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Preparative and Spectral Investigation of Palladium(II) Complexes with Aryl Tellurols and Diaryl Ditellurides

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Abstract

Tellurol complexes of types [Pd(COD)Cl(TeAr)] (I) and [Pd(COD)(TeAr)$_2$] (II) (COD = 1,5-cyclooctadiene; Ar = phenyl, *p*-tolyl, *p*-anisyl and *p*-phenetyl) are readily obtained by the reactions between Pd(COD)Cl$_2$ and NaTeAr in 1:1 and 1:2 molar ratios respectively in ethanol at room temperature. Similar complexes also resulted in reactions between Pd(COD)Cl$_2$ and Ph$_3$SnTeAr in chloroform. The diolefin ligand is easily displaced by PPh$_3$ in chloroform to give the corresponding phosphine complexes [Pd(PPh$_3$)$_2$Cl(TeAr)] (III) and [Pd(PPh$_3$)$_2$(TeAr)$_2$] (IV). ^{31}P NMR spectra of III and IV indicate their *cis* configuration in solution. Solid Te$_2$Ar$_2$ on treatment with [Pd$_2$Cl$_2$(μ-Cl)$_2$(PPh$_3$)$_2$] in dichloromethane yielded [Pd$_2$Cl$_4$(μ-Te$_2$Ar$_2$)(PPh$_3$)$_2$] (V) in which the bridging chloride ligands are replaced by one molecule of ditelluride. The complexes were characterized by elemental analysis, IR, Raman, ^{1}H and ^{31}P NMR spectra and where possible, by conductivity measurements and molecular weight determinations.

Introduction

A few tellurol complexes such as [Cu(TePh)]$_n$ [1], Ph$_3$MTeAr (M = Ge, Sn and Pb) [2] and π-CpNiPBu$_3$-TePh [3] have been prepared using sodium tellurol reagent. We have recently reported the formation of complexes [M(PPh$_3$)$_2$Cl(TeAr)], [M(PPh$_3$)$_2$(TeAr)$_2$], [MCl(TeAr)]$_n$ and [M(TeAr)$_2$]$_n$ (where M = Pt(II)/Pd(II)) using the same reagent [4, 5]. In this paper we report the preparation of complexes [Pd(COD)-Cl(TeAr)] (I) and [Pd(COD)(TeAr)$_2$] (II) using NaTeAr as well as Ph$_3$SnTeAr. Also, in continuation of our earlier work on the synthesis of palladium(II)–ditelluride complexes [5], where two molecules of PdCl$_2$ are bridged by two molecules of diaryl ditellurides, interestingly we now report the synthesis of binuclear complexes, where two PdCl$_2$(PPh$_3$) moieties are bridged by only one molecule of diaryl ditelluride.

Experimental

All operations were carried out under purified argon. Solvents were dried and distilled before use.

Melting points were recorded in open capillary tubes and are uncorrected. IR spectra were recorded in the solid state as CsI pellets on a Nicolet 5DX FT instrument and far IR (450–50 cm^{-1}) on a Polytec FIR model 30 as polyethene pellets. Raman spectra were recorded on a Cary model 82 Raman spectrometer using krypton ion laser at 6471 Å with 120 MW power. ^{1}H NMR spectra were recorded in CDCl$_3$ or DMSO-d$_6$ solutions on a Jeol FX 100 FT instrument with TMS as an internal standard. ^{31}P{^{1}H} NMR spectra were done in CD$_2$Cl$_2$ or CDCl$_3$ solutions on a Brüker AM-500 MHz FT instrument with H$_3$PO$_4$ as an external reference. Elemental analyses were carried out on a Perkin-Elmer 240 C rapid analyser. Conductance was measured in acetonitrile using a high sensitive Pye conductance bridge (Model No. 11700) and molecular weights were determined in chloroform using a Knauer vapour pressure osmometer (Model No. 11.00).

Pd(COD)Cl$_2$ [6], Pd$_2$Cl$_4$(PPh$_3$)$_2$ [7], Ph$_3$SnTeAr [2] and Te$_2$Ar$_2$ [8–10] were prepared by published methods and their purities were verified by elemental analyses and melting points.

