(P–Pd–P) (15–26 Hz; Table 3) is characteristic for a *cis* complex. The free PPh$_3$ liberated in the reaction is observed at $-\delta$ 9.0–9.3 ppm. This strongly suggests that, similar to the platinum analogues, two *cis* complexes [Pd(PPh$_3$)$_2$(TeAr)(SnPh$_3$)] (**Ib**) and [Pd(PPh$_3$)$_2$(Ph)(SnPh$_2$TeAr)] (**IIb**) are also formed in the reaction between Pd(PPh$_3$)$_4$ and Ph$_3$SnTeAr.

It has been observed that the reaction of Ph$_3$SnTeAr with Pd(0) is relatively slower than its reaction with Pt(0). The complexes **Ib** and **IIb** are formed simultaneously from mixing Pd(PPh$_3$)$_4$ with Ph$_3$SnTeAr in 1:2 molar ratio in C_6D_6 solution. The isolation and separation of **Ib** and **IIb** have been unsuccessful.

Conclusions

The reaction of Ph$_3$SnTeAr with Pd(0) and Pt(0) gives rise to two *cis* complexes, [M(PPh$_3$)$_2$(TeAr)(SnPh$_3$)] and [M(PPh$_3$)$_2$(Ph)(SnPh$_2$TeAr)], which are products of insertion of M(Pd, Pt) into the Sn–Te and Sn–C bonds, respectively.

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