Preparation of [Pd(COD)Cl(TeAr)] (I) (Ar = Phenyl, p-tolyl, p-anisyl and p-phenetyl)

Method a

The appropriate Te$_2$Ar$_2$ (0.5 mmol) was dissolved in the minimum amount of ethanol at room temperature and an alcoholic solution of NaBH$_4$ was added dropwise to it under stirring. The red colour of the ditelluride quickly faded due to the formation of NaTeAr. Sodium borohydride addition was stopped when the reduction was complete as indicated by the

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pale yellow (completely free from any tinge of red) colour of the solution. $Pd(COD)Cl_2$ (1 mmol) in ethanol was then added to the tellurolate solution all at once and the mixture stirred for 6 h. A dark brown solid formed during this period was separated by filtration, washed successively with water, alcohol and ether and dried in air.

Method b

Solid $Ph_3SnTeAr$ (Ar = *p*-phenetyl) (1 mmol) was added to a solution of $Pd(COD)Cl_2$ (1 mmol) in chloroform. Immediately after addition, a brown suspension appeared which was kept stirring for 6 h. The solution was centrifuged when a brown solid separated out. It was washed with chloroform and ether and dried in air.

Preparation of [Pd(COD)(TeAr)$_2$] (II) (Ar = phenyl, p-tolyl, p-anisyl and p-phenetyl)

These were prepared in the same way as [Pd(COD)Cl(TeAr)] (**I**) except that $Pd(COD)Cl_2$ and NaTeAr were taken in a 1:2 molar ratio. The same product **II** was obtained on mixing $Ph_3SnTeAr$ (Ar = *p*-phenetyl) and $Pd(COD)Cl_2$ in chloroform in a 2:1 molar ratio.

Reaction between [Pd(COD)Cl(TeAr)] (I) and PPh$_3$ (Ar = phenyl, p-tolyl, p-anisyl and p-phenetyl)

To a suspension of [Pd(COD)Cl(TeAr)] (1 mmol) in chloroform was added PPh_3 (2 mmol) and the solution was stirred overnight. The clear solution thus obtained was concentrated under low pressure and poured into a large volume of petroleum ether–ether (1:1 vol./vol.) mixture when an orange solid was separated out. It was washed with the same mixture and dried *in vacuo*.

Reaction between [Pd(COD)(TeAr)$_2$] (II) and PPh$_3$ (Ar = phenyl, p-tolyl, p-anisyl and p-phenetyl)

A 250 mg sample of $[Pd(COD)(TeAr)_2]$ was treated with an excess of PPh_3 in chloroform and the mixture was kept on stirring overnight. The clear solution thus obtained was concentrated and treated with n-pentane when a brown solid separated out. It was washed with n-pentane and diethyl ether and dried *in vacuo*.

Preparation of [Pd$_2$Cl$_4$(μ-Te$_2$Ar$_2$)(PPh$_3$)$_2$] (V) (Ar = phenyl, p-tolyl, p-anisyl and p-phenetyl)

Solid Te_2Ar_2 (1 mmol) was added to the suspended solution of $Pd_2Cl_4(PPh_3)_2$ (1 mmol) in dichloromethane and the solution was stirred for 6 h. The addition of n-hexane to the clear solution thus obtained afforded a brown solid which was washed with n-hexane and diethyl ether and dried *in vacuo*.

Reaction between [Pd$_2$Cl$_4$(μ-Te$_2$Ar$_2$)(PPh$_3$)$_2$] (V) and PPh$_3$

An excess of PPh_3 in diethyl ether (50 ml) was added to $[Pd_2Cl_4(\mu\text{-}Te_2Ar_2)(PPh_3)_2]$ (Ar = *p*-tolyl or *p*-anisyl) (0.1 g) and the suspension stirred for 1 h at room temperature. The ether solution turned red by this time. It was filtered and the clear filtrate thus obtained was evaporated to dryness to give a red solid. ^1H NMR of this in $CDCl_3$ showed it to be a mixture of Te_2Ar_2 and PPh_3. Ditelluride was also detected *in situ* from a reaction mixture of the two reactants in $CDCl_3$ by ^1H NMR.

Results and Discussion

Reactions between $[Pd(COD)Cl_2]$ and NaTeAr in ethanol led to the formation of complexes **I** and **II** according to the equations given below

$$[Pd(COD)Cl_2] + NaTeAr$$
$$= \underset{\textbf{I}}{[Pd(COD)Cl(TeAr)]} + NaCl$$

$$[Pd(COD)Cl_2] + 2NaTeAr$$
$$= \underset{\textbf{II}}{[Pd(COD)(TeAr)_2]} + 2NaCl$$

(Ar = phenyl, *p*-tolyl, *p*-anisyl and *p*-phenetyl)

Characterization data of the complexes along with their yields are given in Table 1. Elemental analyses for all the complexes were satisfactory. Infrared spectra (in the solid state as CsI pellets) (Table 2) showed a single ν(Pd–Cl) band for **I** and none for **II**. A strong ν(C=C) band for the COD ligand (1469–1488 cm^{-1}) and another for the aromatic group (1560–1587 cm^{-1}) were present in all the cases. The complexes have very low solubility in most of the common organic solvents and hence their ^1H NMR could not be recorded. Since the COD ligand always occupies two adjacent positions in a metal complex, the complexes **I** and **II** have necessarily a *cis* structure (Fig. 1).

It is interesting to note that the reactions between $Pd(COD)Cl_2$ and $Ph_3SnTeAr$ (Ar = *p*-phenetyl) in chloroform at room temperature also afforded the complexes **I** and **II** according to the equations given below

$$[Pd(COD)Cl_2] + Ph_3Sn{-}TeAr$$
$$= \underset{\textbf{I}}{[Pd(COD)Cl(TeAr)]} + Ph_3SnCl$$

TABLE 1. Physical and analytical data

Complex	Yield (%)	Colour	Decomposition temperature (°C)	Analysis[a] (%)				M[b]
				C	H	Cl	Te	
$[Pd(COD)Cl(TePh)]$	75	dark brown	140	36.8 (36.9)	3.7 (3.7)	7.5 (7.8)	28.4 (28.0)	
$[Pd(COD)Cl(TeC_6H_4Me\text{-}4)]$	50	dark brown	134	38.5 (38.4)	4.0 (4.0)	7.2 (7.5)	27.5 (27.2)	
$[Pd(COD)Cl(TeC_6H_4OMe\text{-}4)]$	80	dark brown	143	37.3 (37.1)	3.8 (3.9)	7.5 (7.3)	26.6 (26.3)	
$[Pd(COD)Cl(TeC_6H_4OEt\text{-}4)]$	67	dark brown	137	38.4 (38.5)	4.2 (4.2)	7.4 (7.1)	25.7 (25.5)	
$[Pd(COD)(TePh)_2]$	54	dark brown	130	38.3 (38.4)	3.4 (3.5)		41.2 (40.9)	
$[Pd(COD)(TeC_6H_4Me\text{-}4)_2]$	58	dark brown	135	40.6 (40.5)	3.8 (3.9)		40.0 (39.1)	
$[Pd(COD)(TeC_6H_4OMe\text{-}4)_2]$	57	dark brown	132	38.6 (38.6)	3.8 (3.8)		37.7 (37.3)	
$[Pd(COD)(TeC_6H_4OEt\text{-}4)_2]$	70	dark brown	145	40.5 (40.4)	4.2 (4.2)		36.1 (35.8)	
$[Pd(PPh_3)_2Cl(TePh)]$	65	orange	165	57.9 (57.8)	4.0 (4.0)	4.4 (4.0)	14.0 (14.6)	915 (870)
$[Pd(PPh_3)_2Cl(TeC_6H_4Me\text{-}4)]$	56	orange	171	58.6 (58.3)	4.2 (4.1)	4.5 (4.0)	14.7 (14.4)	850 (884)
$[Pd(PPh_3)_2Cl(TeC_6H_4OMe\text{-}4)]$	54	orange	175	57.2 (57.3)	4.2 (4.1)	4.3 (3.9)	14.8 (14.1)	958 (900)
$[Pd(PPh_3)_2Cl(TeC_6H_4OEt\text{-}4)]$	56	orange	186	57.8 (57.7)	4.2 (4.2)	4.6 (4.2)	14.5 (13.9)	875 (914)
$[Pd(PPh_3)_2(TePh)_2]$	50	brown	151	55.2 (55.4)	3.7 (3.8)		25.1 (24.5)	988 (1039)
$[Pd(PPh_3)_2(TeC_6H_4Me\text{-}4)_2]$	52	brown	147	56.3 (56.2)	4.2 (4.1)		24.2 (23.9)	1014 (1067)
$[Pd(PPh_3)_2(TeC_6H_4OMe\text{-}4)_2]$	48	brown	149	54.7 (54.5)	4.1 (4.0)		23.6 (23.2)	1051 (1099)
$[Pd(PPh_3)_2(TeC_6H_4OEt\text{-}4)_2]$	50	brown	144	55.2 (55.3)	4.2 (4.2)		23.0 (22.6)	1091 (1127)
$[Pd_2Cl_4(PPh_3)_2(Te_2Ph_2)]$	41	brown	135	43.9 (43.7)	2.9 (3.1)	10.6 (11.0)	18.6 (19.8)	1265 (1288)
$[Pd_2Cl_4(PPh_3)_2(Te_2(C_6H_4Me\text{-}4)_2)]$	55	light brown	141	44.7 (45.5)	3.2 (3.3)	10.2 (10.7)	19.0 (19.3)	1293 (1316)
$[Pd_2Cl_4(PPh_3)_2(Te_2(C_6H_4OMe\text{-}4)_2)]$	65	reddish-brown	138	44.3 (44.5)	3.0 (3.2)	10.1 (10.5)	19.2 (18.9)	1321 (1348)
$[Pd_2Cl_4(PPh_3)_2(Te_2(C_6H_4OEt\text{-}4)_2)]$	50	light brown	145	45.0 (45.3)	3.3 (3.4)	9.8 (10.3)	18.0 (18.5)	1352 (1376)

[a] Calculated values are given in parentheses. [b] Molecular weights were determined in $CHCl_3$.

$$[Pd(COD)Cl_2] + 2Ph_3Sn\text{–}TeAr$$

$$= [Pd(COD)(TeAr)_2] + 2Ph_3SnCl$$

II

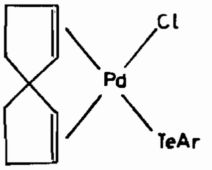
These reactions are reasonable since $SnPh_3TePh$ has been found to afford the tellurol complex $[Cu(TePh)]_n$ from CuCl in good yield [1, 11].

With an aim to prepare $Na_2[Pd(TeAr)_4]$, a reaction between $Pd(COD)Cl_2$ (1 mol) and NaTePh

TABLE 2. Important IR[a] frequencies of $[Pd(COD)Cl(TeAr)]$ (I) and $[Pd(COD)(TeAr)_2]$ (II)

Complex	ν(Pd–Cl) (cm^{-1})	ν(C=C) from COD (cm^{-1})	ν(C=C) from aromatic group (cm^{-1})
$[Pd(COD)Cl(TePh)]$	302	1472	1560
$[Pd(COD)Cl(TeC_6H_4Me\text{-}4)]$	309	1485	1561
$[Pd(COD)Cl(TeC_6H_4OMe\text{-}4)]$	315	1488	1584
$[Pd(COD)Cl(TeC_6H_4OEt\text{-}4)]$	311	1487	1583
$[Pd(COD)(TePh)_2]$		1469	1567
$[Pd(COD)(TeC_6H_4Me\text{-}4)_2)]$		1483	1587
$[Pd(COD)(TeC_6H_4OMe\text{-}4)_2)]$		1486	1584
$[Pd(COD)(TeC_6H_4OEt\text{-}4)_2)]$		1485	1585

[a] Recorded in the solid state as CsI pellets, for $Pd(COD)Cl_2$, ν(Pd–Cl) 334, 297 cm^{-1} and ν(C=C) from COD 1534 cm^{-1}.



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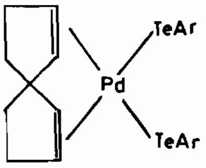


II

Fig. 1. Suggested structures for complexes I and II.

(4 mol) was carried out in ethanol. However, the isolated compound showed the stoichiometry corresponding to **II** only.

In order to study the displacement reactions of COD by PPh_3, complexes **I** and **II** were treated with appropriate amounts of PPh_3 in chloroform. These reactions led to the isolation of corresponding phosphine complexes **III** and **IV** according to the equations given below

$$[Pd(COD)Cl(TeAr)] + 2PPh_3 = \underset{\textbf{III}}{[Pd(PPh_3)_2Cl(TeAr)]} + COD$$

$$[Pd(COD)(TeAr)_2] + 2PPh_3 = \underset{\textbf{IV}}{[Pd(PPh_3)_2(TeAr)_2]} + COD$$

The yield, colour, decomposition temperature, analytical and molecular weight data of all the isolated complexes **III** and **IV** are compiled in Table 1. All the monotellurol complexes **III** are orange while bis-tellurol complexes **IV** are brown in colour. They are soluble in benzene, dichloromethane, chloroform, DMF and DMSO but insoluble in petroleum ether and diethyl ether. Conductivity measurements in acetonitrile showed their non-electrolytic nature. IR spectra (in the solid state as CsI pellets) showed one ν(Pd–Cl) band in **III** (Ar = phenyl, 302 cm^{-1}; Ar = *p*-tolyl, 298 cm^{-1}; Ar = *p*-anisyl, 309 cm^{-1}; Ar = *p*-phenetyl, 300 cm^{-1}) suggestive of the presence of a terminal chloride ligand, and none in **IV**. All complexes showed the absence of the ν(C=C) band for COD ligand indicating its displacement by PPh_3 from the complexes **I** and **II**. ^1H NMR spectra (Table 3) showed all the expected resonances in the correct intensity ratio. $^{31}P\{^1H\}$ NMR spectra (Table 3) (in $CDCl_3$ solution, H_3PO_4 as an external reference) of **III** showed two doublets of equal intensities which indicated the non-equivalence of the two phosphorus atoms in the complexes. The coupling constant $^2J(P\text{–}Pd\text{–}P)$ is of the order of 20–26 Hz characteristic of a *cis*-$Ph_3P\text{–}Pd\text{–}PPh_3$ system [12, 13]. Complex **IV** showed one ^{31}P signal in the high downfield side. Thus a *cis* configuration (Fig. 2) is reasonably assigned to complexes **III** and **IV**.

Complex **III** (Ar = phenyl) slowly turned black with H_2 in $EtOH/C_6H_6$ indicating the reduction of Pd(II) into the metallic state.

Solid Te_2Ar_2 on treatment with *trans*-$[Pd_2Cl_2(\mu\text{-}Cl)_2(PPh_3)_2]$ in dichloromethane yielded complexes of the type $[Pd_2Cl_4(\mu\text{-}Te_2Ar_2)(PPh_3)_2]$ (**V**), consistent with their molecular weight measurements. Their elemental analyses corresponded to the empirical formula $[PdCl_2(TeAr)(PPh_3)]$. The possibility of any ionic structure is eliminated by their conductance measurements in acetonitrile which showed their non-electrolytic nature. ^{31}P NMR spectra of the complexes **V** (in CD_2Cl_2, H_3PO_4 as an external reference) (Table 4) showed a single ^{31}P resonance indicating thereby the equivalence of the two phosphorus atoms in the *trans* position in the complexes (*trans*-$[Pd_2Cl_4(\mu\text{-}Cl)_2(PPh_3)_2]$ showed a single ^{31}P chemical shift at δ33.25 ppm). The red to brown colour of the products strongly suggests that the tellurium–tellurium bond has survived the complex forming reactions. The presence of the ditelluride ligand in the complexes was confirmed by their reactions with PPh_3 in diethyl ether which liberated the ditelluride. Far IR spectra (Table 4) of the complexes (in the solid state as polyethene pellets) showed only two ν(Pd–Cl) bands in the regions 322–314

TABLE 3. ^{1}H NMR^a and ^{31}P NMR^b spectra of $[Pd(PPh_3)_2Cl(TeAr)]$ (III) and $[Pd(PPh_3)_2(TeAr)_2]$ (IV)

Complex	δ Aromatic H (ppm)	δCH$_2$ (ppm)	δCH$_3$ (ppm)	δP (ppm)	^2J(PP) (Hz)
$[Pd(PPh_3)_2Cl(TePh)]$	6.97–7.81(m)			18.6(d) 17.6(d)	22
$[Pd(PPh_3)_2Cl(TeC_6H_4Me\text{-}4)]$	6.45–7.68(m)		2.27(s)	19.2(d) 18.7(d)	20
$[Pd(PPh_3)_2Cl(TeC_6H_4OMe\text{-}4)]$	6.50–7.90(m)		3.75(s)	17.2(d) 16.5(d)	25
$[Pd(PPh_3)_2Cl(TeC_6H_4OEt\text{-}4)]$	6.24–7.76(m)	3.88(q) (J = 7 Hz)	1.37(t) (J = 7Hz)	17.1(d) 16.3(d)	23
$[Pd(PPh_3)_2(TePh)_2]$	6.71–7.82(m)			26.1(s)	
$[Pd(PPh_3)_2(TeC_6H_4Me\text{-}4)_2]$	6.67–7.49(m)		2.26(s)	29.4(s)	
$[Pd(PPh_3)_2(TeC_6H_4OMe\text{-}4)_2]$	6.49–7.34(m)		3.76(s)	29.5(s)	
$[Pd(PPh_3)_2(TeC_6H_4OEt\text{-}4)_2]$	6.56–7.78(m)	3.91(q) (J = 7 Hz)	1.36(t) (J = 7 Hz)	23.3(s)	

[a] Recorded in $CDCl_3$, s = singlet, t = triplet, q = quartet, m = multiplet. [b] Recorded in $CDCl_3$, s = singlet, d = doublet, H_3PO_4 as external reference.

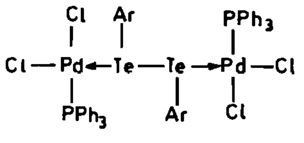
TABLE 4. ^{1}H^a, ^{31}P^b and IR^c spectra of $[Pd_2Cl_4(\mu\text{-}Te_2Ar_2)(PPh_3)_2]$ (V)

Complex	δ Aromatic H (ppm)	δCH$_2$ (ppm)	δCH$_3$ (ppm)	δP (ppm)	ν(Pd–Cl) (cm^{-1})	ν(Te–Te) (cm^{-1})
V, Ar = Ph	6.40–8.54(m)			36.50	315, 318	191 182(R)
V, Ar = C_6H_4Me-4	6.27–8.33(m)		2.30(s)	35.87	312, 322	177
V, Ar = C_6H_4OMe-4	6.36–8.31(m)		3.76(s)	36.37	305, 314	197
V, Ar = C_6H_4OEt-4	6.33–7.68(m)	3.99(q) (J = 7 Hz)	1.36(q) (J = 7 Hz)	35.88	310, 317	198

[a] Recorded in DMSO-d_6, s = singlet, t = triplet, q = quartet, m = multiplet. [b] Recorded in CH_2Cl_2, H_3PO_4 as external reference. [c] Recorded in the solid state as polyethene pellets.

III IV

Fig. 2. Suggested structures for complexes III and IV.



V

Fig. 3. Proposed structure for complex V.

and 315–305 cm^{-1} suggestive of the presence of terminal chloride and the absence of bridging chloride ligands; thus favouring the facile replacement of the bridging chloride ligands in the initial palladium complexes by diaryl ditelluride. Further, the ν(Te–Te) band appeared in the far IR spectra of all the complexes in the region 177–198 cm^{-1} and at 182 cm^{-1} in the Raman spectrum of complex V (Ar = phenyl). Other complexes (V, Ar = *p*-tolyl, *p*-anisyl and *p*-phenetyl) decompose on exposure to laser radiation and hence their Raman spectra could not be recorded. ^{1}H NMR spectra (Table 4) (in DMSO-d_6) showed the equivalence of all aryl groups in the complexes V. Thus a *trans* dimeric structure with two $PdCl_2(PPh_3)$ moieties bridged by one molecule of ditelluride is reasonably proposed for complexes V (Fig. 3).

Complex V (Ar = phenyl or *p*-anisyl) on refluxing with $CHCl_3$ or MeOH undergoes decomposition as suggested by the qualitative test and ^{1}H NMR. However, the ditelluride complex of the type $[Pd_2Cl_4(Ar_2Te_2)_2]$ on refluxing with ethanol/methanol, as reported earlier, resulted in the formation of polymeric tellurol [5].

$$[Pd_2Cl_4(Ar_2Te_2)(PPh_3)_2] \xrightarrow[\text{CHCl}_3\text{ or MeOH}]{\text{reflux}} 2PdCl_2 + 2PPh_3 + Te + Ar_2Te$$

$$[Pd_2Cl_4(Te_2Ar_2)_2] \xrightarrow[\text{EtOH/MeOH}]{\text{reflux}} [PdCl(TeAr)]_n$$

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